AN-4

306
Scientific Notebook # 127

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AN-4

Natural Analog Research Project and
Radionuclide Transport KTI
Lab and Data Analysis Work
Informal Number "AN-04"

DAP = David Pickett
JP = Jim Prikryl

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[signature] 8/25/2000

SEE MEMO ON 2nd CONTENTS PAGE.

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Subject: **SCIENTIFIC NOTEBOOK No. 126 / 127**
Date: Fri, 25 Aug 2000 12:24:42 -0500
From: Bruce Mabrito <bmabrito@gargol.cnwra.swri.edu>
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The purpose of this e-mail message is to document the location of scientific notebook number 127.

First, scientific notebook 126 was issued to Gordon Wittmeyer on 12/8/1994. That notebook was utilized and then returned to QA Records 2/22/1996.

During the August 2000 call for scientific notebooks, another scientific notebook, also numbered 126, was returned. It has been determined that QA "double issued" the number 126. Apparently, I issued No. 126 to Gordon Wittmeyer in 1994 and Alice Cortinas (Records Custodian) issued a second scientific notebook with the same number 126 on 12/21/1994 to Jim Prikryl. In the call for notebooks, we discovered this fact. We had assumed that scientific notebook No. 127 was lost and a memorandum was issued from Dr. English Pearcy on June 15, 2000 so stating. It is very clear that this notebook should have been identified number 127 because all other identifying information (issuer, receiver of notebook, dates, and project numbers) otherwise match well

The following actions are being taken. A B. Mabrito signed copy of this e-mail message will be attached to the E. Pearcy memo of 6/15/2000 and also to the scientific notebook issued to Jim Prikryl. Second, the number 126 on the front of Jim Prikryl's notebook (which is now being used by Dr. David Pickett) will be changed to number 127 and initialed and dated by B. Mabrito. The Scientific Log Notebook will be corrected to reflect that scientific notebook No. 127 was not lost and is now in use by Dr. David Pickett.

Bruce Mabrito 8/25/2000

Bruce Mabrito

7/26/95 JP

Natural Analog Research Project

Initial entry 7/26/95 by James D. Prikryl JP

This notebook chronicles the laboratory investigations of the Geochemical Natural Analog Research Project.

7/26/95 [illegible]

Portions of bulk rocks samples from the +10 level of Wopal I (NOPI-459 thru NOPI-493) were powdered using a SPEX Mixer/Mill and tungsten carbide vial. These powdered samples were labeled as below:

Label	Wt. of powder
NOPI-459-WR1	26.01 g
NOPI-460-WR1	20.99 g
NOPI-461-WR1	24.32 g
NOPI-462-WR1	22.41 g
NOPI-463-WR1	23.51 g
NOPI-464-WR1	26.59 g
NOPI-465-WR1	24.51 g
NOPI-466-WR1	26.01 g
NOPI-467-WR1	25.91 g
NOPI-468-WR1	26.27 g
NOPI-469-WR1	26.85 g
NOPI-470-WR1	24.58 g
NOPI-471-WR1	24.42 g

Label	Wt. of powder
NOPI-472-WR1	28.55 g
NOPI-473-WR1	27.48 g
NOPI-474-WR1	27.14 g
NOPI-475-WR1	25.76 g
NOPI-476-WR1	31.08 g
NOPI-477-WR1	25.12 g
NOPI-478-WR1	21.23 g
NOPI-479-WR1	24.34 g
NOPI-480-WR1	29.30 g
NOPI-481-WR1	20.75 g
NOPI-482-WR1	26.12 g
NOPI-483-WR1	25.52 g
NOPI-484-WR1	26.86 g
NOPI-485-WR1	24.16 g
NOPI-486-WR1	22.03 g
NOPI-487-WR1	23.45 g
NOPI-488-WR1	32.69 g
NOPI-489-WR1	24.10 g

Label	Wt of powder
NOPI-490-WR1	28.54g
NOPI-491-WR1	29.61g
NOPI-492-WR1	23.31g
NOPI-493-WR1	34.46g

8/2/95 JP

Uranophane separates of NOPI-494.

Fracture filling uranophane from Nopal I (sample NOPI-494) was separated by hand picking + washing.

Three separate <1g uranophane samples were processed.

The uranophane was placed in glass vials and labeled as follows:

NOPI-494-SEP1
NOPI-494-SEP2
NOPI-494-SEP3

These samples have trace impurities; mostly silicates.

8/2/95 gp

Whole rock powders were placed and sealed in polyethylene vials for gamma analysis. Samples were labeled as below and amount of powder placed in each vial is also shown. Silicone cement was used to seal vials

Sample	Wt. of powder
NOPI-466-GAM1	3.001 g
NOPI-467-GAM1	3.003 g
NOPI-468-GAM1	3.002 g
NOPI-469-GAM1	2.999 g
NOPI-470-GAM1	3.000 g
NOPI-471-GAM1	3.002 g
NOPI-472-GAM1	3.001 g
NOPI-473-GAM1	3.001 g
NOPI-474-GAM1	3.003 g
NOPI-475-GAM1	2.999 g

8/2/95

Uranophane separates dissolution.

Separates of uranophane NOPI-494-SEP1, NOPI-494-SEP2 and NOPI-494-SEP3 were weighed and placed in Teflon beakers.

100 ml of 0.1m $HClO_4$ were added to each beaker to dissolve the uranophane.

These solutions will be used for U-series analysis of the uranophane.

Weight of uranophane placed in beaker -

gp 8/2/95

NOPI-494-SEP1 - ~~0.0417 g~~ 0.0705 g
NOPI-494-SEP2 - ~~0.0206 g~~ 0.0489 g
NOPI-494-SEP3 - ~~0.0335 g~~ 0.0353 g

8/3/95

The uranophane separate samples that were dissolved in 0.1 m $HClO_4$ were analyzed for U + Th isotopes by α-spectrometry using procedure in Notebook 875 (AN-2) on p 59-62.

Acid digestion step by microwave was not performed since samples were already dissolved

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

Sample ID's are
NOPI-494-SEP1
NOPI-494-SEP2
NOPI-494-SEP3

8/10/95 JJP

Uranophane separates analyzed by α-spec on previous page will have to be reanalyzed.

During mounting of U on membrane filters a green precipitate formed

9/5/95 gp

Gamma spectrometry of rock powders was conducted on samples from Nopal I using the procedure on the following pages.

The following sample powders were analyzed:

NOPI-466-GAM1
NOPI-467-GAM1
NOPI-468-GAM1
NOPI-469-GAM1
NOPI-470-GAM1
NOPI-471-GAM1
NOPI-472-GAM1
NOPI-473-GAM1
NOPI-474-GAM1
NOPI-475-GAM1

GAMMA SPECTROMETRY OF ROCK POWDERS

Objective: measure natural decay-series radionuclide concentrations in rock powders

Method: gamma spectrometry; counts detected at specified gamma lines are normalized to counts from standard powders (after correction for sample self-attenuation) and corrected for sample size

Materials:
-round hinged-lid polypropylene PP vials, 0.5"X1.5"
-silicone sealant
-NIST SRM gamma point source 4275C
-EPA (SP-1, SM-1, DP-1, DM-1, CM-1) and CANMET (BL-5) U and Th series natural rock standard reference powders

Equipment: Canberra low energy germanium detector (Model GL2020R) with a carbon composite window with associated cryostat and electronic components, controlled by Canberra PC-Genie software

Procedure:

1. Weigh approximately 3 g powder into PP vial; record weight of powder. Store samples for 120 days before analysis if there is a possibility of ^{238}U-^{234}Th disequilibrium (e.g., weathered surface materials). Reference powders should also be 3 g in weight.

2. Seal vial with silicone sealant, taking care not to spill powder or contaminate powder with sealant. Over the next few hours, check vial to ensure the lid is evenly closed; the lid may rise up during drying and break the seal.

3. Let samples sit for at least 19 days to ensure equilibrium between radon isotopes and their daughters.

4. Self-Attenuation Measurement (may be done during 19-day wait):
   a. Place plastic cap on detector.
   b. Center SRM 4275C disk on top of an empty vial centered on detector cap and close shield.
   c. Acquire gamma spectrum using standard acquisition mode for 1800 sec.
   d. Center SRM 4275C disk on top of sample vial centered on detector cap, close shield, and acquire spectrum for 1800 sec. (*Note-ensure an even layer of powder across the vial, with no gaps before counting)
   e. Repeat for all samples and standards to be used. Perform these measurements within a few days of each other so than no decay corrections for SRM 4275C are necessary.
   f. In Spectroscopy Assistant, perform a manual data analysis including only steps for peak locate and peak analysis.

5. Sample Counting
   a. Remove detector cap if present. Platic wrap should be covering detector window.
   b. Center sample vial on window, avoid applying pressure to the window. Close

shield.

c. Acquire gamma spectrum for 80,000 sec (approx. 22 hours).

d. Repeat for all samples, standards to be used for normalization (e.g., DP-1 and DM-1), and standards to be used for accuracy check (e.g., BL-5).

e. In Spectroscopy Assistant, perform a manual data analysis for each sample and standard including the steps for peak locate, peak analysis, efficiency correction, background correction, and nuclide identification.

6. Offline Self-Attenuation Correction

a. In a Excel spreadsheet, enter count data for gamma peaks from self-attentuation measurements at 27.3, 42.9, 48.6, 86.4, 105, 123, 176, 248, 592, 600, and 636 keV.

b. Calculate attentuation at each of these peaks relative to the empty vial using:

$$\text{"A/O"} = [\ln(T/I)]/[(T/I)-1]$$

where T is the transmitted intensity through the sample and I is the intensity through the empty vial.

c. Calculate a correction factor at each SRM 4275C gamma peak for each sample (subscript S) relative to each normalization standard to be used (subscript N, e.g., DP-1 and DM-1) using:

$$CF_S = (A/O)_S/(A/O)_N$$

d. For each sample, after ensuring that CF values for >200 keV peaks are within error of one, fit a polynomial curve (usually of 4th order) through a plot of CF versus keV for peaks <200 keV.

e. For all samples and standards, calculate CFs for natural series gamma peaks at 25.6, 27.4, 46.5, 50, 53.2, 63.3, 67.7, 92.6, 129, and 186 keV using the polynomial equation.

7. Offline Activity Calculations

a. At the natural series gamma peaks listed in Table 1, calculate the corresponding radionuclide activity using:

$$A_S\ (Bq/g) = I_S * CF_S * A_N * W_N / I_N * W_S$$

where A_S, I_S, CF_S, and W_S are the radionuclide activity, peak intensity (cps), correction factor relative to the normalization standard N, and weight (g), respectively, of the sample, and A_N, I_N, and W_N are the nominal radionuclide activity (Bq/g), peak intensity, and weight of the normalization standard. Note that I_S and I_N should be the background-corrected values from the Spectroscopy Assistant output.

b. As mentioned above, CFs should be equal to one for >200 keV peaks.

c. At 53 and 186 keV, it will be necessary to apply interference corrections; these have not yet been fully developed.

d. It is preferable to use one standard (e.g., DP-1) for normalization of ^{238}U and ^{235}U series nuclides, and another (e.g., DM-1) for ^{232}Th series nuclides. Other reference powders will be treated as unknowns for checking accuracy.

e. When multiple peaks are available for a given nuclide, the analyst may either take the average (e.g., ^{214}Pb from the average of 295 and 352 keV) or choose a single peak (e.g., ^{234}Th from 63, rather than 92, keV) based on considerations such as peak uncertainty and possible interferences.

f. The analyst should be aware of possible decay corrections from shorter-lived nuclides.

g. The reported uncertainty for a given radionuclide should be computed as the square root of the sum of squares of the following uncertainties: (1) sample counting uncertainty, (2) standard counting uncertainty, and (3) reported standard activity uncertainty. In practice, the standard will be chosen such that the uncertainty added by the self-attenuation correction is negligible. The total uncertainty should be compared wit the accuracy as determined from analysis of standards as unknowns.

Table 1 - Gamma peaks and corresponding nuclides

keV	nuclide
25.6	$^{231}Th \rightarrow ^{235}U$
27.4	^{231}Pa
46.5	^{210}Pb
50.0	^{227}Ac
53.2	$^{234}U + ^{214}Pb$
63.3	$^{234}Th \rightarrow ^{238}U$
67.7	^{230}Th
92.6	$^{234}Th \rightarrow ^{238}U$
129	$^{228}Ac \rightarrow ^{228}Ra$
186	$^{235}U + ^{226}Ra$
236	$^{227}Th \rightarrow ^{227}Ac$
239	$^{212}Pb \rightarrow ^{228}Th$
242	$^{214}Pb \rightarrow ^{226}Ra$
295	$^{214}Pb \rightarrow ^{226}Ra$
352	$^{214}Pb \rightarrow ^{226}Ra$
583	$^{208}Tl \rightarrow ^{228}Th$
609	$^{214}Bi \rightarrow ^{226}Ra$

Results from these analyses will be kept in a 3-ring binder entitled "LeGE Gamma Spectrometry of Nepal I Samples."

9/18/95 gp

Portions of the following bulk rocks were powdered using a SPEX Mixer/Mill + tungsten carbide vial. These samples are from the original 2m EW transect that cuts the western boundary of the orebody. Samples were labeled as below.

Label	Wt of powder
NOPI-113-WR2	28.76 g
NOPI-114-WR2	25.81 g
NOPI-115-WR2	16.75 g
NOPI-116-WR2	24.63 g
NOPI-117-WR2	24.52 g
NOPI-118-WR2	21.56 g
NOPI-119-WR2	19.41 g
NOPI-120-WR2	22.58 g
NOPI-121-WR2	21.95 g
NOPI-122-WR2	18.61 g
NOPI-123-WR2	28.33 g

9/19/95 gp

Portions of whole rock powder on previous page were placed + sealed in polyethylene vials for gamma analysis. Samples were labeled as below & amount of powder placed in vials is recorded. Vials were sealed with silicone cement and allowed to equilibrate for 20 days before analysis.

Results of gamma analyses on these samples will be kept in a 3-ring binder titled "LeGE Gamma Spectrometry of Napal I Samples.

Label	wt
NOPI-113-GAM3	3.002 g
NOPI-114-GAM3	3.006 g
NOPI-115-GAM3	3.001 g
NOPI-116-GAM3	3.002 g
NOPI-117-GAM3	3.001 g
NOPI-118-GAM3	3.002 g
NOPI-119-GAM3	3.002 g
NOPI-120-GAM3	3.002 g
NOPI-121-GAM3	3.003 g
NOPI-122-GAM3	3.004 g
NOPI-123-GAM3	3.003 g

10/10/95 JP

Rerun of uranophane separates by alpha-spec (see p 8)

The following volumes of solution was used in each analysis.

Sample No		
NOPI-494-1A	1 ml	of NOPI-494-SEP1
NOPI-494-1B	2 ml	" "
NOPI-494-2A	1 ml	of NOPI-494-SEP2
NOPI-494-2B	2 "	" "
NOPI-494-3A	1 ml	of NOPI-494-SEP3
NOPI-494-3B	2 m	" "

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

Procedure in notebook 075 (AN-2) p. 59-62 was used to do analyses.

12/28/95 JP

During gamma analysis of samples listed on p 10 problems with the detector were encountered.

Tests conducted on the detector by Canberra repair staff required that the detector be sent to the manufacturer for repair.

The detector was returned to CNWRA Bldg 57 on 12/8/95. The detector was installed, tested, and calibrated.

Analysis of samples on p 10 will now continue using procedure outlined on p11-13.

1/30/96
DAP
(= David Pickett)

Uranophane Separates for U/Pb — continued from notebook 080/303 ("AN-3")

Have produced six uranophane "separates" from Nopal I ore deposit samples by scraping uranophane ("uph") from surfaces and doing a limited amount of hand-picking.
Will now inspect previous XRD data to determine what mineral contaminants are possible.

NOPI-494 from which three samples were taken (080/302).
XRD data from a printout provided by J. Prikryl.
File name = NOPI4941.MDI 6/14/95
Presumably a whole-rock powder, but not sure.

Quartz @ 26.6 2θ is minor. I% = 2.5 ~~(d=7.78)~~ DAP 1/31/96
Uranophane @ 11.3 2θ is largest peak. (≡100%) (d=7.78)
@ 22.6 2θ also large (54%) (d=3.92)
@ 34.2 2θ 12.1% (d=2.62)
@ 46.1 2θ 12.2% (d=1.97)
It seems one cannot distinguish α from β. X-ray lists are very similar.

Kaolinite @ 12.5 2θ, d=7.06 only 3.7%. Is one of two most abundant peaks.
@ 24.9 2θ, d=3.57 9.5% Not bad.
@ 20 2θ, d=4.41 not found (should be 60%).

Other large peaks:
2θ = 13.6 d = 6.52 I = 52% Not hematite, goethite, pyrite, ianthinite, schoepite, soddyite, weeksite.
Conclusion — it is uph, which has a 7-40% peak at 6.61 to 6.66 Å which is due to the 001 or 100 cell (different in different refs.). It is "amplified" to 50% by virtue of cleavage (100) and/or crystal faces (100 or 001) preferentially aligned.

1/30/96 cont. Note — I am using as a reference: the Mineral Powder Diffraction File - Data Book, 1986, publ. by the International Centre for Diffraction Data.

It looks like uranophane can account for all significant peaks (i.e. > 5%).
Since this was apparently not a whole-rock powder, it is not particularly useful for deciding what potential contaminants are.

NOPI-102 AKA NOPI-ECP-26.85/11.65
Data from a brown "Accopress" binder labeled "Nopal 1 Random Sample," in form of (1) a table of observed peaks and (2) chart recorder record of scan.
Peaks not covered by uranophane or kaolinite.
d = 6.33 Å very small. Bump at base of larger peak.
Probably an artifact.
6.33 Å doesn't show upon slower scan. (XRD2)

1/31/96
DAP
Therefore, it looks like uph and kaolinite can account for all significant peaks.
Again, this was apparently a uph, rather than whole-rock, sample.

NOPI-104 AKA NOPI-ECP-24.0/10.5
Data from same brown binder as NOPI-102.
All peaks accounted for by uph, kaolinite, quartz.
Note: spectra from other uph samples from nearby have a suggestion of hematite.

NOPI-291 XRD data from white notebook labeled "XRD Analyses — Nopal I Deposit - Level 0 - Chih. Mex." DAP 1/31/96
Spectrum and table of peaks.
Qtz. present and is largest peak. (@ 26.6 2θ. Also 13% @ 50.1 2θ)
Therefore. Qtz, uph, and kaolinite again take care of all peaks.

1/31/96 DAP

cont.
Want a rough idea of sample sizes of uph "separates."
Did not weigh empty vials before adding sample, so
take average of weights of four empty vials:
3.7636 3.7227 3.8120 3.7413
average = 3.7599 = 3.76 g
range 3.72 – 3.81 g
Now weigh samples in vials

	wt. using avg.	wt. range
NOPI-104-UPb		
3.7414	—	0 – 0.02 g
NOPI-102-UPb		
3.8618	0.10	0.05 – 0.14 g
NOPI-291-UPb		
3.8638	0.10	0.05 – 0.14 g
NOPI-494-UPb-1		
3.8696	0.11	0.06 – 0.15 g
NOPI-494-UPb-2		
3.8191	0.06	0.007 – 0.096 g
NOPI-494-UPb-3		
3.8246	0.06	0.01 – 0.10 g

So, samples probably range between 50 and 150 mg.

By visual inspection, the largest samples appear to be 102, 291 and 494-UPb-2.
Therefore, use NOPI-494-UPb-2 as first sample.

2/5/96 DAP w/ JDP (Jim Prikryl)

Heavy Liquid Separation of NOPI-494-UPb-2
Using methylene iodide, spec grav. = 3.32
125 ml sep funnel w/ MI added to just below ~~letting~~ DAP 2/5/96, lettering on funnel.
Shake well because clumps formed and sank. Let sit overnight.

2/6/96 DAP with JDP

Uph has collected, though much remains stuck to walls.

Set up filter funnel on top of Erlenmeyer flask (300 ml funnel, 1 liter flask) w/ vacuum pump attached. Use 0.45 μm, 47mm diameter, nylon membrane filters (Whatman catalog # 7404 004).
Let out just enough w/ stopcock to remove all collected uph. Pump long enough to dry out (10–15 sec.).
To recover more of uph stuck to walls, shake up separatory funnel and let settle again.
Rinse filtrate with acetone by (1) adding to filter funnel and (2) turning on pump for ~30 sec while adding more acetone.
Some uph got stuck between flanges between filter funnel and filter holder. Rinse with acetone onto new filter placed on holder with pump running.
Filters are placed in a petri dish (w/ uph on them).

Considering using HCl to dissolve out Fe oxides (and perhaps Mn? oxides in β-uranophane coating).
Take some of the synthetic uranophane and add HCl.

Weigh some uph from container marked "URAN*SYN 9A+10A" into 15 ml PP beaker.

beaker + uph 2.7479
empty beaker 2.4579
0.2900 g uph.

Beaker labeled "UPH TEST". Add 10 ml 8M HCl.
Beaker is too small. Use 50 ml.
empty 50 ml beaker = 8.6486 g
Transfer uph & acid to large beaker and rinse with more HCl.
Total soln ~40 ml. It is yellow, so it is dissolving uph.

2/6/96 cont. 4 hours later, not completely dissolved but most is dissolved. Don't use 8M HCl.
Transfer to 60 ml bottle labeled "UPH TEST SOLN."

Back to heavy liquid sep. — A small amount of uph has settled. Filter out and collect in another filter. Clean up.
Retain filtrate from remaining MI by rinsing with acetone in a beaker. Rinse 3x with acetone. Label "NOPI-494-UPb-2 Remainder."

2/7/96 DAP Another test using synthetic uph for finding a reagent that will dissolve oxides.
Use Coffin's reagent which is used in selective leaching procedure to attack crystalline oxides.

50 ml beaker labeled "UPH TEST 2"
Add uph from "URAN*SYN 9A + 10A"

+ uph	8.8601
empty beaker	8.6462
total uph	0.2139 g

Add ~20 ml Coffin's Reagent at 10:30. Then add 1 g sodium dithionite. Add magnetic stir bar and put on low on stir plate 10:45.

Back to NOPI-494-UPb-2. Products of heavy liquid separation are viewed under binoc. microscope. Still quite a bit of oxides (Fe, probably). Can hand pick some larger grains, as well as some pinkish grains appearing to contain clays and/or quartz ~~an~~ and/or feldspar.
When finished hand-picking, transfer uph back to original snap-cap vial (rinsed) with H_2O. Decant excess water from vial.

2/7/96 cont.
at 13:30, pour Coffin's test (UPH TEST 2) into centrifuge tube + rinse 3x with H_2O. Centrifuge, ~~+ 2 min.~~ DAP 2/7/96 5 mins.
Transfer with water rinses to pyrex beaker.
empty wt. beaker = 28.8530 g

Dry and filter. Filtration was a bad idea, because much of the uph passes through. Dry the H_2O that has passed through.

Meanwhile, do oxide leaching on NOPI-494-UPb-2
Add 15 ml of Coffin's reagent to 50 ml Teflon Oak Ridge tube. Add 0.75 g sodium dithionite. Transfer uph sample (which has been through heavy liquid separation) to tube using reagent to wash out of vial with plastic pipet. Close tightly and place on wrist shaker for 1 hour.
Timer on shaker out of whack. It must have been more like two hours that it shook.

Centrifuge + decant. Add 15 ml H_2O, centrifuge, decant. Rinse solids three times w/ H_2O.
Transfer to original snap-cap vial.
Still see dark grains.

2/8/96 DAP
UPH TEST 2 — uph is now in two places: dried in bottom of pyrex beaker, and on filter paper
weight beaker 28.9602
→ uph in beaker = 0.1072 g
weight filter paper + uph = 0.7281
" mother piece filter paper = 0.5514
→ est. wt uph on paper = 0.1767 g
Sum of uph = 0.2839 g Much higher than initial — probably moisture still on paper.

2/8/96 cont.
DAP

NOPI-494-4Pb-2 Visual inspection ⇒ leaching did not remove appreciable amount of oxides. Do some hand picking of larger grains.

Took a small amount of this uph (with a few grains of oxides, both black and reddish) and added <1 ml 1M HCl. The uph dissolved in a matter of minutes! Oxides were not. Here is the way to effect separation!

Transfer uph back to orig. vial with H_2O.
empty vial = 3.6714 g

Decant excess H_2O and dry under heat lamp.

2/9/96
DAP

When dry, weight vial + uph = 3.7262 g

⇒ Wt. uph = 0.0548 g

Mail to Larry Mach at U. Texas at Austin.

2/20/96 JP

The following powders were sealed in polyethylene vials for gamma analysis. These samples are from the 2m transect on level +10 that were powdered by B. Leslie in
Samples were labeled as below & the weight of powders placed in vials is recorded. Vials were sealed with silicone cement + allowed to equilibrate for 20 days before counting.

Results will be kept in a 3-ring binder entitled "LeBe Gamma Spectrometry of Nopal I samples".

Label	Weight
NOPI-113-GAM4	1.340 g
NOPI-114-GAM4	2.119 g
NOPI-115-GAM4	1.965 g
NOPI-116-GAM4	1.815 g
NOPI-117-GAM4	2.923 g
NOPI-118-GAM4	2.741 g
NOPI-119-GAM4	2.820 g
NOPI-120-GAM4	3.001 g
NOPI-121-GAM4	3.006 g
NOPI-122-GAM4	2.928 g
NOPI-123-GAM4	2.834 g

2/27/96 JP

The following rock standards were analyzed for U-Th isotopes by alpha-spectrometry

BL-5 Canmet pitchblende
DP-2 Diluted pitchblende
DM-1 Diluted monazite

Procedure in notebook 075 (AN-2) p 5962 was used to do analyses.

Results are kept in a 3 ring binder entitled "Alpha-Spectrometry of U-Th standards".

2/27/96 JP

Uranophane separation of NOPI-494.

Additional uranophane from sample NOPI-494 from Nopal I was separated by handpicking + washing

Three separate uranophane samples were processed.

The uranophane was weighed and placed in plastic vials which were labeled as below:

	weight
NOPI-494-SEP4	.035 g
NOPI-494-SEP5	.0296 g
NOPI-494-SEP6	.0481 g

These samples contain trace impurities of iron oxides + silicates.

2/27/96 JP

Uranophane dissolution

Uranophane separate samples NOPI-494-SEP4, NOPI-494-SEP5, and NOPI-494-SEP6 were placed in beakers and dissolved in ~80 ml of 0.1 M HCl

These solution will be used for U-Th isotope analysis of the uranophane. They are labeled as above.

2/27/96 JP

Uranophane separates were analyzed for U-Th isotopes by alpha spectrometry

The following volume of solution was used in each analysis

Sample No	
NOPI-494-SEP4	2 ml
NOPI-494-SEP5	2 ml
NOPI-494-SEP6	2 ml

In addition previous uranophane separate solution will be reanalyzed for U-Th isotopes

Sample NO	
NOPI-494-SEP1	2 ml
NOPI-494-SEP2	2 ml
NOPI-494-SEP3	2 ml

Procedure in notebook 075 (AN-2) p 59-62 was used to do analyses.

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

4/10/96 JP

The following whole rock powders were sealed in polyethylene vials for gamma analysis. These samples are from trenches on the level +0 m surface of Nopal I.

Samples were labeled as below + the weight of powder placed in each vial is recorded. Vials were sealed with silicone cement + allowed to equilibrate for 20 days before counting via procedure of pp 11-13

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

Label	wt
NOPI-308-GAM2	3.006g
NOPI-309-GAM2	3.002g
NOPI-311-GAM2	3.004g
NOPI-315-GAM2	3.003g
NOPI-319-GAM2	3.005g
NOPI-323-GAM2	3.005g
NOPI-326-GAM2	3.007g
NOPI-328-GAM2	3.004g
NOPI-269-GAM2	3.007g
NOPI-272-GAM2	3.004g
NOPI-275-GAM2	3.009g
NOPI-278-GAM2	3.006g
NOPI-355-GAM2	3.005g
NOPI-358-GAM2	3.002g
NOPI-361-GAM2	3.002g

Label	wt
NOPI-254-GAM2	3.001g
NOPI-258-GAM2	3.006g
NOPI-262-GAM2	3.003g
NOPI-265-GAM2	3.005g
NOPI-338-GAM2	3.003g
NOPI-342-GAM2	3.008g
NOPI-346-GAM2	3.002g
NOPI-350-GAM2	3.009g

4/11/96
DAP

Making arrangements for chemical analysis of Nopal I water samples.
Phone conversations with Cassia Wolfson, (512)471-4810, University of Texas, Geological Sciences Department, indicate that she could perform cation/anion analyses on our samples.
List she gave over phone:
anion: F, Cl, NO_3, NO_2, Br, SO_4, PO_4
cation: Al, Ba, Ca, Co, Cr, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Si, Sr, Zn.

Our filtered acidified samples (see books 121/29-34, 117/44-64, 080/299-301) are ready for cation analysis.
Unacidified samples were merely decanted, so filtering may be necessary before anion analysis. Need to check.

5/13/96 gp

The following whole rock powders were sealed in polyethylene vials for gamma counting. See page 30 for procedure.

Label	wt
NOPI-310-GAM2	3.008 g
NOPI-312-GAM2A	3.000 g
NOPI-312-GAM2B	3.003 g
NOPI-313-GAM2	3.001 g
NOPI-314-GAM2	3.003 g
NOPI-316-GAM2	3.005 g
NOPI-317-GAM2	3.005 g
NOPI-318-GAM2	3.005 g
NOPI-320-GAM2	3.004 g
NOPI-321-GAM2	3.006 g
NOPI-322-GAM2	3.003 g
NOPI-324-GAM2	3.006 g
NOPI-325-GAM2	3.003 g
NOPI-327-GAM2	3.007 g
NOPI-329-GAM2	3.005 g

6/7/96 gP

The following whole rock powders were sealed in polyethylene containers for gamma counting. See p. 30 for procedure.

Label	Wt
NOPI-255-GAM2	3.011 g
NOPI-256-GAM2	3.000 g
NOPI-257-GAM2	3.000 g
NOPI-259-GAM2	3.006 g
NOPI-260-GAM2	3.009 g
NOPI-261-GAM2	3.009 g
NOPI-263-GAM2	3.002 g
NOPI-264-GAM2	3.006 g
NOPI-266-GAM2	3.004 g
NOPI-267-GAM2	3.008 g
NOPI-268-GAM2	3.006 g
NOPI-339-GAM2	3.005 g
NOPI-340-GAM2	3.012 g
NOPI-341-GAM2	3.002 g
NOPI-343-GAM2	3.008 g
NOPI-344-GAM2	3.002 g
NOPI-345-GAM2	3.006 g
NOPI-347-GAM2	3.002 g
NOPI-348-GAM2	3.001 g
NOPI-349-GAM2	3.007 g
NOPI-351-GAM2	3.009 g
NOPI-352-GAM2	3.002 g

8/2/96 gP

The following rock standards were analyzed for U-Th isotopes by alpha-spectrometry

DP2 Diluted pitchblende
DM1 Diluted monazite
DP2DM1 mix

Procedure in notebook 075 (AN-2) p 59-62 was used to do analyses

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

8/5/96
DAP

Locations ~~of Samples from which Uph removed~~ OK DAP 8/5/96

NOPI-102 +10 Level
26.85/11.65 (EW/NS)

NOPI-104 +10 Level 24.00/10.5

NOPI-291 Vertical Wall above +00 Level.
Number on wall is 9; distance from "9" is 0.4 m to right.
Vertical height is 1.1 m

NOPI-494 +10 Level 22.00/12.00

Refer back to pp. 18-24 of this notebook.

Sending two more samples to Larry Mack at Univ. of Texas at Austin (see p 24) for U/Pb analysis for age determination. His preliminary work on NOPI-494-UPb-2 suggests an age of ~3 Ma.
Select for sending:

NOPI-102-UPb - because it is further from NOPI-494 than is NOPI-104, and it has good, visible Uph needles.

NOPI-291-UPb - because ① it is from just 1.1 m above Level +00, while the others are from Level +10, ② it's texture is quite different from the others (080/303).

Mailed to Mack today.

8/8/96
DAP

Nopal Water Samples Revisited

Preparations for sending samples of Nopal water out for chemical & isotopic analysis.

Field collection of samples is recounted in notebooks of Murphy (#117, pp. 44-64) and Pickett (#121, pp. 29-34). Conducted August 23-24, 1995.

Pre-field notes in 080/299-300.

A sample and blank were sent to Univ. of Minnesota on 10/30/95. See 080/301.

Inventory of filtered water samples:
Volumes are approximate, estimated visually.

BH12W95-04

UF - 100 ml
UFA - 150
F - 300

UF = unfiltered
UFA = unfiltered + acidified
F = filtered + acidified

BH12W95-05

UF - 90 ml
UFA - 200
* F - 200 - Sent earlier to U. of Minnesota (080/301)
F-2 - 50

BH12W95-11

UF - 100 ml
UFA - ~~400~~ 350 - DAP 8/8/96
F - 400

continued

WNW95-02

UF - 100 ml
UFA - 350
F - 500

WVW95-03

UF - 100 ml
UFA - 400
F - 500

W95-Blank

UF - 100 ml
UFA - 200
* F - 350 sent to U. of Minnesota (080/301)
F-2 - 100

Results of initial analyses at Univ. of Minnesota are shown on opposite page — print-out of an e-mail from John Hoff, dated 3/28/96.

Calculations based on these data are on page 40.

DJP 8/8/96

cc:Mail for: David Pickett

Subject: !st sample and blank
From: "John A. Hoff" <hoffx003@maroon.tc.umn.edu> at internet 03/28/96 11:57 AM
To: David Pickett at CNWRA
To: dpickett@swri.edu at Internet

David:

I just got through reducing the data. In short it is definbately dooable on your waters which are enriched in both 230 and 234 relative to the seawaters I'm used to running. Following are the data:

	230 fg/g	232 pg/g	230/232 ppm	238 ng/g	234 pg/g	del234U
BH12W95	0.311(23)	278.1(1)	1.13(8)	4.754(8)	0.558(1)	1180(3)
typical seawater	2-20 fg/kg	25-100 pg/kg	10-250 ppm	~3 ng/g	~0.2 pg/g	~150

BLANKS:
I got odd numbers here. The 238U blank was high (88pg). Chemistry blanks are typically <5pg. Very little 234U though (<1fg). The 232Th blank was 11pg which is not too bad considering how you had to gather and process samples. However the 230 blank was high (1.4fg). My seawater blanks are typically 3pg for 232 and 0.1 fg for 230. If we proceed we'll have to take a closer look at the blanks.

Given the enrichments in 230 and 234 it looks like no problem at the moment. Let me know what you think.

Cheers, John

JOHN A. HOFF
Minnesota Isotope Lab
Geology & Geophysics
Univ. Minnesota
475 Shepherd Labs
100 Union Street SE
Minneapolis, Mn. 55455
phone: (612) 624-9598
FAX: (612) 625-3819

Calculations of activity ratios in Nepal Waters analyzed at U. of Minn.

BH12 W95-05

① $^{230}Th/^{232}Th = 1.13 \times 10^{-6}$ atom

$(^{230}Th/^{232}Th) = 1.13 \times 10^{-6} \times \frac{9.1952 \times 10^{-6}}{4.9475 \times 10^{-11}} = 0.210$

where (~) means activity ratio.

② $\delta^{234}U = 1180$

$(^{234}U/^{238}U) = \left(\frac{1180}{1000}\right) + 1 = 2.18$

③ $^{230}Th/^{234}U = \frac{0.311 \times 10^{-15}}{0.558 \times 10^{-12}} = 5.573 \times 10^{-4}$ by weight

$(^{230}Th/^{234}U) = 5.573 \times 10^{-4} \times \frac{234.041}{230.033} \times \frac{9.1952 \times 10^{-6}}{2.835 \times 10^{-6}}$

$= 0.00184$

④ $(^{230}Th/^{238}U) = 2.18 \times 0.00184 = 0.00401$

⑤ $(^{238}U/^{232}Th) = \frac{0.210}{0.00401} = 52.4$

⑥ $^{238}U = 4.754 \times 10^{-9}$ g/g

$= 4.754 \times 10^{-6}$ g/kg

$= (4.754 \times 10^{-6})\left(\frac{1}{238.029}\right) = 2.0 \times 10^{-8}$ molal

DAP 8/8/96

⑦ $^{232}Th = 2.781 \times 10^{-10}$ g/g

$= (2.781 \times 10^{-10})(1000)\left(\frac{1}{232.038}\right) = 1.2 \times 10^{-9}$ molal

8/8/96 cont.

W95 - Blank

① $(^{230}Th/^{232}Th) = \frac{1.4 \times 10^{-15}}{11 \times 10^{-12}} \times \frac{232.038}{230.033} \times \frac{9.1952 \times 10^{-6}}{4.9475 \times 10^{-11}}$

$= \cancel{23.9}$ 24 DAP 8/8/96

② $(^{234}U/^{238}U) < \frac{10^{-15}}{88 \times 10^{-12}} \times \frac{238.051}{234.041} \times \frac{2.835 \times 10^{-6}}{1.551 \times 10^{-10}}$

< 0.2 too low! But uncertainty on ^{234}U blank is probably very high.

③ $(^{230}Th/^{234}U) = \frac{1.4 \times 10^{-15}}{< 10^{-15}}$ weight, that is

" (activity) $> 1.4 \times \frac{234}{230} \times \frac{9.1952 \times 10^{-6}}{2.835 \times 10^{-6}}$

> 5 Again, ^{234}U seems too low.

④ $(^{230}Th/^{238}U) = \frac{1.4 \times 10^{-15}}{88 \times 10^{-12}} \times \frac{238.051}{230.033} \times \frac{9.1952 \times 10^{-6}}{1.551 \times 10^{-10}}$

$= 0.98$ Reasonable!

⑤ $(^{238}U/^{232}Th) = \frac{24}{0.98} = 24$

~~⑥ $^{238}U =$~~ DAP 8/8/96

Compare $(^{238}U/^{232}Th)$ in BH12W95-05 to that in Nepal rocks.

According to B. Leslie in the P. robusta paper, the U ore has $(^{238}U/^{232}Th)$ of ~ $30/0.14 = 210$

Water is 4 times lower, suggesting that Th is relatively enriched in water!!

However, BH12 is in area of Level +10 where U is not really enriched.

continued

The most distal sample from the 13 m N fracture (traverse B) has U/Th = 10 ⇒ (238/232) = 31. So, compared to this, U is relatively enriched. 8/22/96 DAP

At location of BH-12 hole, rock U/Th is very likely significantly lower. BH-12 is ~30 m from the edge of the ore deposit. On traverse B, this projects to ~200 ppm U. Assuming 35 ppm Th (P. robusta paper), this gives (238/232) = 18.

On traverse E at 30 m distance, (238/232) is around ~~volcanic rock values, i.e.~~ DAP 8/8/96
1.4

Therefore BH-12 water is enriched in U relative to Th, as expected.
However, ^{230}Th and ^{232}Th are still higher than normal. Colloids?

DAP 8/22/96

Infiltrating Waters Collected in Nopal I Adit Last Fall (1995)

Water collection system was constructed in the Level +00 adit during field work at Nopal I Aug. 23 & 24, 1995. Set up by J. Prikryl. Idea was to collect waters which have travelled down fractures after storms, from the level +10 surface ~8 m above. Plastic sheets were hung from the adit ceiling, leading to filtered funnels feeding a tube into a 1 liter bottle.
During September 1995, several storms supplied water to the collection system.

On the following two pages is a copy of a fax from Ignacio Reyes (IR), who collected the bottles and observed conditions in the adit after the storms.

Bottles have been sitting in the lab since October 1995, unopened. All have some particulates which have settled out.

I will give them new sample numbers, using the numbers already on the bottles: 3, 4, 6, 8, 9. See IR's diagram.

Prefix for samples is ADIT95-__.

So, now have: ADIT95-3
ADIT95-4
ADIT95-6
ADIT95-8
ADIT95-9

All are one liter.

Inserted 8/22/96 DAP

OCT- 9-95 MON 12:44 FAC DE INGENIERIA UACH 91 14 135055 P.01

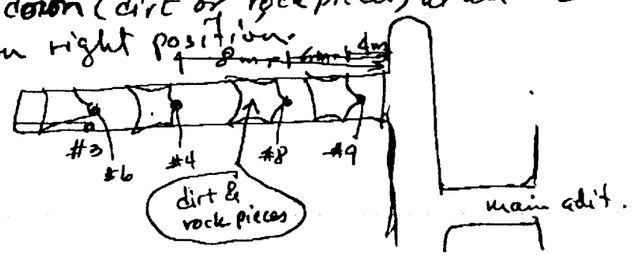
Oct. 9 of 1995

To Dr. David A. Pickett. CNWRA.
fax 210/522-5184
from Prof. Ignacio A. Reyes UACH
fax 011 5214 135055

RECEIVED 9 1995

David,

The bottle sequence from the dead end to the exit is
#3 at the very bottom, which collected water from the wall. It does not show any solid particulates, the filter was fine. It was overflowed the funnel was in position oky.
#6 at the bottom from the ceiling. It does not show any dirt, everything is wet, ceiling walls and plastic collector. It was overflowed and filter handled fine with water.
4 It is located at 8 m from the exit (crossing with the other adit.) It is from the ceiling. the plastic holds some dirt and rock pieces from the ceiling. The filter is out of its right position. It looks like when it got wet it moves a little bit and let dirt go within the bottle.
#8 It is located at 6 m from the exit. The plastic that colect water from the ceiling had lots of dirt and rock pieces, even within the filter had more than 100 gr of dirt. The filter itself was moved when it becomes wet and let dirt goes inside the bottle. Nevertheless, there is not enough dirt inside the bottle.
#9.. it is located at 4 m from the exit. Here there is no fall down (dirt or rock pieces) at all. The bottle and filter is on right position.



OCT- 9-95 MON 12:45 FAC DE INGENIERIA UACH 91 14 135055 P.02

I went to El Nopal 4 days after the last storm and there were no ponded water at the +10 level. There were 3 thunder storms during a 10 days period. all of them had more than 15 minutes rain and make run offs along the arroyos and cuestas. The second thunder storm let the arroyo la venada flow and overflow on the road from El Nopal to Aldama, and it destoys parts of the road. particularly where the road crosses the arroyo. After these three storms had been 4 more little rains which do not cause produce run offs at all.
The storms that produce filtrations toward the adit fall down during Sept 13 to - Sept. 23.. the water was picked up on Sep. 26. and sended to you on Oct. 2nd.
The total rain of the month (Sept) according to the aldama monitoring center was 155 mm.
all of the bottles were overflowed. and one interesting thing is that the plastic curtain avoid any evaporation and lost of humidity so everything inside of the curtain was wet, muddy, meanwhile outside of that plastic barrier was wet but no that muddy.
I hope I can be there in december.

Best regards -
Nacho -

Will now filter ADIT95-6 and ADIT-9 for U/Th isotopic analysis.
Chosen because ① they are from opposite ends of the drift, ② they did not have large amounts of debris on the plastic sheets, and ③ they have less particulate material than others.

Shake bottles. Do this so that water and sediments can re-interact. Sediments may have removed U, Th, others from solution. This shaking does not ensure that they will be redissolved, but it does approximately re-create the initial condition as best as can be done.

Will filter using procedure described in notebooks 121/29-34 (DAP) and 117/44-64 (WMM).

One significant difference: different syringe filters. I am now using Supor Acrodisc 25 filters, with 0.2 μm pore size.
Pore size is same.

ADIT95-9-F

Filtered water,
empty bottle = 55.09 g Scale serial # = 120461

+ total filtered sample = 573.35

add 0.5 ml conc HNO_3 (Mallincrodt select) = 574.12

ADIT95-9-F2 aliquot of ADIT95-9-F after acidification.
empty 125 ml bottle = 19.10 g
+ water 122.34

Final wt. ADIT95-9-F + bottle = 470.90

Weights of solutions:

ADIT95-9-F = 470.90 - 55.09 = 415.81 g

ADIT95-9-F2 = 122.34 - 19.10 = 103.24 g

ADIT95-9-UF poured ~60 ml into 60ml bottle from ADIT95-9 bottle during filtration of ADIT95-9-F.
So, this is unacidified, unfiltered.

ADIT95-6-F filtered
empty 500 ml bottle = 54.96 g

Filters are clogging too frequently. Let settle for ~~a couple~~ 3½ hours.
DAP 8/22/91

Doesn't help. Filters still getting intolerably slow after only ~10 ml passed.

Filter 500 ml of this water with 10 μm mesh "Spectra/Mesh" filter in a funnel. Pours through quickly.
Take this pre-filtered water and use syringe filters (0.2 μm) – Still slow!
Try 0.45 μm sfca syringe filters. Still slow!
Stop filtering after ~250 ml.

wt bottle + total filtered 323.01 g
add 0.25 ml conc HNO_3 323.39

ADIT95-6-F2 Aliquot of ADIT95-6-F
empty 125 ml bottle = 18.95 g
+ water 69.65

Final wt. AD1T95-6-F = 272.66 g
(bottle + soln)

So, weights of solutions:

AD1T95-6-F = 272.66 − 54.96 = 217.70 g
AD1T95-6-F2

AD1T95-6-UF Poured from water which was filtered w/ 10 μm filter in funnel.

Aliquot two other samples for U/Th work.

BH12W95-11-F

Initial total wt soln + bottle = 463.70 g

BH12W95-11-F2 aliquot from ...-11-F
empty 125 ml bottle 18.95
+ sample 115.35

BH12W95-11-F
final wt = 367.32 g
wt soln using empty wt from 080/300 (bottle #30)
= 367.32 − 54.97 = 312.35 g

WVW95-03-F
initial total wt soln + bottle = 527.25 g

WVW95-03-F2 aliquot
empty bottle = 19.24
+ water 116.54

WVW95-03-F final wt = 429.95
wt soln using bottle #31 empty (080/300)

cont.
= 429.95 − 54.78 = 375.17 g

Mail to John Hoff, University of Minnesota:

1. AD1T95-6-F
2. AD1T95-9-F
3. BH12W95-11-F
~~BH~~ DAP 8/27/96
4. WVW95-03-F

8/27/96 Make aliquots of two filtered samples:
DAP

BH12W95-04F-2
empty bottle = 11.45 g
+ water = 60.94

BH12W95-04F final weight = 295.77 g

WVW95-02F-2
empty bottle = 11.49 g
+ water = 60.61 g

WVW95-02F final wt. = 470.34 g

Bottles to be taken to Mike Dammam of Div. 1 for full analyses:

AD1T95-6-F2
AD1T95-6-UF
AD1T95-9-F2
AD1T95-9-UF
W95-Blank-F-2
W95-Blank-UF

continued

continuation...

BH12W95-04F-2
BH12W95-04UF
BH12W95-05F-2
BH12W95-05UF
BH12W95-11-F2
BH12W95-11UF
WVW95-02F-2
WVW95-02UF
WVW95-03-F2
WVW95-03UF

8/30/96 DAP These 16 samples delivered to Mike Dammann, SwRI Div. 1 for cation/anion determination, pH, conductivity, TDS, and alkalinity.

1/23/97 DAP

1/23/97 DAP

Nopal I Water Chemistry Results

This is a summary of work related to the chemistry of waters collected at Nopal I. Previous work is documented on pages 37-50 of this notebook and in locations referenced therein.

These notes have been compiled from previous work which is not yet documented here.

The purpose of the water chemistry effort is to understand better the potential role of unsaturated and saturated zone groundwaters in radionuclide transport at the Nopal I analog. Chemical data allow modelling of speciation and saturation states, which have implications both for radionuclide transport and for the effect of water/rock interaction on the mineralogy of the host rock.

Chemistry results on the seven water samples sent to Div. 1 ~~for~~ DAP 1/23/97 are documented on two files included on the attached diskette labelled "Nopal water chemistry."

8/95 field water
Div 1 water chem 9/96
) Both Macintosh Excel format

These results were combined, and concentrations calculated as molarity, in the file:
Water chem summary – also Excel

These files include data on:
field: temperature, pH, conductivity, Eh, oxygen, carbon dioxide, alkalinity, hardness.
lab: a large number of cations (from filtered samples) and anions (unfiltered).

EQ3 modeling

Four samples were chosen for analysis of chemistry results:

BH12W95-05
WVW95-03
ADIT95-06
ADIT95-09

These were chosen by virtue of Eh data being available. Also – other BH12 and WVW samples were not calculated because chemistry results did not vary significantly among samples from the same locations. Would have been redundant.

EQ3 was run on Gordon Wittmeyer's Sun ("dopey") via eXodus 6.1 from David Pickett's Macintosh.

Input species were:

redox – Use field-measured $\log f_{O_2}$ for BH12W95-05 and WVW95-03; use value for air (-0.68) for ADIT samples, for which ~~not~~ DAP 1/23/97 no field data exist.

H^+ – Use field-measured pH for BH12W95-05 and WVW95-03; use pH = 7 for ADIT samples.

HCO_3^- – Use field alkalinity for BH12... + WVW95..., and lab alkalinity for ADIT...

In both cases, alkalinity was reported as mg/L $CaCO_3$. Following Hem (1970), this was converted to mg/L HCO_3^- by dividing by 0.8202, then to molar HCO_3^- using 61.0171 g/mole.

Temperature: Use field T for BH12... + WVW; use BH-12 field average (26°C) for ADIT...

Cl^-, F^-, NO_3^-, HPO_4^-, SO_4^{2-}, Al^{3+}, Ba^{2+}, $B(OH)_3(aq)$, Ca^{2+}, Cd^+, Li^+, Mg^{2+}, MoO_4^{2-}, K^+, $SiO_2(aq)$, Na^+, Sr^{2+}, V^{3+}, Zn^{2+}, ~~UO_2^{2+}, Th^{4+}~~ DAP 1/23/97 from Div. 1 analyses.

UO_2^{2+}, Th^{4+} – from Univ. of Minnesota analyses.

Fe^{3+} – set $Fe(OH)_3$ as present. Fe was not detected in any lab analyses.

Ca^{2+} was allowed to vary to get charge balance. This was used as a test for completeness/quality of the analyses. eg tests if HCO_3^- is measured accurately.

The EQ3 output files were saved as Mac WordPerfect files and are included on the attached "Nopal water chemistry" diskette.

General Remarks:

BH12W95-05

Balancing on Ca^{++}: from 105 to 82.7 mg/L. Not bad.

Minerals: Silicate phases tend to be oversaturated, including quartz, clinoptilolite, montmorillonite, kaolinite.

Calcite: right near saturation. Consistent with setting.

U phases are all well below saturation. Soddyite has the least negative Log Q/K of -2.8.

Th – thorianite is very supersaturated.

In "bh12o5si.3i" run, set quartz present to hold down Si, and set thorianite present to get a sense of Th oversaturation.

Fewer silicate phases are supersaturated. ~~Proba~~ DAP 1/23/97

Does not change the overall picture much.

Th is held to 1.9×10^{-13} m, contrasted with measured 1.2×10^{-9} m. Probably due to colloids. Bill Murphy believes Si oversaturation is more likely due to kinetics.

WVW95-03 (from regional carbonate aquifer).
Balancing on Ca^{++}: change from 32.2 to 31.2 mg/L.
Excellent.
Minerals: Silicates again supersaturated, though not to same degree as in BH12. Quartz is merely "saturated."
Calcite is very near perfect for saturation. Good.
U is more below saturation than was BH-12.
(Soddyite log Q/K = -4.8)
Th - Thorianite is again supersaturated.

Setting Thorianite present gives Th = 9.0×10^{-15} m, contrasted with measured 6.1×10^{-13} m.

ADIT95-06
Balancing on Ca^{++}: Change from 21.3 to 17.4 mg/L.
Minerals: Level of Si supersaturation is lower than in BH12 and WVW, but still is the case. (Quartz log Q/K = +0.96).
Calcite unsaturated. Consistent with lack of equilibrium in rapidly moving waters. Perhaps did not see caliche, other calcite.
U - well under saturation. It is notable that U speciation has a higher percentage of neutral species than in BH12 and WVW (41% rather than essentially zero). Probably due to low carbonate.
Th - Thorianite supersatured.

Setting Thorianite present gives Th = 7.2×10^{-15} m, contrasted with measured 1.9×10^{-12} m.

ADIT95-06 generally much higher in all constituents than ADIT95-09.

ADIT95-09
Balancing on Ca^{++}: change from 8.3 to 7.2 mg/L.
Minerals: only significant supersatured silicates are illite and kaolinite. Mn and Al seem to be high.
Calcite unsaturated.
U dominated by neutral species (89%). Well below saturation.
Th: Thorianite again supersatured.

Setting Thorianite present gives Th = 7.2×10^{-15} m, contrasted with measured 5.3×10^{-12} m.

General Summary:

In all waters, there is much capacity for more U. [U] is always well below any saturation control.
Th is always well above saturation, especially in BH-12. Probably colloid-related; BH-12 water is "dirtiest."
Si is nearly always supersaturated. Difficult to use silicates in any modelling scheme because of kinetic effects. There is probably also a colloidal component, given that ADIT95-06 is also > Quartz saturation.

Chemical analyses have done a pretty good job of representing reality, it appears. The necessity of lowering Ca^{++} to get balance in each case suggests that bicarbonate may be underestimated, in general.

Water U/Th data

Obtained by TIMS at Univ. of Minnesota.
File "Minn U/Th data" is included on the "Nopal Water Chemistry" diskette. Condensed version on next page.

BH12W95-05 and -11 are very similar to each other in isotopic characteristics: $^{234}U/^{238}U$ = 2.2 for each (activity ratio).
As discussed on pp 39-42 Th/U ratios are high, but U is still enriched in waters relative to Th when compared with likely host rock.

ADIT95-06 and -09
Marked by high $^{234}U/^{238}U$ = 5.1 and 2.8, respectively.
But low [U].
It makes sense that U is higher in 9 than in 6, because 9 is from closer to the ore body.
See pp 44-45. This despite lower concentrations of other dissolved constituents in 9.
Much lower Th/U than BH-12
The high mobility of ^{230}Th relative to ^{232}Th in rapid processes is shown by high $^{230}Th/^{232}Th$ ratios.

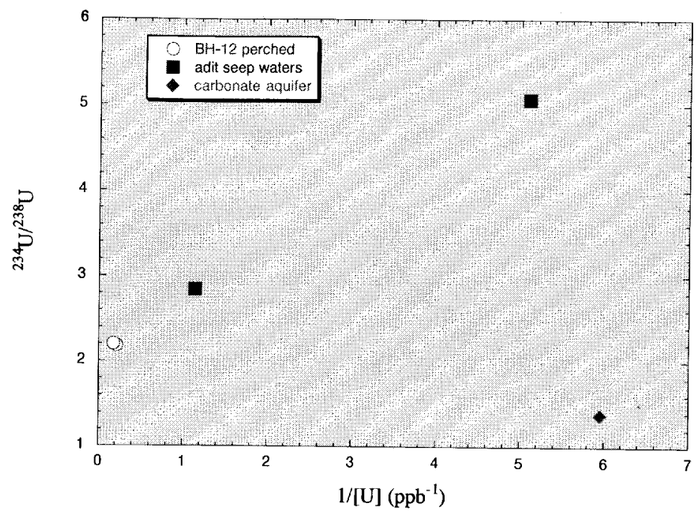
WVW95-03
Relatively low $^{234}U/^{238}U$, low [U].
Low $^{230}Th/^{234}U$ ~~and low [Th]~~ DAP 1/23/97
suggests that colloidal transport of Th isotopes is not important here in regional aquifer.
[Th] is also low, but still above solubility (p. 54).

sample	^{230}Th (fg/g)	^{232}Th (pg/g)	($^{230}Th/^{232}Th$)	molal Th	^{234}U (fg/g)	^{238}U (ng/g)
BH-12W95-05F	0.3004 ± 0.0222	269.21 ± 2.35	0.21	1.16E-09	557.7 ± 1.3	4.7543 ± 0.0084
BH-12W95-11F	0.2318 ± 0.0243	156.42 ± 0.80	0.28	6.74E-10	679.4 ± 2.0	5.7329 ± 0.0146
Adit95-6F	0.0361 ± 0.0040	0.4412 ± 0.0261	15.34	1.90E-12	53.35 ± 1.50	0.1955 ± 0.0055
Adit95-9F	0.4285 ± 0.0113	1.231 ± 0.018	65.26	5.31E-12	134.51 ± 0.88	0.8789 ± 0.0057
WVW95-03F	0.00279 ± 0.00197	0.1404 ± 0.0143	3.73	6.05E-13	12.54 ± 0.41	0.1684 ± 0.0055

Th DAP 1/23/97

	molal U	$d^{234}U$ present	($^{234}U/^{238}U$)	$[^{230}Th/^{238}U]_{act}$	($^{238}U/^{232}Th$)
BH-12W95-05F	2.01E-08	1180.3 ± 3.2	2.18	0.003880 ± 0.000290	53.92
BH-12W95-11F	2.43E-08	1202.9 ± 3.2	2.20	0.002480 ± 0.000260	112.02
Adit95-6F	8.27E-10	4071.5 ± 7.9	5.07	0.011322 ± 0.001263	1354.86
Adit95-9F	3.72E-09	1844.9 ± 2.8	2.84	0.029906 ± 0.000789	2182.12
WVW95-03F	7.13E-10	384.5 ± 2.8	1.38	0.001017 ± 0.000717	3663.20

	$[^{230}Th/^{234}U]_{act}$
BH-12W95-05F	0.001778 ± 0.000128
BH-12W95-11F	0.001126 ± 0.000118
Adit95-6F	0.002232 ± 0.000249
Adit95-9F	0.010512 ± 0.000277
WVW95-03F	0.000734 ± 0.000518



The trend in the BH-12 and ADIT waters shown above suggests the effects of progressive leaching to the lower left. Rapid leaching could give the high-$^{234}U/^{238}U$, low-U point.

The WVW ("carbonate aquifer") point appears unrelated to the others. No apparent link between U transport at Nopal I and in the regional aquifer.

General Comment - Large ^{234}U enrichments are possible from unsaturated-zone U transport.

Uranophane U/Pb Data

1/27/97 DAP

This is a summary of previous work related to U/Pb age dating of uranophane from Nopal I. The objective was to determine the timing of uraninite oxidation..

For previous notebook notations: see pp 18-24 and 36 of this notebook, and citations therein

As discussed previously, over a period of several months samples of uranophane scraped from Nopal I ore deposit rocks were sent to Larry Mack at the Univ. of Texas at Austin for U/Pb analysis by mass spectrometry.

The following is a record of e-mail discussions between Mack and DAP concerning the U/Pb work.

DAP 1/27/97

Subject: U-Pb data
From: LMack75952@aol.com at internet 02/25/96 9:20 PM
To: David Pickett at CNWRA
To: dpickett@swri.edu at Internet

David

Refers to NDPI-494-UPb-2
DAP 1/27/97

I analysed the first sample today.

206/204 = 33.07
207/204 = 16.26
208/204 = 38.59

Not terribly radiogenic. In order to determine an age, we will need to: 1) try a chemical leach to see whether we can eliminate some common Pb; or 2) try to clean the sample up physically before digestion (there was some crud in this sample). If these approaches don't yield a more radiogenic composition, we'll have to try to construct an isochron by: 1) analysing different separates (from the same or different samples); or analysing different minerals which formed at the same time. Got any ideas?

I have the data to determine U and Pb concentrations, but haven't done the calculations yet. I'll send them down soon.

Larry

Subject: ages
From: LMack75952@aol.com at internet 02/28/96 10:22 PM
To: David Pickett at CNWRA
To: dpickett@swri.edu at Internet

David

The first sample contained 597,700 ppm U (60 weight % -- WOW!), and 2079 ppm Pb. (If I've done the calculations correctly -- so don't publish yet!) The Pb seems awfully high, considering that most of it is common Pb. Using these data and the ratios which I sent you before, model age calculations come out at 4 Ma or younger - depending upon assumed initial ratios. This, of course, assumes a closed system for U-Pb.

On a different subject: Has there been anything published on the geology of this deposit? I'd like to know a little about the area. Could you send me a reference?

Larry

Subject: good news
From: LMack75952@aol.com at internet 03/10/96 12:56 PM
To: David Pickett at CNWRA
To: dpickett@swri.edu at Internet

David

1. Thanks for sending references.

2. New data from first sample. I leached ca. 3 mg in 0.2 N NH4OAc; 3 treatments, about 5 mins. each, then rinsed in water. On a second fraction, I wanted to hand-pick uranophane crystals, but I didn't have appropriate tools for material that is this fine grained. So wound up just slurping up "clean-looking" piles of crystals from material that I spread around a watch glass under an alcohol-water mix. Then rinsed in water.

data for "leached": 206/204=38.80; 207/4=16.51; 208/4=38.58

data for "picked": 206/4=342.87; 207/4=30.07; 208/4=38.61

Leaching helped a little, but picking helped a lot. Note that the 208/204 ratios from these 2 samples + the first analysis are identical within uncertainty. Evidently no thoragenic 208Pb moving around. Can't do any geochronology with new data because I didn't determine U/Pb concentrations.

Next step? Try to improve the "picking" technique and work on the same sample again? Let me know. Eric James will be working in the Pb lab for next 1-2 weeks, so we're on hold for a while.

Larry

Subject: Re: good news
From: David Pickett 03/14/96 1:44 PM
To: LMack75952@aol.com at internet

Larry,

Thanks for the new info. You wrote:

>Next step? Try to improve the "picking" technique and work on the same sample again?<

Sounds good. Did you just not have enough to take a U/Pb concentration split? To state the obvious, it wouldn't be worth doing another analysis of the same sample unless you can get a U/Pb ratio, and thus an apparent age, out of it. I'm not sure what you mean by "improving" the technique, but go ahead.

I'm getting tempted to go ahead and send other samples to you. What other info do we need before I should decide to do that?

Subject: Re: good news
From: LMack75952@aol.com at internet 03/15/96 9:39 AM
To: David Pickett at CNWRA
To: dpickett@swri.edu at Internet

David

1. The reason that I didn't do complete concentration + ratio analyses last time is that this requires about an order of magnitude more work (which would have been wasted if there were no change in Pb ratios). In hindsight -- it would have been worthwhile for the "hand-picked" sample.

2. OK -- I'll go ahead and re-analyse another split of the first sample. By "improving the technique", I meant that the technique that I used is not as efficient (in terms of obtaining a pure separate) as hand-picking discrete crystals would be. I'm sure that I "slurped up" some other crud along with the uranophane.

3. The only reason that I can see to not begin on other samples is that we haven't established that the uranophane has been a closed system, i.e. that it will yield a reliable radiometric age. What about trying to obtain a co-genetic, non-U-bearing phase for this first sample to try to put some limits on initial Pb ratios?

Larry

Subject: new data
From: LMack75952@aol.com at internet 04/12/96 12:15 PM
To: David Pickett at CNWRA
To: dpickett@swri.edu at Internet

David

The second attempt on sample #1 gave the following data:

206/204 = 62.1863 (cf. 33.07 for try #1)
207/204 = 17.5396 (cf. 16.26)
238/204 = 78997 (cf. 22000)
235/204 = 572.94 (cf. 160).

Two-point isochrons give following "ages" and initial ratios:
for 238-206: age=3.29 Ma; initial 206/204=21.78
for 235-207: age=3.14 Ma; initial 207/204=15.77

Ideally, both ages should be identical. The difference may be due to a common-Pb-bearing phase whose Pb ratios are different than the initial Pb ratios in the uranophane.

Larry

Subject: new data
From: LMack75952@aol.com at Internet 05/05/96 9:40 AM
To: David Pickett at CNWRA
To: dpickett@swri.edu at Internet

David

Finally, some good news. Two new analyses of hand-picked grains yielded 206/204 ratios of 995 and 695, and U/Pb ratios that were higher by a factor of 20 compared to earlier analyses. Model ages still at 3 Ma.

Having now gotten the common Pb problem under control, can begin to address question of possible open-system Pb loss (leading to erroneously young ages). Thought I might try to compare coarser vs. finer-grained uranophane.

Larry

Subject: Re[2]: Newest U/Pb data
From: David Pickett 10/11/96 9:24 AM
To: LMack75952@aol.com at Internet

Larry,

Well, my boss managed to arrange for some money so I can spend time on writing up the data (along with some other stuff). I will have to complete something by Nov or Dec, and then that charge code will be gone forever!

I've been playing with the numbers a little. The five 494 points sure do make nice 206/238 (3.2 Ma) and 207/235 (3.1 Ma) isochrons, and the two other samples also fall on them. It still seems to me that the spread in U/Pb in the five 494 fractions obviates the need for a non-radiogenic phase.

Two wrinkles (mentioned by you, as well):

1. The 6/4 intercept is 26.1, which seems high. But maybe not, considering the setting? 7/4 intercept is 15.99, which is not so high. I guess I should calculate model ages for these ratios given reasonable initials and U contents.

If they gave the same age, then it would seem correct to attribute the high initials to U-rich precursors. I'll start these calculations. (I guess item #2 below suggests this won't quite work.)

2. The future Pb/Pb age. The slope of the sample 494 7/4 vs 6/4 line is 0.0443, lower than the zero-age value of 0.04605. This is a very tight line, with R of 0.99995, and the other two samples fall right on it. I don't know enough about U/Pb systematics to speculate much on the possible cause of this future age.

David

Note: the letter reporting these data was not located. At any rate, they are superceded by later data.
DMP 1/27/97

Subject: Re: Re[2]: Newest U/Pb data
From: LMack75952@aol.com at Internet 10/23/96 10:24 PM
To: David Pickett at CNWRA
To: dpickett@swri.edu at Internet

Hi David,

Here are the numbers that I'm getting (different from what you e-mailed me -- probably because I'm now using a slightly different value for U concentration in the spike [this is something that is still not resolved]):

206/204-238/204: age=3.35 Ma, init.206/204= 22.639
207/204-235/204: age=3.32 Ma, init 207/204= 15.821

This is using all data. If I use juse the 494 data:
206 age=3.35 Ma, init.=22.226
207 age=3.22 Ma, init=15.811

If I use all the data except the analysis that I've called 494 try 3-2:
206 age=3.46 Ma, init.=22.510
207 age=3.34 Ma, init.=15.814

The latter option gives better-fitting "isochrons".

I don't have an answer yet for: 1) the consistently higher age for 238-206 compared to 235-207; 2) the future 207*/206* age. If the sample loses daughters, the 238-206 age is supposed to become younger than 235-207 (238 has longer-lived mobile daughters), and the 207*/206* age can become too old. This is not what we see here.

Another problem that is still unresolved: do we have isochrons or just mixing lines? There is some evidence for the latter. The samples that have the lowest 206/204 ratios (of ca. 30) have a lot of Pb -- over 2000 ppm in 494 #1. Because this sample has ca. 600,000 ppm U, which is close to the stoichiometric value for uranophane, this suggests that there is some trace phase in which Pb may be a major element. If this phase has the same initial Pb ratios as does the uranophane, then no problem, but if it formed at a different time with different ratios, then our "ages" may be meaningless.

I'll try to get away and call you tomorrow (Thursday).

Larry

ect: Re[4]: Newest U/Pb data
om: David Pickett 10/24/96 12:17 AM
To: LMack75952@aol.com at Internet

Larry,

Thanks for your note. Are you able to send me the newest version of the data? If you can't attach an Excel file to e-mail (you should be able to), then just insert the new U concentrations, if that's all that's different, in the text of an e-mail. I also would like some error bars at some point. At this late hour (I have to finish prepping my poster tomorrow), I'm going to leave the numbers as I have them, throw up the word "preliminary" on the plots, and include discussion of the caveats. Besides, 3.4 vs 3.1 Ma is unimportant for the poster.

By the way, a secondary phase (jarosite) at another U deposit a few km away has a K/Ar 3.6 Ma age. For whatever that's worth....

Regarding Pb/Pb age, uncertainties might be important here. It's hard to tell how significantly "future" 0.0443 is. Could there be anything in your correction procedures here?

You ask: "do we have isochrons or just mixing lines?"

I'm sure that, as you say, we have mixing lines, which could nevertheless give ages. The Fe oxides that also line the fractures on these rocks (chiefly as a substrate to the uranophane) could have quite high Pb contents. We have measured up to 4000 ppm in them elsewhere at the site. In fact, we think pyrite was a precursor to the Fe oxides, so it's not surprising that there's a fair amount of Pb around. Did your treatment of those two high-Pb samples (494 #1 and 291) have something in common which could have led to leaching of Pb from Fe-rich grains?

OK - as I see it, here's a scenario that makes our ages meaningless (well, not completely...see below): Some mineralization event (probably oxidizing) gives you a U phase and some Pb-rich Fe oxides. A few or several Ma pass, and the U phase gets high 6/4 and 7/4. U is remobilized and a new U phase is deposited, but there is no mixing between the Pb in the Fe oxides and the highly radiogenic initial Pb incorporated by the new U phase. So, the U phase we are measuring is actually younger than 3.X Ma, with higher initial Pb ratios. So, as I was alluding to above, the age does actually have some meaning as a max.

Did I miss something here? It _is_ late.

I did mixing plots (1/Pb vs 6/4 and 7/4) and got a _sorta_ linear-looking array EXCEPT for that low-U sample, which is also low-Pb. Three components? Ugh.

I guess I am still biased against the possibility of non-Pb isotope equilibration with Fe phases on U mineralization. After all, if it's that easy to leach...OK, maybe you didn't leach out the Pb. My geochem colleague here

might have some feelings on the matter, e.g., how closed Fe phases could have been. The problem is, I don't really know how we can test this without getting our hands on a phase that we know was exactly cogenetic with the uranophane. Again, am I missing something here?

Are we just going to be stuck with the max age constraint? It looks like we're going to have to sit down with all the info in one place and hash it out.

As it happens, I'm coming to Austin for a conference Nov 4-5. Should we think about a pow-wow?

Enough rambling for now. Hope to hear from you tomorrow (Thur).

Thanks,

David

1/28/97 DAP On 11/5/96, DAP visited with Mack in Austin, and further discussions took place.

DAP obtained a file of the U/Pb data. It is included in the attached diskette titled "Uranophane U/Pb." Name of Mack's original file is "SRI urphane new spk." ~~edited.~~ DAP 1/28/97
DAP edited and condensed this file and named it "SRI urphane new spk edited."
The diskette also contains a KaleidaGraph data file and three plots: "new 7/4 vs 5/4 isochr," "new 6/4 vs 8/4 isochr," and "Pb/Pb isochron all."

On the following three pages are attached copies of the first three pages of Mack's printout of "SRI urphane new spk" with DAP's handwritten notes of Mack's verbal descriptions of the different uranophane fractions, and of uncertainties

Sheet1

Sample ID	Date Run	Sample Wt. gms	Spike Wt. gms	235/238 measured	208/206 meas. ID run
SWRI #1 U	25-Feb-96	1.424E-06	1.0982	0.1388	
SWRI #1 Pb	25-Feb-96	0.00056973	0.2594		1.7035
SWRI #1 try 2 U	30-Mar-96	7.7953E-07	1.6464	2.70385	
SWRI #1 try 2 Pb	30-Mar-96	0.00089547	0.205		4.49318
SWRI #1 try 3-1 U	4-May-96	8.954E-07	1.5064	0.32079	
3-1 Pb	4-May-96	0.00005034	0.0227		1.6975
SWRI #1 try 3-2 U	4-May-96	1.0071E-06	1.5347	0.29764	
3-2 Pb	4-May-96	0.00006326	0.0204		1.3899
SWRI-494 try 4 U	31-Aug-96	2.3234E-07	1.1741	0.80928	
SWRI-494 try 4 Pb	31-Aug-96	0.00030172	0.1095		1.16698
SWRI 291 U	31-Aug-96	5.4909E-07	2.1171	0.73548	
SWRI 291 Pb	31-Aug-96	0.0017946	0.7941		1.900919
SWRI 102 U	31-Aug-96	2.2373E-07	1.4838	1.22971	
SWRI 102 Pb	31-Aug-96	0.00072216	0.2222		1.23247

est.

[U] ± 2 % 2σ

(weighing errors)

U/Pb ~ ± 2 %

Pb isotopes ~ ±0.1 %

(fractionation corr. uncert.)

→ * Pb/Pb - ±0.1 %

Pb 206/204, 207/204

ion counter uncert.

?? 0.5 % ??

Sheet1

206/204 meas. IC run	207/204 meas. IC run	208/204 meas. IC run	cor 206/204	cor 207/204	cor 208/204	
33.0247	16.2264	38.4849	33.0725253	16.2616479	38.5963653	494 try 1
			#DIV/0!	#DIV/0!	#DIV/0!	
			#DIV/0!	#DIV/0!	#DIV/0!	
62.00717	17.46386	38.32112	62.1863478	17.5395561	38.5425877	494 try 2
			#DIV/0!	#DIV/0!	#DIV/0!	
			#DIV/0!	#DIV/0!	#DIV/0!	
992.1	58.693	37.54	994.711242	58.924723	37.7376132	494 try 3-1
			#DIV/0!	#DIV/0!	#DIV/0!	
			#DIV/0!	#DIV/0!	#DIV/0!	
693.6	45.151	37.56	695.425579	45.3292585	37.7577185	494 try 3-2
			#DIV/0!	#DIV/0!	#DIV/0!	
			#DIV/0!	#DIV/0!	#DIV/0!	
546.335	39.2117	38.454	547.57826	39.3455474	38.6290146	494 try 4
			#DIV/0!	#DIV/0!	#DIV/0!	
			#DIV/0!	#DIV/0!	#DIV/0!	
32.4533	16.19749	38.40096	32.5271519	16.2527794	38.5757332	291
			#DIV/0!	#DIV/0!	#DIV/0!	
			#DIV/0!	#DIV/0!	#DIV/0!	
122.2225	20.15907	38.3997	122.500634	20.2278821	38.5744675	102

494 Uph (all from same batch)

try #1 => as rec'd.

try #2 => attempt to hand-pick non-uph, cursory

try #3 => hand-pick uph

try 4 = best of hand-picked uph throwing out fines
→ coarser xtalline uph.

3-1 : fell out of ref bkr after acetone wash & dry (tapping)

3-2 : stayed in bkr. Finer (?). off isochron.

1+2 - washed in H_2O, picked in ethanol

3+4 - picked in ethanol, washed in acetone, then H_2O

291, 102 - " " "

Sheet1

No of stds run	206/204	207/204	208/204	208/206	206/204	207/204
	NBS 981 #1	NBS 981 #1	NBS 981 #1	NBS 981 #1	NBS 981 #2	NBS 981 #2
2						
2	16.91725	15.46222	36.61132	2.1641316	16.91392	15.45466
1						
1	16.89052	15.4247	36.49523	2.160693		
1						
1	16.8959	15.4318	36.513	2.161034		
1						
1	16.8959	15.4318	36.513	2.161034		
2						
1	16.9018	15.4387	36.53944	2.161924		
2						
1	16.9018	15.4387	36.53944	2.161924		
2						
1	16.9018	15.4387	36.53944	2.161924		

crit.

291 : amorphous coarser stuff, most yellow

102: xtalline. Double picking for best uph. All fine, so not much size separation (omitted powder).

(These descriptions are concerned with Mark's treatment of the samples received from us — not of our treatment.)

The KaleidaGraph plots on the attached "uranophane U/Pb" diskette show the results of linear regressions of the [$^{206}Pb/^{204}Pb$ vs $^{238}U/^{204}Pb$ and $^{207}Pb/^{204}Pb$ vs $^{235}U/^{204}Pb$] data for sample 494. Regressions were done with Kaleidagraph itself.

Results for 494

$^{206}Pb/^{204}Pb$ vs $^{238}U/^{204}Pb$

Slope = 0.00051995 = m

yintercept = 22.2

The equation of this line is

$$\frac{^{206}Pb}{^{204}Pb} = \left(\frac{^{206}Pb}{^{204}Pb}\right)_0 + \frac{^{238}U}{^{204}Pb}\left(\left[e^{\lambda t}\right]-1\right)$$

with $\lambda = 1.551 \times 10^{-10}\ yr^{-1}$

Therefore, the y intercept = initial Pb isotope ratio $\frac{206}{204}$.
= 22.20

and

the age $t = \cancel{\frac{1}{\lambda}(\ln m + 1)} =$ DAP 1/28/97

$= \frac{1}{\lambda}\ln(1+m)$

$= \therefore 3.4$ Ma

$^{207}Pb/^{204}Pb$ vs $^{235}U/^{204}Pb$

Similarly: slope = 0.0031733 $\Rightarrow$ age = 3.2 Ma

$\left(\frac{^{207}Pb}{^{204}Pb}\right)_0 = 15.81$

The other two samples (102 and 291) fall on the same isochrons

If the fractions represented mixtures between uranophane and other phase(s) with low U/Pb, it is possible that the original "slope" of the isochrons would have been more shallow, giving younger ages.

However, the calculated ages, which are reasonably close to one another (3.4 and 3.2 Ma), are considered reliable. This is because it is unlikely that there was no Pb isotope homogenization on the hand-sample scale when the uranophane formed. Therefore, the contaminant phases most likely had the same $^{206}Pb/^{204}Pb$ and $^{207}Pb/^{204}Pb$ ratios as the uranophane when it formed.

Yet to be done:

1. Full uncertainty analysis.

2. Satisfactorily explain the "future" Pb-Pb age given by the "Pb/Pb isochron all" plot slope.

I have reviewed this notebook. It is in general compliance with QAP 001. There is adequate information for another qualified person to repeat the activities.

E.C. Pearcy

1/30/97

E.C. PEARCY

5/17/99
DAP

Nopal Samples to DOE

The DOE has requested 12 samples of rock material from Nopal I.
They asked for samples in the "forbidden zone" on the $^{234}U/^{238}U$ vs $^{230}Th/^{238}U$ plot, and they asked that they span various distances from the ore body.
In addition, they asked for powder splits when possible.

12 samples were selected with these criteria in mind. There are four samples from the 13m N fracture, five from the prominent N-S fracture, one from just off the north edge of the ore body, and two from Level +00.

Regarding the 13m N fracture, I could find no powder splits from materials used previously to measure U-Th ratios well into the "forbidden zone." There are newer powders for several samples with a "-PWD" suffix. These are apparently not the same powders used for the alpha measurements as reported in our U-series paper (Pickett et al., in reviews). However, Jim Prikryl did make alpha measurements on the "-PWD" materials (NOP1-417, -418, -420, -421, -423, -425). In general, these are not as far into the "forbidden zone" as the previous powders. Possible reasons:

1. Systematic error. Analyses were all made in 1995.
2. Powder preparation. Perhaps more older material (e.g., tuff fragments, old fracture lining) was incorporated into the second powder because Jim could not be as selective the second time around.
2. Dissolution. In some cases, a different combination of acids were used to dissolve:

1st set: 10 ml HCl + 0.5 ml HF
2nd set: 3 ml HCl + 7 ml HF + 1 ml HNO_3.

The 1st set acid may have tended to attack preferentially younger, less crystalline material.

Whatever the cause, I see no reason to send "-PWD" powders that are not substantially in the "forbidden zone." Instead, I will send hand samples for most (as before). LANL can separate out Fe oxide materials as they did previously. Another factor is that I do not wish to again send any type of material from NOP1-418, -421, or -425 — these were the three samples sent previously. That leaves only NOP1-417, -420, and -423 among the "-PWD" powders from which to choose. Of these, only NOP1-420-PWD is satisfactorily in the "forbidden zone."
Choose the three hand samples based on (1) "forbidden zone" location and (2) spread along the fracture. Of course, I cannot predict what their data will look like because the fracture fill is clearly heterogeneous.

The northern fracture samples are chosen for their "forbidden zone" nature and for spread along the fracture — out to its greatest extent, 36 m from the ore body. I also preferred a sample if (i) it had higher U and (ii) the sample custody log states that it is fracture fill, rather than tuff.
The Level +00 samples: NOP1-309: forbidden zone; NOP1-320: low $^{230}Th/^{238}U$. These data have not been presented publicly yet.

Powders: Measure out of sample powder vial into a small snap-cap plastic vial (Kartell milano TS 732). Scoop powder with a piece of weighing paper rolled into a scooper. Weigh vial empty, then with powder.

Northern Fracture (Traverse E)

NOPI-403-DOE — from NOPI-403-WR1

vial + powder	5.207
empty vial	3.389
wt. powder	1.818 g

NOPI-404-DOE — from NOPI-404-WR1

vial + powder	5.380
empty	3.420
	1.960

NOPI-407-DOE — from NOPI-407-WR1

vial + powder	5.340
empty	3.418
	1.922

NOPI-410-DOE — from NOPI-410-WR1

vial + powder	5.371
empty	3.391
	1.980

NOPI-411-DOE — from NOPI-411-WR1

vial + powder	5.538
empty	3.390
	2.148

Level +00, SE traverse ("F")

NOPI-309-DOE — from NOPI-309-WR1

vial + powder	5.387
empty vial	3.412
	1.975

NOPI-320-DOE — from NOPI-320-WR1

vial + powder	5.519
empty vial	3.389
	2.130

Northern Short Traverse ("C")

NOPI-376-DOE — from NOPI-376-WR1

vial + powder	6.306
empty vial	3.407
	2.899

13m N Fracture ("B")

NOPI-420-DOE — from NOPI-420-PWD

vial + powder	5.172
empty vial	3.400
	1.772

(see next page)

Hand-samples: Take one or two pieces and place in labelled zip-lock bag.

NOPI-419-DOE	from	NOPI-419	one lg. piece
NOPI-423-DOE	from	NOPI-423	one lg. piece
NOPI-424-DOE	from	NOPI-424	one med, one small

Decide to go ahead & send NOPI-423-PWD split.
It is not clear that the small hand samples I'm sending have very much true fracture-fill Fe oxide. Send this powder so they will definitely have two powders made from material judged by Jim Prikryl to be fracture fill.

8/11/99
DAP

NOPI-423-PWD-DOE — from NOPI-423-PWD (not very much available)

vial + powder	3.963
empty vial	3.410
	0.553

This makes 13 samples, though two are from the same field sample.

5/18/99
DAP

Pack samples. Wrap powder vials with parafilm and place all 10 in zip-lock. Pack with hand samples in box.

Used Ludlum Model 3 Survey Meter to check activity on exterior of zip lock bags (powders and hand samples).
Less than 100 cpm, corresponding to <0.1 MR/hr.

Carbonate and Opal Geochronology

This activity is carried out under the ENFE (Evolution of the Near-Field Environment) KTI, account 20-1402-561.

Activities center around obtaining new age information of secondary deposits that may shed light on the timing of U release at Nopal I.
The statement below was used to justify the work.

Results of previous uranium-series studies at the Nopal I natural analog have suggested that radionuclide release from the uranium deposit is episodic. These and earlier studies have contributed to estimations of release rates at YM. A solid phase present at Nopal I that may be useful for better understanding the absolute timing of uranium release events is calcium carbonate, present (chiefly in fractures) as both caliche and crystalline calcite. Only one U-Th age date of 54 ka exists for caliche, but numerous samples have been obtained and are available for analysis. This proposed task would involve obtaining mass-spectrometric U-Th isotopic analyses from the University of Texas at Austin (UT) on approximately 15 samples and subsamples of Nopal I calcium carbonate to better estimate the timing of U release events and to test for any periodicity or episodicity evident. Mass spectrometry provides better sensitivity and precision than alpha counting, allowing, for example, analysis of discrete mineral layers in fracture fills.

Aside from U-Th ages on carbonates and opals, this work may also involve K-Ar on jarosite and possibly U-Pb on U minerals.

It may also make sense to obtain chemical and stable isotopic data on the sample powders.

To date, I have spent a large amount of time studying the CNWRA sample custody log and sample collection to find appropriate samples. Bret Leslie provided a list of Nopal I caliche, calcite and opal samples, taken from going through the sample custody log.

Using Bret's list, I selected sample powders already prepared of carbonates – and for which separates were made, especially if discriminated by grain size.

My first objective is to prepare a set of powders of carbonates to send to Univ. of Texas (UT) for initial U concentration by ID-TIMS. This set will include multiple grain size fractions for most – or maybe all – of the samples.

I have also selected a set of hand samples from which I will prepare such samples, to complement the previously prepared samples.

Objectives of carbonate sample selection:

- prefer caliches. We expect that these will be younger than the crystalline calcites and so will better reflect recent behavior. The crystalline calcites may even be older than the U-series range in some cases. I still believe it would be useful to get [U] data from a reasonable number of calcites in order to understand U mobilization further back in time.

- use available fractions and powders when possible – to save effort.

- focus on "downhill" samples, but try to get a good spatial spread of samples around the deposit. Among available samples, caliches tend to be from just south of the deposit or from upper levels. This is not surprising, because they tend to form near the ground surface. I do believe it's possible that crystalline calcites

from Level +10 and its walls may yield info on U mobilization.

- try to get cleaner samples. Failing this, use grain size fractions in an isochron approach for ^{230}Th – ^{234}U – ^{238}U dating. ("Clean" means relatively free of non-carbonate grains.)

- See if I can physically separate caliche layers from field samples. See if there is age zoning in caliche layers.

- Take a fraction of each powder and set aside for possible chemical and stable isotopic analysis. Chemistry might be especially interesting for dark calcites (Mn-rich?).

I will first go through the samples for which powders and/or separates have been prepared. I will describe what I have, how I treat them, and any new splits/separates. Included will be a brief description and the rationale for selecting it.

I will use the NOPI-# nomenclature, superseding older sample names. If I am retaining a container with an old sample # (i.e., not simply "NOPI-#") I will mark it with the NOPI-# name.

NOPI-12

Collected 9/91, field notebook 021/25, separate preparation 024/263. Calcite from a veinlet on -100 level.

~~Exist~~ DAP 8/11/99 Selected as representative of three calcite ("cc") samples collected here. Only one of the three for which good separates exist. Unfortunately, I could not find NOPI-12-SEP2.

Existing subsamples:

NOPI-12-SEP1 250 μ size fraction ~1.3 g.
~~NOPI~~ DAP 8/11/99 Was powdered after sieving.
NOPI-12-GAM <63 μ size. 0.08 g. in PP "test tube."

Because there is so little of NOPI-12-GAM, I will not prepare any for [U] determination; i.e., I will save it all in case I want U-Th isotopes.

NOPI-12-SEP1

I will keep this in the vial. Some ~~rep~~ DAP 8/11/99 removed for -B split.

NOPI-12-SEP1-B

A small split for stable isotopes/chem. Use a small glass bottle with a black screw cap. Transfer with spatula.

vial + powder 6.448
empty 6.307 g
0.141 g

Note: Unless otherwise noted, powder weighings are done on an OHAUS Precision Standard Balance, ser # 2883, calibration due 26 Sept 99.

NOPI-12-GAM

Leave as is, for now.

NOPI-19

Collected 9/91, field notebook 021/30, separate preparation 024/263. Calcite vein from Level +00, perhaps a meter or two higher than +00, NE of deposit. Selected as representative of dark calcite from this side of the U deposit.

Existing subsamples gathered:

NOPI-19-SEP1 250 μ, ~10 g, not powdered
NOPI-19-SEP2 63 μ, ~1.5 g, not powdered

NOPI-19-SEP1-P

I want to powder some of this separate for ease of dissolution. Use an agate mortar and pestle (cleaned by crushing some W510 quartz in it, then rinsing with water, nanopure water, and acetone). I will not sieve it or test for grain size; just crush until it appears "powdery". Weigh this powder into a new plastic vial labelled NOPI-19-SEP1-P. Vial is first blown out with "duster." Crush a small amount of sample in mortar + pestle, wipe with kimwipe, wipe with acetone, then blow out. Then crush sample for keeps.

vial + powder 6.352
empty vial 5.116
1.236 g

NOPI-19-SEP1-PB

Small split of NOPI-19-SEP1-P for chem/S.I. (SI = stable isotopes). In small glass vial. Weight above for parent powder is after removing this split.

vial + powder 6.456
empty 6.356
g

NOPI-19-SEP2 Leave as is.

8/12/99
DAP

NOPI-24

Collected 9/91, field notebook 021/32, separate prep 024/263. Calcite veinlet in vitrophyre. 10-15 m WNW of NOPI-19. Selected because it is a white carbonate, in contrast to dark NOPI-19.

Existing subsamples gathered:
NOPI-24-SEP1 250 μ fraction, ~2.5 g, powdered.
-SEP2 63 μ fraction, 0.5 g ?, powdered.
-GAM <63 μ, ~0.1 g

NOPI-24-SEP1 No need to treat. Looks a little dirty. Take some for following sample

NOPI-24-SEP1-B Small split in glass vial for chem/S.I
+powder 6.523
empty vial 6.364
g

NOPI-24-SEP2 - No treatment. Probably won't do I.D. (i.e., probably won't aliquot for U conc. measurement)

NOPI-24-GAM - No treatment. Probably won't do I.D.

NOPI-29

Collected 9/91, field 021/38, separates 024/263. Calcite on slickenside surface ("in "pit"?), middle of Level +10. White calcite representative of this area of Level +10.

Existing subsamples gathered:
NOPI-29-SEP1 250 μ fraction, 1.4 g, powdered
-SEP2 63 μ fraction, <0.5 g, powdered.
-GAM <63 μ. ? g

NOPI-29-SEP1 No need to treat. Take some for following splits.

NOPI-29-SEP1-B In glass vial, for chem/S.I.
+powder 6.497
empty vial 6.370
g

NOPI-29-SEP2 No treatment. Probably won't I.D.

NOPI-29-GAM " " " "

NOPI-164

Collected 9/92, ~~fat~~ DAP 8/13/99, field 042/89, separates 024/255. "Calcite-cemented breccia" in wall of Level +10. Representative of this location. ~~XRD suggests the powdered material is pure $CaCO_3$.~~ DAP 8/13/99

Existing subsamples gathered:
NOPI-164-SEP1 250 μ fraction, 5.7 g, powdered
-SEP2 125 μ fraction, 1.5 g, "
-SEP3 63 μ fraction, 1.2 g, "
-SEP4 <63 μ fraction, 1.1 g, "

NOPI-164-SEP1 No treatment. Take split for following.

NOPI-164-SEP1-B Glass vial, for chem/S.I.
+powder 6.810
empty vial 6.643
g

NOPI-164-SEP2 No treatment. Probably won't I.D.

NOPI-164-SEP3 " "

NOPI-164-SEP4 " "

8/13/99
DAP

~~NOPI-166~~

~~Collected 9/92, field 042/90, separates 024/263.~~
~~DAP 8/13/99~~

~~Decide not to use this sample, because it is very similar to NOPI-164. Originally, I thought it was possibly purer calcite than the NOPI-164 separates. However the notes from 024/255 state that XRD showed the NOPI-164 separate to be quite pure.~~ DAP 8/13/99 Wrong! The notes at 024/255 are referring to NOPI-163.

NOPI-166

Collected 9/92, field 042/90, separates 024/263 (originally NOPI-10-16/4.0). Close to NOPI-164 location, but the NOPI-164 separates were made from a "bulk rock", the calcite purity of which is unknown. The NOPI-166 separates were made from a "calcite separate."

Existing subsamples gathered:

NOPI-166-SEP1	250 μ fraction,	several grams, not powdered
-SEP2	63 μ fraction,	<1 g, " "
-GAM	<63 μ,	small amount (~0.2 g)

NOPI-166-SEP1-P

Make a powder from part of NOPI-166-SEP1. See notes on page 83 for NOPI-19-SEP1-P.

+ powder	6.846
empty vial	5.623
	g

NOPI-166-SEP1-PB Portion of preceding powder for chem/S.I.

+ powder	6.749
empty vial	6.592

NOPI-166-SEP2 No treatment

NOPI-166-GAM No treatment.

NOPI-179

Collected 9/92, field 042/94, separates 024/263. (originally NOPI-20-26/1, though some of the labels on the separates bottles say ~~"NOPI-2-~~ DAP 8/13/99 "NOPI-10-26/1." There is no such sample. Obviously an error. This sample represents dark calcite cutting Nopal tuff higher up - wall of Level 20. Separates are calcite.

Existing subsamples gathered:

NOPI-179-SEP1	250 μ fraction,	a few grams, not powdered
-SEP2	63 μ fraction,	<1 g, " "
-GAM	<63 μ fraction,	0.1 g

NOPI-179-SEP1-P

Powder a portion of NOPI-179-SEP1. See page 83 notes on NOPI-19-SEP1-P.

+ powder	6.127
empty vial	5.319
	0.808
	g

NOPI-179-SEP1-PB Portion for chem/S.I.

+ powder	6.536
empty vial	6.430
	0.106

NOPI-179-SEP2 No treatment

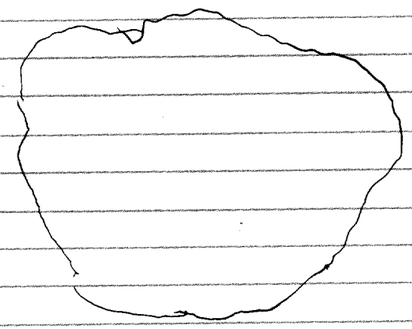
NOPI-179-GAM No treatment

8/16/99
DAP

NOPI-436 "DAP-4" in field.

The field sample consists of a large (~30 cm long) block of tuff with caliche coatings on all faces. I have taken a loose piece of caliche, 24 g in weight, 4.5 x 6.0 cm x ~1 cm thick. I will try to separate two layers from this piece. Failing that, I will prepare the entire piece.

Actual tracing of piece:



One face is light orangish brown. The opposite face is off white with brown.

First try to separate pieces of the 2-3 mm thick layer on the orange side. Use an old Exacto knife as a chisel, and tap it gently along a selvage below the orangish surface layer - parallel to the flatness of the piece. Chip away until I have a few grams of pieces of thickness 1 to 3 mm. Call this:

NOPI-436-L1 L=layer.

Comprises ~ 3/4 of layer of one face. (3/4 of plan area in drawing above). total 3.4 g.

Sieve: Use a set of sieves: Dual Mfg. Co, U.S. Standard Sieve Series, ASTM E-11 Specifications, 250 μ, 125 μ, 63 μ.

But first, crush layer chips in a large agate mortar and pestle until no grains are larger than approx sand size. Combine in a weighing boat. Pour into 250 μ sieve stacked on top of 125 μ + 63 μ + bottom holder.

250 μ fraction is 2.0 g. 125 μ is 1+ g. I did not crush fine enough. Very little is on 63 μ sieve or bottom. Take the 250 μ + 125 μ fractions and crush more in mortar + pestle.

Transfer fractions to small glass vials via weighing paper.

NOPI-436-L1-SEP1 250 μ fraction (i.e., >250 μm)

+ powder	7.446
empty vial	6.600
	0.846 g

NOPI-436-L1-SEP2 125 μ fraction (i.e. 125 < x < 250)

+ powder	7.792
empty vial	6.846
	0.946

NOPI-436-L1-SEP3 63 μ fraction (i.e., 63 < x < 125)

+ powder	7.232
empty vial	6.392
	0.840

This fraction was crushed after the first sieving, because not much was in the bottom holder (<63 μ). The additional crushing resulted in better distribution. The weight above is final.

NOPI-436-L1-SEP4 <63 μ fraction

See above. Contains materials from initial sieving, then additional crushing of DAP 8/16/99 and sieving of 63 μ fraction.

	6.908
empty vial	6.303
	0.605

NOPI-436-L1-SEP2-B Small aliquot of NOPI-436-L1-SEP2 for chem/S.I.

	6.487
empty vial	6.345
	0.142

NOPI-436-L2

Sample from opposite side of the NOPI-436 piece introduced on page 88. The "off white with brown" face. Again use Exacto blade as a chisel. The resulting pieces are up to 5 mm thick. The largest piece was this thick at its thickest — there was no layering that permitted finer sampling of layers. The chips came from ~ ½ the plan area of this face of the piece. The remaining material between ~~these~~ DAP 8/16/99 the layer represented by these pieces and that sampled for L1 is typically 3-5 mm and is preserved intact; i.e., it could be later sampled. Sum mass of L2 is 3.5 g.

Crush and Sieve in same manner as L1 (page 88-89). This time, crush more thoroughly.

Note - L2 is generally more friable than L1.
Also - during crushing of L2, numerous resistant pieces were encountered and removed. These are probably tuff and/or quartz fragments. L2 is likely to be more "contaminated" than L1.

NOPI-436-L2-SEP1 250 μ fraction

+ powder	7.002
empty vial	6.671
	0.331 g

Very little powder in bottom of sieve stack. Combine 125 μ + 63 μ fractions and crush more.

The fine caliche tends to clump in the mortar + pestle. This clumping is responsible for some of the larger "grain sizes," when clumps fail to break up during sieving

8/17/99 DAP

NOPI-436-L2-SEP2 125 μ fraction

	7.112
empty vial	6.372
	0.740 g

NOPI-436-L2-SEP3 63 μ fraction

+ powder	7.834
empty vial	6.373
	1.461

NOPI-436-L2-SEP4 <63 μ fraction

+ powder	6.883
empty vial	6.421
	0.462

NOPI-436-L2-SEP2-B small aliquot for chem/S.I.

	6.707
empty vial	6.605
	0.102

NOPI-499 field name DAP-18

The field sample consists of several fragments of tuff with some fracture faces coated by a white material. Select the two largest pieces, which fit together. They share a fracture face with white coating. I thought this was caliche, but the hardness convinced me it was silica. Break off a small piece and put a drop of 1M HCl on. It fizzes only in small locations — this is not caliche.
From the two adjoining pieces (~ 6 cm and 7 cm across), sample the outermost silica layer, which is whiter than the underlying buff-colored silica(?) layer. Do this by using an Exacto knife as a chisel and chipping away at the outer layer. The resulting chips are no thicker than ~ 1.5 mm. I removed ~ ½ of this layer from the two pieces (plan view).

After removing ~4 grams of this outer layer, I found that the next layer down does fizz a little under 1M HCl. Testing other pieces, I found that some are carbonate-layered.
I do not wish to prepare a sample that has both carbonate and silicate fracture lining material. The reason is that the two substances may have distinct ages. If tuff residual fragments are also sampled, there is the potential that the sample will have three components in U-Th isotopes space. Undesirable.

Note: looking at pieces, it is now obvious that most have a silica coating. Grayish light brown or buff, with the typical microcrystalline silica appearance. I don't know if this would be termed "opal." I see no irridescence. Most coatings are layered, and are typically a total thickness of 2-3 mm.

In order to avoid carbonate in my silica sample, I decided to leach in HCl. I don't think it would be wise to try to sample carbonate from this sample, because it is more difficult to imagine being able to isolate the carbonate from the silica. It is simpler to isolate the silica from the carbonate by leaching.
Note: on some pieces, carbonate forms the outer layer. It appears to me that carbonate is later — it also fills small pits on the face of the silica, and it may have formed ~~bef~~ DAP 8/10/99 below the silica by waters infiltrating cracks.

Call this sampled layer NOP1-499-L1.

NOP1-499-L1

Leaching:
- In agate mortar + pestle, break up the chips into pieces with maximum dimension ~4 mm. (Thickness is much less, typically.) Discard powder, keeping chips only.
- In weighing boat with chips, add 1M HCl, 1 ml at a time. Lots of fizzing. Add ~4 ml, then decant and rinse three times with Nanopure water. Repeat twice.

Continues to fizz! I may have been mistaken in thinking that this outer layer was mainly silica.
Do a fourth time. Then add 8 ml 1M HCl and let sit, starting at 14:55.
45 mins. later, rinse 3x. Add 5 ml 1M HCl. Still fizzes.
45 mins later, do again. Still fizzes, though less vigorously.

What was the more brownish side of the chips is quite dark when wet, and shows grains of dark minerals.

I think it best to not use this subsample.

NOP1-499-L2

Chip off pieces of caliche from the face of another piece. Buff colored, clearly contaminated with silicates. Avoid including darker tan layer below, and avoid dark Fe-Mn materials below that layer.
Break up in mortar + pestle, then sieve.

NOP1-499-L2-SEP1 250μ fraction

+ powder	7.065
empty vial	6.271
	0.794

NOPI-499-L2-SEP2 125 μ

+ powder	6.722
empty vial	6.345
	0.377

NOPI-499-L2-SEP3 63 μ

+ powder	6.971
empty vial	6.412
	0.559

NOPI-499-L2-SEP4 <63 μ

+ powder	6.501
empty vial	6.332
	0.169

NOPI-499-L2-SEP3-B portion for chem/S.I.

+ powder	6.527
empty vial	6.432
	0.095

Note: L2 was much easier to crush than L1. L2 was similar to NOPI-436 materials crushed yesterday.

There is a piece of NOPI-499 that has a hard coating (2-3 mm) that is probably silica. Problem: I can't break into smaller pieces or chip off the layer. Check into this tomorrow.

8/18/99 DAP L1 soaked in HCl overnight. Rinsed in H_2O, then added 5 ml 1M HCl. Still a small amount of fizzing. Abandon this sample.

NOPI-499-L3

This is the siliceous layered coating mentioned on previous page. The coating is light brown to tan with thin banding (<1 mm bands), hard, concoidal fracturing, and the layer varies from ~3 to 6 mm thick.

1M HCl causes fizzing on ~~the clean surface~~ DAP 8/18/99 fresh surfaces of the hard material, though drops on a light colored coating produce more vigorous fizzing. I suspect that carbonate is finely disseminated in the silica.

Using an iron base, hammer at the original pieces until I can separate pieces of the silica layers that don't have any adhering tuff substrate. It is not possible without extensive effort to remove pieces that have the apparent caliche coating (very thin, <<1 mm) because essentially all pieces have it. I decide that this is OK because: If the caliche is U-rich, but the silica is not, the low mass fraction of the caliche is such that the bulk [U] will still be low. The sample will therefore be of limited interest. If the silica is U-rich, the caliche will have little effect on the U-Th results.

The selected chips have dimensions of 1 to 9 mm.

Rinse the pieces several times in nanopure H_2O and dry covered in 120°C oven. (I did not do this with caliches because they are so friable and porous. This subsample is hard and dense.)

Crush the L3 material (~3.7 g) in a metal (WC and stainless steel?) mortar + pestle assembly. (Impactor, ring, and base; hit with hammer.)

Sieve in 250-125-63-base stack

NOPI-499-L3-SEP1 250 μ fraction

+powder	7.430
empty	6.442
	0.988

NOPI-499-L3-SEP2 125 μ

+powder	7.563
empty	6.421
	1.142

NOPI-499-L3-SEP3 63 μ

+powder	7.194
empty	6.368
	0.826

NOPI-499-L3-SEP4 <63 μ

+powder	7.053
empty	6.397
	0.656

NOPI-499-L3-SEP2-B portion for chem/S.I./XRD

+powder	6.959
empty	6.810
	0.149

NOPI-305

White caliche from layers south of ore body. Collected 7/93, field 35/52. No separates prepared. I will prepare sieve fractions of this & NOPI-306, which is from another layer in the 7-15 cm-thick caliche deposit.

Field name 793-CAL2.

There are nine sizable pieces. The largest is 60 mm long, 40 mm wide, and 15 mm thick. Sample from this large piece exclusively.

First chip away the face that is weathered brown. I want only the fresher white caliche.

The layer represented by this piece is friable and porous, and has little internal banding.

I am left with two pieces totalling 12 g, each ~10-12 mm thick. I can see banding is better developed than I had thought, so I will take one piece and split it.

Do this by standing the piece on edge and using an Exacto as a chisel. Forms a clean parting.

NOPI-305-L1 6-7 mm thick, mostly ~6 mm.
~25 x 17 mm in plan view

8/19/99 DAP Crush in agate mortar + pestle, then sieve.

NOPI-305-L1-SEP1 250 μ

+powder	6.679
empty	6.333
	0.346 g

NOP1-305-L1-SEP2 125 μ

+powder	7.694
empty	6.840
	0.854

NOP1-305-L1-SEP3 63 μ

+powder	7.377
empty	6.679
	0.698

NOP1-305-L1-SEP4 <63 μ

+powder	6.917
empty	6.391
	0.526

NOP1-305-L1-SEP2-B Portion for chem/S.I.

+powder	6.835
empty	6.731
	0.104

NOP1-305-L2

Caliche layered with L1. Product, along with L1 of splitting original piece approximately in half along banding. White (like L1), with obvious brown & red alteration removed.
5-8 mm thick, typically 6 mm.
~20 x 30 mm in plan view.
Crush in agate mortar + pestle + sieve. (Like L1, it is very easy to crush.)

NOP1-305-L2-SEP1 250 μ

+powder	7.507
empty	6.309
	1.198 g

NOP1-305-L2-SEP2 125 μ

+powder	7.481
empty	6.358
	1.123 g

NOP1-305-L2-SEP3 63 μ

+powder	7.117
empty	6.316
	0.801

NOP1-305-L2-SEP4 <63 μ

+powder	6.882
empty	6.428
	0.454

NOP1-305-L2-SEP2-B portion for chem/S.I.

+powder	6.505
empty	6.402
	0.103

NOP1-306

Collected 7/93, field notebook 35/52.
A layer of caliche from the same fracture as NOP1-305. Field name 793-CAL3.
Separated from NOP1-305 by ~ 5-10 cm.
(Fracture total thickness 7-15 cm, all filled with caliche.
Purpose of this sample is to get caliche from downslope of ore, and to look at layering (age variation?) with respect to NOP1-305.
305 + 306 are relatively "clean" caliches, but NOP1-306 is considerably more brown than NOP1-305.
Some pieces contain Fe-Mn oxide-rich layers.

Layering in most pieces: - brown, "dirty" weathered face
- more whitish, less porous, bended, 3-5 mm
- porous, friable, brownish caliche, 5-12 mm
- weathered surface (brown)

Using hammer and Exacto (as a chisel), separate out the second layer in the list above; i.e., more whitish, relative hard, low porosity layer. Eliminate brown weathered outer layer. Small amounts of the more porous caliche underlayer may remain adhering to the desired layer, but these are minor.
Rinse pieces (~1-5 mm) several times in nanopure water, then dry in oven.

8/20/99 DAP Start to crush the dried pieces in an agate mortar + pestle. It is hard, much like NOP1-499-L3 (page 95), though not as much.
Therefore, crush in the steel "impact" mortar + pestle (see p 95), then in agate. Sieve.

NOP1-306-SEP1 250μ
+powder 8.237
empty 6.715
1.522 g

NOP1-306-SEP2 125μ
+powder 7.606
empty 6.627
0.979

NOP1-306-SEP3 63μ
+powder 7.609
empty 6.651
0.958

NOP1-306-SEP4 <63μ
+powder 7.340
empty 6.654
0.686 g

NOP1-306-SEP2-B
+powder 6.796
empty 6.674
0.122

NOP1-434
Collected 9/94. Field notebook 121/3. Field name "DAP-2."
Many pieces have large clasts of tuff included. This sample is from near NOP1-17 (U-Th dated 54ky) and NOP1-306. How close is not known, because field locations were not precise.
This sample has some similarities to NOP1-306 in that it has a hard layer just under the surface. The surface layer itself is typical porous caliche. ~~Also d~~ DAP 8/20/99
Thus, the surface is light colored. The hard layers are grayish. Also different from NOP1 306 is that the selected pieces have a tuff substrate. I found four pieces that fit together. No large tuff clasts.

~~NOP1-434 L1~~ DAP 8/20/99 - Decide not to sample more than one layer.
Try to isolate the gray, hard layer. Break the pieces down to ~5-10 mm, then break down further in steel mortar + pestle. Then hand-pick gray pieces devoid of light-colored outer layers + inner layers + tuff. Look at pieces under scope and remove those with adhering caliche.
I can see that this material (i.e. selected layer) is very siliceous and riddled with residual grains.

Sieve the hand-picked grains to remove those smaller than 1 mm. 8/27/99

Rinse with nanopure H_2O; wet sieve w/ nanopure H_2O at 250 μm; rinse with H_2O; dry in oven

Crush in steel mortar + pestle, then ~~sieve~~ DAP 8/20/99 with agate mortar + pestle, then sieve

NOPI-434-SEP1 250 μ

7.322
6.737
0.585

NOPI-434-SEP2 125 μ

7.831
6.613
1.218

NOPI-434-SEP3 63 μ

8.082
6.693
1.389

NOPI-434-SEP4 <63 μ

7.481
6.752
0.729

NOPI-434-SEP2-B portion for Chem/S.I.

6.849
6.691
0.158

NOPI-501

Collected 5/94, field 121/25, field name DAP-20.
From near large calcite deposit in pit.
White and gray calcite.
Separate the two colors by breaking up with rock hammer.

NOPI-501-W

"W" = white. actually clear. Pieces up to ~3 mm. Hand pick under microscope, removing pieces with dirt on surface or large amts of black inclusions. Crush in agate mortar + pestle.
Will not make size fractions, because almost pure calcite. Put in glass vial

+ powder 7.209
empty vial 6.314
0.895 g

Take a small subsample
NOPI-501-W-B

+ powder 6.486
empty vial 6.384
0.102

Put rejected ("dirty") white calcite in plastic vial.

NOPI-501-G

Gray calcite. Ensure not clear or containing clear cc.
Hand pick under microscope to remove weathered pieces and those including clear calcite.
Crush in agate mortar + pestle, sieve as for previous samples

NOP1-501-G-SEP1 250 μ
+ powder 7.020
empty vial 6.252
0.768 g

NOP1-501-G-SEP2 125 μ
+ powder 7.497
empty vial 6.418
1.079

NOP1-501-G-SEP3 63 μ
+ powder 7.971
empty vial 6.329
1.642

NOP1-501-G-SEP4 <63 μ
+ powder 6.752
empty vial 6.334
0.418

NOP1-501-G-SEP3-B portion for chem/S.I.
+ powder 6.544
empty vial 6.389
0.155

NOP1-458
Collected 5/95, field 021/51. Caliche from above Level +40. Subhorizontal fracture ~1 m below pre-mine surface.
The caliche is evidently siliceous. Very hard. Cannot avoid sampling either some of the tuff substrate or Fe oxide staining on outer surface.
Crushed using metal mortar + pestle.
Sieve.

NOP1-458-SEP1 250 μ
+ powder 6.958
empty 6.481
0.477 g

NOP1-458-SEP2 125 μ
+ powder 7.931
empty 6.772
1.159

NOP1-458-SEP3 63 μ
+ powder 7.533
empty 6.433
1.100

NOP1-458-SEP4 <63 μ
+ powder 7.285
empty 6.646
0.639

NOP1-458-SEP2-B portion for chem/SI
+ powder 6.572
empty 6.458
0.114

8/28/99
DAP

After a conversation with Bret Leslie (NRC), I decide it would be useful to fire the samples at 900°C in order to:

1. be able to calculate [U] in the carbonate and non-carbonate component
2. simplify total dissolution.

The ability to calculate [U] in the two components depends on measuring ① weight loss from firing and ② [U] in >1 fraction.
The weight loss from firing is due to CO_2 (g) loss, and therefore reflects the carbonate component of $CaCO_3$. Can thus calculate the mass of $CaCO_3$ in the sample.

Go through the ~~14~~ DAP 8/28/99 18 samples prepared in previous days (pp 82-105) and select two fractions from each for firing. Select on the basis of ① desire for spread in grain size and ② amount available.
Two samples (NOP1-12 and NOP1-501-W) - take only one fraction. NOP1-12: the small grain size fraction is too small (0.08 g). NOP1-501-W: very pure CC, and no size fractions taken.

Because this is for [U] analysis only, take only a portion of the sample (preferably ½) so still have some if need non-fired sample.

Some of the samples are supposed to be calcite separates, but I'm taking size fractions for them anyway.
Clear calcites: I see non-white particles in coarse fraction.
Gray calcites: There may be significant U in inclusions that help give it its color.

Select 34 samples in all.

Do in batches of 20 and 34.

Weigh ½ of sample powder into a 20 ml Pyrex beaker for the 100°C drying step.

But first, take 250μ and 125μ fractions and crush all of fraction in agate mortar + pestle. Want small grains so $CaCO_3$ is free to react & evolve CO_2. Place powder back in same container. (Some were already fine enough and were not crushed again)

Powders crushed in this way: Note: don't know grain sizes.

NOP1-12-SEP1
NOP1-24-SEP1
NOP1-29-SEP1
NOP1-501-G-SEP1
NOP1-436-L1-SEP1
NOP1-436-L2-SEP2

Will do more tomorrow

Now, weigh ~½ into 20 ml Pyrex beaker. Add "-F" to name.

NOP1-12-SEP1-F Beaker 1

empty beaker	14.009
+ powder	14.649

NOP1-501-W-F Beaker 2

empty	14.603
+ powder	15.023

NOP1-19-SEP1-P-F Beaker 3
empty 13.694
+powder 14.321

NOP1-19-SEP2-F Beaker 4
empty 14.928
+powder 15.683

NOP1-24-SEP1-F Beaker 5
empty 13.600
+powder 15.088 More than others: replicate for UT lab

NOP1-24-SEP2-F Beaker 6
empty 14.942
+powder 15.240

NOP1-29-SEP1-F Beaker 7
empty 13.636
+powder 14.291

NOP1-29-SEP2-F Beaker 8
empty 13.834
+powder 14.031

NOP1-164-SEP1-F Beaker 9
empty 13.858
+powder 15.673 More than others: replicate

NOP1-164-SEP4-F Beaker 10
empty 13.900
+powder 14.457

NOP1-166-SEP1-P-F Beaker 11
empty 13.763
+powder 14.316

NOP1-166-SEP2-F Beaker 12
empty 14.973
+powder 15.275

NOP1-179-SEP1-P-F Beaker 13
empty 13.553
+powder 13.928

NOP1-179-SEP2-F Beaker 14
empty 14.731
+powder 14.849

NOP1-501-G-SEP1-F Beaker 15
empty 14.716
+powder 15.111

NOP1-501-G-SEP3-F Beaker 16
empty 14.871
+powder 15.687

NOP1-436-L1-SEP1-F Beaker 17

empty	14.735
+ powder	15.155

NOP1-436-L1-SEP4-F Beaker 18

empty	13.624
+ powder	13.927

NOP1-436-L2-SEP2-F Beaker 19

empty	15.070
+ powder	15.393

NOP1-436-L2-SEP4-F Beaker 20

empty	15.023
+ powder	15.264

time 21:30 Arrange beakers on polypropylene tray:

16	17	18	19	20
11	12	13	14	15
6	7	8	9	10
1	2	3	4	5

Place Blue M Stabil-Therm oven set too 100°C.

8/29/99 DAP

Start sample firing

Remove 10 beakers from 100°C oven, let cool, then weigh powder into 10 ml porcelain crucibles (Coors model 60104).

DAP 8/31/99 Data entered into Excel file "Aug 99 sample info.xls." A check here means entries were checked.

NOP1-12-SEP1-F crucible 1

empty	9.8460	g
+ powder	10.4793	
after firing	10.2143	✓

NOP1-501-W-E crucible 2

empty	9.9503
+ powder	10.3694
after firing	10.1924 ✓

NOP1-19-SEP1-P-E crucible 3

empty	8.5252
+ powder	9.1487
after firing	8.8920 ✓

NOP1-19-SEP2-E crucible 4

empty	9.7359
+ powder	10.4855
after firing	10.1805 ✓

NOP1-24-SEP1-F crucible 5

empty	9.8943
+ powder	~~11.3804~~ 11.3558 ✓
after firing	10.7361

some powder on rim.

DAP 8/25/99 - spill

NOP1-24-SEP2-F crucible 6

empty	8.8241
+ powder	9.1186
after firing	8.9964 ✓

NOP1-29-SEP1-F — crucible 7

empty	9.8079
+ powder	10.4605
after firing	10.1860 ✓

NOP1-29-SEP2-F — Crucible 8

empty	9.7348 DHP 8/23/11
+ powder	9.9304
after firing	9.8498 ✓

NOP1-164-SEP1-F — crucible 9

empty	9.7708
+ powder	11.5774
after firing complete?	11.2342 ✓

NOP1-164-SEP4-F — crucible 10

empty	9.8987
+ powder	10.4545
after firing	10.2863 ✓

time
10:45 Place 10 crucibles in Fisher Isotemp Muffle Furnace, Model 184A, set to 900° C.
Arrangement of crucibles:

back	9	10
	7	8
	5	6
	3	4
front	1	2

11:15 760°C
11:30 800°C. Turn up a bit.
12:00 860°C
12:20 890°

14:50 Remove from oven. Let cool, then weigh crucible + powder and record above as "after firing."

Need to crush remaining 250μ and 125μ fractions before firing. Use agate mortar + pestle.

Samples crushed: NOP1-499-L2-SEP1
NOP1-499-L3-SEP1 *
NOP1-305-L1-SEP2
NOP1-305-L2-SEP1
NOP1-306-SEP1
NOP1-434-SEP2
NOP1-458-SEP1 *

* Particularly difficult to crush, especially 499-L3-SEP1.

For final 14 samples, weigh ~½ into 20 ml pyrex beaker

NOP1-499-L2-SEP1-F — Beaker 21

empty	13.753
+ powder	14.157

NOP1-499-L2-SEP3-F — Beaker 22

empty	13.724
+ powder	14.003

NOP1-499-L3-SEP1-F — Beaker 23

empty	13.679
+ powder	14.163

NOP1-499-L3-SEP4-F — Beaker 24

empty	13.846
+ powder	14.185

NOP1-305-L1-SEP2-F Beaker 25
empty 13.838
+ powder 14.212

NOP1-305-L1-SEP4-F Beaker 26
empty 13.682
+ powder 13.957

NOP1-305-L2-SEP1-F Beaker 27
empty 13.910
+ powder 14.500

NOP1-305-L2-SEP4-F Beaker 28
empty 13.907
+ powder 14.133

NOP1-306-SEP1-F Beaker 29
empty 13.823
+ powder 14.825 More than most → replicate

NOP1-306-SEP4-F Beaker 30
empty 14.951
+ powder 15.311

NOP1-434-SEP2-F Beaker 31
empty 13.971
+ powder 14.499

NOP1-434-SEP4-F Beaker 32
empty 13.969
+ powder 14.335

NOP1-458-SEP1-F Beaker 33
empty 14.865
+ powder 15.100

NOP1-458-SEP4-F Beaker 34
empty 14.795
+ powder 15.117

16:30 Put Beakers 21-34 in 100°C oven. ↗

back
31 32 33 34
26 27 28 29 30
21 22 23 24 25

As samples are finished firing and weighing, transfer to small glass vial. Numbering scheme: "N99-#." N99 = Nopal 1999. Want simple numbering for convenience of U-Th analysts and for hiding blind replicates

N99-1	=	NOP1-12-SEP1-F	0.36 g
N99-2	=	NOP1-501-W-F	0.2415 g
N99-3	=	NOP1-19-SEP1-P-F	0.3667
N99-4	=	NOP1-19-SEP2-F	0.4437
N99-5	=	NOP1-24-SEP1-F (N99-22 is replicate)	~~0.8426~~ ~0.417 AP 8/29/99
N99-6	=	NOP1-24-SEP2-F	0.1702
N99-7	=	NOP1-29-SEP1-F	0.3809
N99-8	=	NOP1-29-SEP2-F	0.1156
N99-9	=	NOP1-164-SEP1-F (N99-21 is replicate)	0.7188
N99-10	=	NOP1-164-SEP4-F	0.3882
N99-11	=	NOP1-166-SEP1-P-F	0.3249
N99-12	=	NOP1-166-SEP2-F	0.1794
N99-13	=	NOP1-179-SEP1-P-F	0.2340
N99-14	=	NOP1-179-SEP2-F	0.0718
N99-15	=	NOP1-501-G-SEP1-F	0.2381
N99-16	=	NOP1-501-G-SEP3-F	0.4825

N99-17 = NOP1-436-L1-SEP1-F 0.2619 g
N99-18 = NOP1-436-L1-SEP4-F 0.1818
N99-19 = NOP1-436-L2-SEP2-F 0.2060
N99-20 = NOP1-436-L2-SEP4-F 0.1494
N99-21 = NOP1-164-SEP1-F replicate 0.7360
N99-22 = NOP1-24-SEP1-F replicate 0.4256

2nd Firing Batch

Samples dried since yesterday in 100°C oven.

NOP1-166-SEP1-P-F Crucible 1
empty 9.8462
+powder 10.3968
after firing 10.1717 ✓

NOP1-166-SEP2-F Crucible 2
empty 9.9505
+powder 10.2507
after firing 10.1294 ✓

NOP1-179-SEP1-P-F Crucible 3
empty 8.5255
+powder 8.8973
after firing 8.7583 ✓

NOP1-179-SEP2-F Crucible 4
empty 9.7363
+powder 9.8529
after firing 9.8084 ✓

NOP1-501-G-SEP1-F Crucible 5
empty 9.8945
+powder 10.2893
after firing 10.1303 ✓

NOP1-501-G-SEP3-F Crucible 6
empty 8.8241
+powder 9.6368
after firing 9.3009 ✓

NOP1-436-L1-SEP1-F Crucible 7
empty 9.8082
+ powder 10.2257
after firing 10.0717 ✓

These four 436 powders are the first to be whitish rather than dark brown after firing. They are also the first caliches to be fired.

NOP1-436-L1-SEP4-F Crucible 8
empty 9.7349
+powder 10.0364
after firing 9.9166 ✓

NOP1-436-L2-SEP2-F Crucible 9
empty 9.7726
+ powder 10.0944
after firing 9.9781 ✓

NOP1-436-L2-SEP4-F Crucible 10
empty 9.8989
+ powder 10.1396
after firing 10.0492 ✓

17:10 Place crucibles 1→10 in muffle furnace set to 900°C.

Arrangement

back	9	10
	7	8
	5	6
	3	4
front	1	2

Knob on furnace: 3 4

21:10 Remove (temp is 900°C), let cool, weigh, record "after firing" weight above.
Oven off.
Transfer powders to vials - pp 115 - 116.

8/30/99
DAP

3rd Firing Batch

Samples in 100°C oven since yesterday.

NOP1-499-L2-SEP1-F — Crucible 1

empty	9.8460	g
+ powder	10.2459	
after firing	10.1190	✓

NOP1-499-L2-SEP3-F — Crucible 2

empty	9.9503
+ powder	10.2284
after firing	10.1359 ✓

NOP1-499-L3-SEP1-F — Crucible 3 — whitish

empty	8.5252	
+ powder	9.0061	
after firing	8.8637	✓

NOP1-499-L3-SEP4-F — Crucible 4 — whitish, but a little darker than SEP1

empty	9.7361
+ powder	10.0728
after firing	9.9704 ✓

NOP1-305-L1-SEP2-F — Crucible 5 — almost white

empty	9.7724
+ powder	10.1433
after firing	10.0201 ✓

NOP1-305-L1-SEP4-F — Crucible 6 — DAP 8/30/99 whiter than

empty	8.8241
+ powder	9.0973
after firing	9.0038 ✓

NOP1-305-L2-SEP1-F — Crucible 7 — almost white

empty	9.8989
+ powder	10.4862
after firing	10.2962 ✓

NOP1-305-L2-SEP4-F — Crucible 8

empty	9.7348
+ powder	9.9588
after firing	9.8787 ✓

time 10:10

Place crucibles in muffle furnace set to 900°C.
(temp at 800°C now, but still rising.)

Arrangement:

back	7	8
	5	6
	3	4
front	1	2

14:10 T = 900°C. Remove samples from furnace.
Let cool, then weigh as "after firing" recorded above.

Transfer fired powder to small glass vial

N99-23	= NOP1-499-L2-SEP1-F		0.2657
N99-24	= NOP1-499-L2-SEP3-F		0.1798
N99-25	= NOP1-499-L3-SEP1-F		0.3335
N99-26	= NOP1-499-L3-SEP4-F		0.2297
N99-27	= NOP1-305-L1-SEP2-F		0.2441
N99-28	= NOP1-305-L1-SEP4-F		0.1742
N99-29	= NOP1-305-L2-SEP1-F		0.3927
N99-30	= NOP1-305-L2-SEP4-F		0.1400
N99-31	= NOP1-306-SEP1-F	N99-37 is replicate	0.3547
N99-32	= NOP1-306-SEP4-F		0.2564
N99-33	= NOP1-434-SEP2-F		0.3013
N99-34	= NOP1-434-SEP4-F		0.2175
N99-35	= NOP1-458-SEP1-F		0.2254
N99-36	= NOP1-458-SEP4-F		0.3021
N99-37	= NOP1-306-SEP1-F	replicate	0.3653

4th Firing Batch

Samples in 100°C oven since yesterday.

NOP1-306-SEP1-F Crucible 1 white

empty	9.7348
+ powder	10.7286
after firing	10.4590 ✓

NOP1-306-SEP4-F Crucible 2 white

empty	9.9507
+ powder	10.3088
after firing	10.2078 ✓

NOP1-434-SEP2-F Crucible 3 beige

empty	8.5255
+ powder	9.0511
after firing	8.8297 ✓

NOP1-434-SEP4-F Crucible 4 beige

empty	9.7362
+ powder	10.1018
after firing	9.9536 ✓

NOP1-458-SEP1-F Crucible 5 pinkish (Fe?)

empty	9.7726
+ powder	10.0072
after firing	9.9996 ✓

NOP1-458-SEP4-F Crucible 6 pinkish

empty	8.8242
+ powder	9.1454
after firing	9.1332 ✓

time 16:00 Place 6 crucibles in muffle furnace set to 900°C.

Arrangement

back	5	6
	3	4
front	1	2

20:05 T = 900°C

Remove crucibles, let cool, then weigh "after firing" and record on p. 120.

Transfer powders to small glass vials and record on page 119.

~~6/26~~ 6/26/00

6/21/00 DH

Weigh out part of NOPI-494-UPb-3 for Neutron Activation test

NOPI-494-UPb-3-NA
weigh into plastic ~7 ml scintillation vial.
weigh out & original vial

+ powder	3.6015
empty scint. vial	3.5473
	0.0542 g

Out of orig vial

wt. before	3.8254
wt. after	3.7706
	0.0548

This powder will be tested for neutron activation analysis using the Cf-252 source as part of James Weldy's IR+D project.

6/26/00 Try to prepare more Uranophane separates

NOPI-102
Bag still labelled "NOPI-ECP-26.85/11.65 5/23/92"

Take a large piece of NOPI-102 and scrape off yellow material on one face, including from cracks and crevices.

Sieve
>1000 μ : put back with bulk sample
250-1000 μ
125-250 μ
<125 μ
} Place in vials { NOPI-102-UPb1-1, NOPI-102-UPb1-2, NOPI-102-UPb1-3

In all, it looks like it will be too difficult to hand-pick for "pure" uranophane.

In 250-1000 μ fraction, few grains are pure uph. In other two, the effort to produce a sizable separate would be prohibitive. It would not be too difficult to produce small amounts.

6/27/00 HEAVY LIQUID SEPARATION ON NOPI-102-UPb1-X

Follow procedure on p. 20-21 of this notebook.
Use less methylene iodide.
Add NOPI-102-UPb1-3 at 11:00. Shake to break up clumps.
However, with NOPI-102-UPb1-3, use filter paper #5 instead of membrane filter. Wash filter down with acetone. Dry with pumped air. Put uph into petri dish using (i) policeman and (ii) washing down filter with acetone into petri dish
See a lot of Fe minerals.

NOPI-102-UPb1-2. Pour into same methylene iodide in same sep. funnel at 11:40 AM. Swirl to wash down walls.
~1:00 PM, let heavies out bottom. This time, use membrane filter set-up as on pp 20-21. However, use a "Metricel" membrane. Wash w/ acetone. Wash solids out of filter funnel into petri dish with acetone. When disassemble filter funnel, find that the membrane has dissolved! Put solids in a paper funnel (#5) and wash voluminously with acetone to rinse out dissolved membrane material.

NOPI-102-UPb1-1 When try to let heavies out, clogs the stopcock. Pipet out from the top into paper filter (#5) in funnel. Again, rinse well with acetone. Wash from paper into petri dish with acetone.

Magnet: Using a strong red magnet from structure analogue lab, see if Fe minerals respond – as potential separation tool. No. No effect on any grains. 6/29/00

So, now have three separates in petri dishes. Acetone evaporating.
Each is extensively contaminated with black to reddish brown Fe (?) mineral grains.

When dry, they are very sticky, clumping.

Rinse three or more times with nanopure H_2O, decanting from petri dish. I can see that some uph is lost because it is "floating" on the water surface.
Rinse into plastic weighing boat, then dry in 50°C oven.

NOPI-234-UPb

Prepare another "uranophane" sample from NOPI-234, which is in a bag labelled 393-26.85/11.3.
I add "NOPI-234" to the bag.
Scrape yellow grains off face of several pieces, all having similar appearance. Try to disturb other materials less; therefore I collect less uph, but perhaps purer. Still not very pure.
Place is snap-cap vial labelled.

6/28/00 Sieve using 125 μ sieve.

>125 μ = NOPI-234-UPb-1
<125 μ = NOPI-234-UPb-2

Pretty rich in uph, but see other grains. Uph especially rich in ?.

More from NOPI-494.

Take four subsamples from NOPI-494 set 8 uph-rich samples.
Call them
NOPI-494-1
NOPI-494-2
NOPI-494-3
NOPI-494-4.

However, none of these has sufficient uph to justify separating, considering that we already have a U-Pb isochron from NOPI-494.
Return to storage in trailer.

Now have five uph "separates" in glass vials. Want to estimate weights. (Did not weigh vials before putting sample in.)
Weigh five empty vials:
6.410, 6.413, 6.383, 6.399, 6.378
Average = 6.397 range = 6.378 – 6.413
Now weigh uph-containing vials:

NOPI-234-UPb-1	6.520 g	est. mass = 0.12 g
NOPI-234-UPb-2	6.621	0.22
NOPI-102-UPb1-1	7.169	0.77
NOPI-102-UPb1-2	6.897	0.50
NOPI-102-UPb1-3	7.263	0.87

7/17/00
DAP

Nopal Opal

Several months ago, B. Leslie and I looked at samples provided by I. Reyes from in and near the ore zone. We selected two that contained opal that looked good for sampling for U-Th age dating.

Today, take photos of the two using digital camera.

Photos ~~1-5~~ DAP 7/17/00 IR-99-1 after/by erasing, 1-4
Photos ~~6-10~~ IR-99-6 " " 5-9

It is difficult to get close-up enough to clearly show relationships.

Fields of view ~ 3" across, maximum ~4" across.

Take photos of late-stage Uranophane

Photos 10-14 NOPI-234 (see p. 124)
Photos 15-16 NOPI-494-1 (see p. 125)

7/18/00
DAP

Photos transferred to PC using Olympus Camedia Master software (version 1.1).

Photos are now numbered
P1010003.jpg, ~~through~~ DAP 7/18/00
P1010004.jpg, P1010006.jpg, and P1010007.jpg are IR-99-1 photos.
P1010010.jpg, P1010011.jpg, P1010012.jpg, P1010013.jpg, and P1010014.jpg are IR-99-6 photos.
P1010016.jpg, P1010017.jpg, P1010018.jpg, P1010019.jpg, and P1010020.jpg are NOPI-234 photos.
P1010021.jpg and P1010023.jpg are NOPI-494-1 photos.

Save good photos under more descriptive names.

P1010004.jpg → IR-99-1 photo 1.jpg
P1010006.jpg → IR-99-1 photo 2.jpg
P1010007.jpg → IR-99-1 photo 3.jpg

P1010010 → IR-99-6 photo 1
P1010011 → IR-99-6 photo 2
P1010012 → IR-99-6 photo 3
P1010014 → IR-99-6 photo 4

P1010016 → NOPI234 photo 1
P1010018 → NOPI234 photo 2
P1010019 → NOPI234 photo 3
P1010020 → NOPI234 photo 4

P1010021 → NOPI494-1 photo 1
P1010023 → NOPI494-1 photo 2

7/20/00
DAP

IR99-1 Opal preparation

Sometimes labelled "IR-99-1."
Hold geiger counter against yellow, opal-coated part of rock (where arrow is pointing) and get 0.35 mR/hr or ~~0.4~~ DAP 7/20/00 400 cpm.
So there is plenty of U. This count rate is about the same as what I get when I hold a small glass vial of uranophane against the detector.
This rock has many intriguing textural relationships.
The lowest "coating" is a colorless to grayish-brown translucent "opal." On top of this is a white or yellow-green "opal", which is overlain or intergrown with uranophane.

i.e., uranophane is younger than opal.

7/

Also see clear needles that appear to be co-eval with uranophane. Are these also uph?
Tomorrow, make more detailed observations and take photos.

7/21/00 Start observations of IR-99-1 hand specimen.

Use Nikon SMZ-2T binocular microscope with camera attachment. Take pictures using Nikon UFX-IIA controller.
I have print-outs of earlier photos:
IR-99-1 photo 1.jpg shorthand "JPG 1"
IR-99-1 photo 3.jpg. " "JPG 3"

Mark locations of photomicrographs and observations on these.

Photo 1 (new numbers for photomicrographs)
Field of view 9.5 mm across. (Use ruler)
Located on JPG 3.

7/18 DAP Reddish-brown (but perhaps transparent and showing colors of underlying tuff) botroidal (sp?) opal overlain by whitish thin opal layer, which is ~~overal~~ DAP 7/21/00 ~~overal~~ DAP 7/21/00 overlain by, and intergrown with, yellow uranophane (uph) needles.

Photo 2 From center of Photo 1 field, but magnification set at 6.3X.
Field of view 2 mm across.
Patch of underlying "red-brown" opal. Uph needles on top of, but seeminly partly submerged, in whitish opal layer.

Located on JPG3

7/25/00 DAP Photo 3
Patches of underlying dark-appearing opal, overlain by clear to whitish thinner opal, which has two relationships with uph: (1) uph is later, growing out from whitish opal layer, (2) uph needles are coated by clear opal.
(1) is best seen just right of center
(2) is best seen on left side of photo

2x magnification, field of view 6.5 mm across.
↓
on scope

Located on JPG 3

Photo 4
6.3 x view of uph needles on top of and coated by clear opal, which is underlain by dark opal.
See botroydial character of underlying opal.
Yellow uph needle sticking out alone appears un-coated, as well as bundle pointing toward it.
2 mm across.
Located on JPG 3.

Photo 5
Both yellow and clear needles growing out from latest opal.
Located on JPG 1.
6.3 x magnification, 2 mm across

Photo 8 DAP 7/25/00

~~Photo 6~~

Looking down into "window" in yellow-green opal to brownish opal below.
Center of picture has small yellow needles growing last, as in Photo 5.
Is yellow color of opal from included uph?
Can't see embedded needles, but may be present.
1x magnification, 13 mm across.
Located on JPG 1.

Photo 6

Uph needles in cavities.
2.7 x magnification, 5 mm across.
This photo with "1%" area illumination.

Photo 7

Same as Photo 6, except "30%" area ~~##~~ DAP 7/25/00 illumination.

I am seeing "ghosts" of needles inside almost every occurrence of yellow opal.

Photo 9

An example of "bubble" appearance that I now believe is an image of an embedded uph needle (end on) in "yellow" opal. Also see small, colorless, latest-stage needles in pinkish area. Also see conchoidal fracture of yellow opal.
3.2 x magnification, 4 mm across.

No - this is a reflection of the circular fluorescent tube illuminating the view.

Photo 10

2x magnification, 6.5 mm across.
Coated needles in yellow opal. Patch of underlying darker opal.
Located on JPG 3.

Photo 11

Yellow opal and uph needles overlying whitish opal.
1x magnification, 13 mm across.
Located on JGP 3.

Photo 12

1x magn, 13 mm across
Located on JPG 3.
DAP 7/25/00 ~~Topmost~~ yellow opal in center, surrounded by underlying colorless opal. White-pink bumby opal is topmost.

Photo 13

2.7 x magn., 5 mm across
White-pink opal with a few yellow and colorless needles on top. Dark patches are underlying opal.

IR-99-6 Photos

Locations referenced to and marked on Print-out of IR-99-6 photo — .jpg (check later)

Photo 14

Clusters of uph (?) needles overlying colorless opal as also in IR-99-1. 3.4 magnif., 4 mm across.

Photo 15 What is this? Concentric + radial shapes in yellow "bulb." 2.7 x magn, 5 mm across.

Photo 16
What are pinkish-white blades? Zeolite?
See yellow spheres that look – on the outside – like the one in Photo 15.
Both ~~overly~~ DFP 7/25/00 overlie clear opal.

Photo 17
1.4 magnif., 9 mm across.
Whitish opal overlying darker colorless opal.
Clusters of radiating acicular crystals Zeolite?

Photo 18 White botroidal (?) opal of greenish translucent opal
1.6 x magnif., 8.5 mm across.

IR-99-3
Same relationship, though less latest stage uph

Photo 19
Yellow uph needle on top of light opal, which is on top of colorless opal.
Elsewhere in rock, see radial colorless clusters. zeolite? ?
3.8x magnif., 3.5 mm across.

IR-99-4

Do not see uph in this, though I do see yellow opal. Yes – do see a few uph xtals.

Photo 20 1x magnif., 13 mm across.
Yellow-Green + clear opal. Pinkish crystals. Zeolite?

XRD ?

IR-99-2
Like 4, I see no uph. Wait – there are small yellow areas on surface that could be uph.
Photo 21 1x magnif., 13 mm across.

Greenish translucent opal, with conchoidal fracture overlying altered tuff.
IR-99-2a piece.

IR-99-7 Photo 22
1x magnif., 13 mm across
Yellow opal to right + left.
Apparent acicular uph in center.
Difficult to separate opal from uph.

IR-99-8
Photo 23 1.4 magnif.
What is this?
Bret said it's skeletal opal growth on uph needles.

Photo 24 4.2 x magnif., ~3 mm across.
More of the same, but closer up.

7/25/00 DFP

Sampling strategy for now:
I will take one sample only of opal that is older than uph. It does not provide info, necessarily, on release from U minerals.
Take from IR-99-1, because the relative age relationships are better expressed than in IR-99-6.
Clear opal → yellow opal → uph (uph also in yellow opal).

I want two opals with no uph. Select from among IR-99-2, IR-99-3, and IR-99-4.

IR-99-1 Sampling

Area of rock from which I'm chipping off pieces is marked as "Sampling Area" on JPG1.
As I break off the outer layers, I see that uph needles have grown into voids in the clear opal. Again shows that uph is later than clear opal. Also, further suggests that it is uph that is giving yellow opal its color. 7/30/00 DAP

7/28/00 Finish sampling and purifying "IR-99-1 Opal 1".

Broke up pieces ~~of~~ material sampled from area marked on JPG1. botryoidal? (sp?)

Hand pick very carefully so that I only have clean pieces of "glassy" clear opal. It has a faint green tinge to it — not perfectly transparent, but definitely translucent. The green is very faint (otherwise would be perfectly transparent).

~~Ref~~ DAP 7/28/00
Reject pieces that have any other materials adhering, such as Fe oxides, uph, or black inclusions.
Reject any pieces that are at all cloudy or yellow.

I did include some with Fe oxide staining yesterday and leached as follows:
1 M HNO_3, 3 times
1 M HCl, 3 times
Thorough water rinse.
(in teflon Oak Ridge centrifuge tube. Pipet off supernate. Do not centrifuge.)

But Fe oxides remained.
Today, hand-pick only clean pieces and put in a glass scintillation vial.

Stained pieces put in a glass vial labelled "IR-99-1 Opal 1 – Dirty."

I would estimate I have well under 100 mg.

Compared some other opal separates that were prepared years ago.

19.0/5.0 Sep 3 From NOPI-111. Collected from edge of level +10
Preparation + description Notebook 024, p. 234 - 237.
(Sep1 was dated by U-Th in CNWRA lab at 54±2 ky)
Sep 3: Most of the opal resembles the glassy, slightly greenish material I have purified from IR-99-1. Most is attached to an Fe oxide-rich mass that appears to have been a substrate.
Was the dated Sep1 made of this same opal?
I see no U minerals or yellow opal here.

19.0/5.0 Sep 5 From same sample as Sep 3. Same notebook reference. Described in notebook 24 has having Uranophane inclusions.
This looks identical to clear, glassy, botryoidal opal seen in Sep 3 and in oldest layer IR-99-1.
I do see some bright yellow grains, but none are unequivocally inclusions. All are on the surface of the opal (not broken surfaces) and thus appear to post-date the opal — as also seen in IR-99-1.

? 19.0/5.0 Sep 2 ? The ink has faded, but I can read "0/5.0 Sep" and "Slightly stained" on the bottle. I am fairly certain it is 19.0/5.0 Sep 2.
This is the same glassy opal as in other seps.
I conclude that Sep 1 was also.

NOPI-ECP-37-Sep 3 From wall just to right of Level +00 adit. See field notes: 036/2.
Preparation of separates: 024/151-160. On page 166 it is said that opal may have "measurable U within it."
This sample has both the clear layer and the bright yellow layer, as seen in other samples.
In a quick inspection, I could not identify inclusions of U minerals, counter to earlier description: "opal covering beta uranophane." However, it would be reasonable for there to be such inclusions in the yellow opal.

Now that I see the consistent appearance of the two opals (± others): (1) greenish glassy, (2) bright yellow, in several samples, I decide to sample the yellow opal from IR-99-1, so I have both types from one sample.

IR-99-1 Opal 2

Take the remnants of opal 1 selection that I saved in a vial (not "IR-99-1 Opal 1 Dirty") and pick out pieces of yellow opal (translucent).
Avoid:
- crystalline uranium mineral
- clear opal
- opaque and/or milky material
- Fe oxides and other contaminants.

Save the best pieces. Some do have Fe oxides, but these can be avoided later if only a small sample is needed.

Note that the "whitish" opal layer described in the notes on pp 128-132 is not represented by these samples. Recall that some uranophane post-dates even the whitish opal, and some is coated by it.

7/30/00 DAP

IR-99-10

This looks to be a good candidate for a third opal sample for U-Th. Unlike nearly every other sample, it does not contain bright yellow opal or U minerals.

Its opal strongly resembles the greenish botryoidal opal photographed from IR-99-4 and IR-99-2 (see p. 132-133).

Using a gamma detector, the opal-covered surface registers 0.15 mR/hr, well above the background of ~ 0.025 mR/hr. Thus, U is apparently relatively rich.

More Photos Elite Chrome Extra Color slide film, ASA 100.
Take new photos of IR-99-10 + opal separates, also re-do some of the previous roll (pp. 128-133) because they were a bit on the overexposed side.

IR-99-10

Photo 1 1x magnif., 13 mm across (long dimension)
Greenish, botryoidal (sp?) opal overlying oxidized tuff substrate.
See "bumps" on opal, translucence (colors of underlying tuff showing thru), and "window" in opal to tuff.

Photo 2
Same view, but exposure setting on UFX-IIA control box adjusted to $-\frac{1}{3}$. 3.02 seconds compared with 3.80 for Photo 1.

Photo 3
Same; but exposure set to $-\frac{2}{3}$. 2.39 sec shutter.

Photo 4 2.5x magnific., 5.5 mm across.
Botryoidal character of opal, and conchoidal fracture.

Photo 5
Same, with exposure set to $-\frac{1}{3}$.

Photo 6
Opal layer thins here. Greenish. 1x magnif.

Photo 7
Same; exposure set to $-\frac{1}{3}$.

Photo 8 Botryoidal opal. Fe oxide (reddish coating opal in places. 1x magnif.

Photo 9
Same; exposure set to $-\frac{1}{3}$.

IR-99-1 Opal 1

Photo 10 2.2x magnification, 6 mm across.
Clear, clean, greenish, glassy opal

Photo 11 exposure $-\frac{1}{3}$.

Photo 12 exposure $-\frac{1}{3}$.

IR-99-1 Opal 2

Photo 13 2.2x magnif., 6 mm across.
Bright yellow opal. A little dirty.

Photo 14 exposure $-\frac{1}{3}$.

IR-99-1 hand sample re-dos

Photo 15 Same as Photo 6 from previous roll (p. 130) with exposure = $-\frac{1}{3}$.

Photo 16 Same as previous roll Photo 1 (p. 128). 1.5x.
exposure = $-\frac{1}{3}$.

Photo 17 Same as previous roll Photo 2 (p. 128).
exposure = $-\frac{1}{3}$.

Photo 18 Same as previous Photo 3 (p. 129)
exposure = $-\frac{1}{3}$.

Photo 19 Same as previous Photo 4 (p. 129)
exposure = $-\frac{1}{3}$.
May be a little different, because the rock broke here during sampling.

Photo 20 Same as previous Photo 5 (p. 129)
exposure = $-\frac{1}{3}$.

Photo 21 Same as previous Photo 10 (p. 131)
exposure = $-\frac{1}{3}$.

Photo 22 Same as previous Photo 11 (p. 131)
exposure = $-\frac{1}{3}$.

Photo 23 Approximate area of previous Photo 12 (p 131); rock broken up here during sampling.
1.4x magnification. 9.5 cm across.

Photo 24 Same as previous Photo 13 (p. 131)
exposure = $-\frac{1}{3}$.

8/1/00 Prepare IR-99-10 Opal.
Using a nail as a chisel, break off greenish opal coating depicted in photos described on pages 137–138.
Sort pieces, keeping those that tend to be all or mostly opal, preferring cleaner pieces. Use binocular microscope.
Place chosen pieces in Pyrex beaker, add nanopure H_2O_2, and put in ultrasonic bath four times for 15 minutes each. DMP 8/1/00
Between sonications, rinse 2x with nanopure water.

Aside:

If dissolve 1 mg uranophane → 0.5 mg U. 8/3/00 DAP

If in 30 ml solution:

$[U] = 0.5 \times 10^{-3}\ g / 0.03\ L = 1.7 \times 10^{-2}\ g/L$

$= 17\ mg/L$

If dissolve 1 mg opal with 1000 ppm U in 30 ml soln:

$[U] = \frac{(0.001\ g)(0.001\ g/g)}{0.03\ L} = 3 \times 10^{-5}\ g/L$

$= 0.03\ mg/L$

in 10 ml soln: → 0.1 mg/L

Back to IR-SS-10 Opal

Leach opal sample in 1 M HCl about one hour.
Rinse 3x with water.
Sonicate 15 minutes in 0.1 M HCl, 15 mins.
Rinse 3x with water

Dry at 50°C in beaker.

XRD Samples

Prepare splits of carbonate samples for XRD
Weigh out portion of one of the existing separates into a 7-ml plastic scintillation vial.
Tare on empty vial

NOPI-12-SEP1-XRD 0.243 g

~~empty~~ vial DAP 8/3/00

NOPI-501-W-XRD
This is not a separate of NOPI-501-W (the only XRD that is not a split). NOPI-501-W is a small sample, because I carefully picked clear calcite crystals.
For this XRD powder, grind some of the grains in the vial labelled "NOPI-501-W rejects" in an agate mortar + pestle. This will contain some non-calcite minerals, though probably not enough to show up in XRD.

0.440 g

NOPI-19-SEP2-XRD 0.286 g

NOPI-24-SEP1-XRD 0.283 g

NOPI-29-SEP1-XRD 0.298 g

NOPI-164-SEP1-XRD 0.540 g

NOPI-166-SEP1-P-XRD 0.253 g

NOPI-179-SEP1-P-XRD 0.182 g

NOPI-501-G-SEP3-XRD 0.268 g

NOPI-436-L1-SEP3-XRD 0.313 g

NOPI-436-L2-SEP3-XRD 0.331 g

NOPI-499-L2-SEP1-XRD 0.202 g

NOPI-499-L3-SEP3-XRD 0.300 g

NOPI-305-L1-SEP3-XRD 0.303 g

NOPI-305-L2-SEP3-XRD 0.328 g

NOPI-306-SEP3-XRD 0.339 g

NOPI-434-SEP3-XRD 0.340 g

NOPI-458-SEP3-XRD 0.342 g

Powders delivered - along with a sample set from Paul Bertetti - to Jim Spencer of Division 18.

8/4/00
DAP

IR-99-10
Finish hand-picking relatively clean greenish, clear opal.

IR 99-10 Opal Sep 1 Cleanest pieces. Most have no visible inclusions or surface contamination. Some pieces have one or two inclusions that look like Fe oxide. These can be segregated later if desired.

IR-99-10 Opal Sep 2
More "dirty" and contaminated than Sep 1, but could potentially be broken up to produce clean pieces.

IR-99-10 Opal Sep 3

Dirtier still - too dirty to bother trying to get clean pieces from, though it would be possible. "Contaminants" are chiefly Fe oxides - and perhaps other oxides (Mn?).

PREPARATION FOR LEACHING TEST

Will perform a leach of carbonate samples, analyzing leachate for U + Th. Will use enough 1M HNO_3 to dissolve carbonate component.
1 g $CaCO_3$ is 0.01 mole, so 0.01 mole HNO_3 is needed to dissolve.
1 g $CaCO_3$ → 10 ml 1M HNO_3
Can estimate how much carbonate is in a sample based on firing results, which yielded % carbonate. From Excel file.
Samples selected (1) if U-Th isotopic analysis is planned or (2) two grain size fractions had appreciably different [U], U/Th, or % carbonate.
Can also estimate maximum amount U in powder based on U-Th concentration data from Austin.
Amounts of powder used will be on the order of 100 mg (0.1 g), so assemble a table assuming this much powder for each sample.
Calculate (1) mass $CaCO_3$
(2) ml of 1 M HNO_3 needed to dissolve carbonate
(3) maximum mass of U in leachate.
(4) concentration of U if diluted to 30 ml in solution.

DAP 8/4/00

Note: mass of U in carbonate is maximum because concentration data are from total dissolution.

Calculations assuming 100 mg powder

Sample	% carbonate	(mg) mass carbonate	ml HNO_3	meas [U] ppm	soln ng U, max	soln max ng U	ppb U in 30 ml
NOP1-164-SEP1	43.2		0.43	28.0	~~1210~~	2800	93
NOP1-164-SEP4	68.8		0.69	33.8	~~2330~~	3380	113
NOP1-179-SEP1-P	85.0		0.85	9.20	~~782~~	920	31
NOP1-305-L1-SEP4	77.8		0.78	143	~~11100~~	14300	480
NOP1-305-L2-SEP1	73.6		0.74	192	~~14100~~	19200	640
NOP1-305-L2-SEP4	81.3		0.81	157	~~12800~~	15700	520
NOP1-306-SEP4	64.1		0.64	322	~~20600~~	32200	1070
NOP1-434-SEP2	95.8		0.96	83.9	~~8040~~	8390	280
NOP1-434-SEP4	92.2		0.92	83.2	~~7670~~	8320	280
NOP1-436-L1-SEP1	83.9	←	0.84	31.7	DAP	3170	106
NOP1-436-L1-SEP4	90.4	same	0.90	27.5	8/4/00	2750	92
NOP1-436-L2-SEP2	82.2		0.82	24.2		2420	81
NOP1-436-L2-SEP4	85.4		0.85	38.5		3850	128
NOP1-458-SEP1	7.4	←	0.074	112		11200	370
NOP1-458-SEP4	8.6		0.086	86.8		8680	290
~~NOP1-499-SEP~~ DAP 8/4/00	~~72.7~~			~~38.1~~			
NOP1-499-L2-SEP1	72.2		0.72	38.1		3810	127
NOP1-499-L2-SEP3	75.6		0.76	27.1		2710	90
NOP1-499-L3-SEP4	69.2		0.69	63.8		6380	210

DAP 8/4/00

8/4/00 Note: Error I originally make here was that I multiplied ~~calculated~~ U concentration times mass of carbonate. Actually the maximum mass in leachate is the total U in powder, or 100 mg x [U].

Weigh powder into small thin plastic centrifuge tube (3 ml). Amount weighed depends on what is available.

Append "-L" to sample number to indicate "leaching."

Want empty weight on tube so can weigh dried residue after leach.

Re-weighed see p. 151 DAP 8/9/00

Carbonate sample split for leaching

		re-done on 8/9/00	Using chart on p. 144, calculate ml 1M HNO_3
NOP1-164-SEP1-L			
+ powder	~~2.2421~~	2.2493	
empty tube	~~2.0361~~	2.0532	
	~~0.2060~~ g	0.1961 g	0.89 ml
NOP1-164-SEP4-L			
+ powder	~~2.2603~~	2.2061	
empty tube	~~2.0399~~	2.0312	
	~~0.2204~~	0.1749	~~1.52~~ 1.21
NOP1-179-SEP1-P-L			
+ powder	2.1572		
empty tube	2.0488		
	0.1084		0.92
NOP1-305-L1-SEP4-L			
+ powder	2.1684		
empty tube	2.0522		
	0.1162		0.91
NOP1-305-L2-SEP1-L			
+ powder	2.2623		
empty tube	2.0501		
	0.2122		1.57
NOP1-305-L2-SEP4-L			
+ powder	2.1572		
empty tube	2.0464		
	0.1108		0.90
NOP1-306-SEP4-L			
+ powder	2.2397		
empty tube	2.0501		
	0.1896		1.21

		ml 1M HNO_3
NOP1-434-SEP2-L		
+ powder	2.2582	
empty tube	2.0483	
	0.2099 g	2.02 ml
NOP1-434-SEP4-L		
+ powder	2.2416	
empty tube	2.0442	
	0.1974	1.82
NOP1-436-L1-SEP1-L		
+ powder	2.2533	
empty tube	2.0451	
	0.2082	1.75
NOP1-436-L1-SEP4-L		
+ powder	2.1632	
empty tube	2.0419	
	0.1213	1.09
NOP1-436-L2-SEP2-L		
+ powder	2.1704	
empty tube	2.0323	
	0.1381	1.13
NOP1-436-L2-SEP4-L		
+ powder	2.1553	
empty tube	2.0390	
	0.1163	0.99
NOP1-458-SEP1-L		
+ powder	2.1865	
empty tube	2.0442	
	0.1423	0.11

Re-weighed on p. 151-152 DAP 8/9/00

Re-weighed on p. 152 DAP 8/9/00

8/8/00 DAP

		ml 1M HNO_3
NOP1-458-SEP4-L		
+ powder	2.2301	
empty tube	2.0401	
	0.1900 g	0.16 ml
NOP1-499-L2-SEP1-L		
+ powder	2.1336	
empty tube	2.0275	
	0.1061	0.76
NOP1-499-L2-SEP3-L		
+ powder	2.1424	
empty tube	2.0417	
	0.1007	0.77
NOP1-499-L3-SEP4-L		
+ powder	2.1924	
empty tube	2.0427	
	0.1497	1.03

Weigh out splits of opal samples for U concentration analysis. Small amounts into 30 ml FEP Teflon beakers. Weigh by mass added to beaker (without parafilm cap).

IR-99-1 Opal 1 [U]

+ opal		13.43891
empty beaker	~~13.43690~~ DAP 8/8/00	13.43642
		0.00249 g

Place two drops of water on opal pieces to keep them in place in the beaker. Cover with parafilm.

There is a downward, slow drift in the last digit of the balance reading. Last digit is imprecise.

IR-99-1 Opal 2 [U]

+ opal 13.54318
empty 13.53947
0.00371 g

Add water drops and cover, as for previous.

IR-99-10 Opal Sep 1 [U] DAP 8/8/00

+ opal 13.32356
empty 13.31432
0.00924

Prepare a 4th beaker labelled "[U] Blank."

Use Fisher Trace Metal Grade concentrated HNO_3 and Fisher Reagent Grade HF (49%).

11:45 AM Add 1 ml HNO_3 to each beaker.
Add 0.5 ml HF to each beaker.

8/9/00 10:15 AM All three solutions appear clear.

Trying to separate pure uranophane from IR-99-1

Difficult, because when I scrape late-stage uph off a rock surface, the uph breaks into small pieces and there is a lot of contamination by other phases.

IR-99-1 Uph 1

From a fracture face in tuff, ~~within~~ DAP 8/8/00 with tuff above coated by clear opal. However; as shown on piece included in vial, uph needles grow both within and through the ~~tuf~~ 8/8/00 opal layer.
Therefore, not unequivocally youngest uph.
Have good needles, but they are small and there are grains of other phases.

IR-99-1 Uph 2

Growing on surface of outer opal, so does represent youngest uph. However, lots of contamination and uph grains are very small.

IR-99-1 Uph 3

Uranophane needles growing into a void in the clear opal. However, highly contaminated and uph grains are very small.

* IR-99-1 Uph 4

Smallest sample, but most pure. Best uph sample. Contaminants are present, but not abundant.

LEACHING TEST See pp 143-147.

Use 1.0 M HNO_3 (Fisher Trace Metal Lot 119020)
Pipet into tube containing powder in increments.

NOP1-164-SEP1-L : 500 µl + 200 + 200 Overflow! Sample lost
Start over with new powder (p. 144): 500 + 200 + 200 + 10 + 200 + 100 + 200 + 100 + 100 + 100 + 200
NOP1-164-SEP4-L : 1000 µl + 500 Overflow! Sample lost.
Problem: using a disposable pipet to stir up acid + powder (in and out). Get a sudden exposure of powder to acid, bubbles up and overflows.
Start over with new powder (p. 144): 500 + 500 + 200 + 20 + 200 + 100 + 100 + 100 + 200 + 200 + 100

small drop lost. Mixed

NOP1-179-SEP1-P-L: 500 µl + 200 + 200 + 200 + 100 + 100 + 100 + 200 + 200 + 100 + 100

NOP1-305-L1-SEP4-L: 500 + 200 + 200 + 200 + 100 + 100 + 100 + 200 + 200 + 100

I miscalculated the HNO_3 needed to dissolve by a factor of two.
Need two moles HNO_3 per mole $CaCO_3$.
∴ More needed than I thought.

To this point:
NOP1-164-SEP1-L = 1.91 ml
Did not see fizz after final 100 µl.

NOP1-305-L1-SEP4-L = 1.90 ml
Do not think I saw fizz after final 100 µl.

NOP1-164-SEP4-L = 2.22 ml
Still fizzing

NOP1-179-SEP1-P-L = 2.00 ml
Still fizzing.

Add water to 164-SEP1-L and 305-L1-SEP4-L to make ~ even with others.

~~Ber~~ DAP 8/9/00
Centrifuge in old Dynac machine, 10 minutes, set to 50.
Go to p. 153.

Because of problems with dissolution, I need larger centrifuge tubes.
Take the remaining 14 "-L" powders and pour into 50 ml plastic tubes, with conical bottoms. Weigh as before.

Replace weighings on pp 145-147

NOP1-305-L2-SEP1-L			1.0 M HNO_3 to dissolve
+ powder	13.8630		
empty centrifuge tube	13.6512		
	0.2118	g	3.12 ml
NOP1-305-L2-SEP4-L			
+ powder	13.7518		
empty	13.6449		
	0.1069		1.74
NOP1-306-SEP4-L			
+ powder	13.8715		
empty	13.6833		
	0.1882		2.41
NOP1-434-SEP2-L			
+ powder	13.8069		
empty	13.5989		
	0.2080		3.99
NOP1-434-SEP4-L			
+ powder	13.8967		
empty	13.7016		
	0.1951		3.59
NOP1-436-L1-SEP1-L			
+ powder	13.8819		
empty	13.6761		
	0.2058		3.46
NOP1-436-L1-SEP4-L			
+ powder	13.8034		
empty	13.6831		
	0.1203		2.17

NOPI-436-L2-SEP2-L
+ powder 13.6386
empty 13.5027
0.1359 g 2.23 ml

NOPI-436-L2-SEP4-L
+ powder 13.7887
empty 13.6749
0.1138 1.93

NOPI-458-SEP1-L
+ powder 13.8108
empty 13.6711
0.1397 0.21

NOPI-458-SEP4-L
+ powder 13.8494
empty 13.6606
0.1888 0.32

NOPI-499-L2-SEP1-L
+ powder 13.7733
empty 13.6690
0.1043 1.50

NOPI-499-L2-SEP3-L
+ powder 13.7648
empty 13.6675
0.0973 1.48

NOPI-499-L3-SEP4-L
+ powder 13.8350
empty 13.6878
0.1472 2.03

Continue with first four samples (from p. 150).
Centrifugation was incomplete.
Put in "Marathon 21K" centrifuge, set to 6 (5900 RPM) for 10 minutes.
Centrifugation again incomplete, I think.
Set 7 for 15 minutes.
Still not sure. Material adhering to walls makes soln. appear cloudy.
Start pipetting out to see if clear

NOPI-164-SEP1-L Leachate 30 ml PE bottle
empty bottle 8.0363 g

NOPI-164-SEP4-L Leachate
empty bottle 7.9528

☆ NOPI-179-SEP1-P-L Leachate ☆ New bottle on p. 159
empty bottle 7.9861 AP 8/14/00

NOPI-305-L1-SEP4-L Leachate
empty bottle 7.9475

164-SEP1 and 164-SEP4 had clear supernate. Others did not.
179-SEP1P is still bubbling.
179-SEP1-P and 305-L1-SEP4. Stir up with pipet, let settle until bubbling stops (179), then centrifuge again.
Go back to Dynac centrifuge. 10 minutes at 90 setting.

164-SEP1 and 164-SEP4: add 500 µl 0.1 M HNO_3. See a little fizzing in each.
No – not fizzing: Particles in a return flow up the tube – downward flow on other side.
Add another 500 µl 0.1 M HNO_3 to each.
Centrifuge in Dynac, 10 mins., "90."

Pipet supernate from all four. (Still a little bubbling in 179)

To all four, add 1.5 ml 0.1 M HNO_3. Stir up with pipet. I see no bubbling in any.
Centrifuge 10 mins., "90" setting.

After pipetting supernate into Leachate bottle, add first 200 µl 1.0 M HNO_3 to check for bubbling. None. Then add 1.5 ml 0.1 M HNO_3, stir up, centrifuge as before.

Add 1.5 ml 0.1 M HNO_3 to 179-SEP1-P and 305-4-SEP4. Stir up, centrifuge.

Note: pipets used for agitating sample have residue of solid adhering to walls. Thus, some insoluble residue is not still in tube.

Note: The two NOP1-164 leachates are clear. 179 and 305-4 have a little yellow color. Does this mean there is little Fe in 164? Or is it in an insoluble form? XRD may help.

8/10/00 SPCØØ543702

This is a Yucca Mountain region calcite-matrix fault breccia. SDS is interested in U-Th dating.
Break off some chips and grind by hand in agate mortar & pestle. Do not grind a lot - want to break down only the soft calcite. There is a lot of tuff fragments.
Sieve at 63 µm, by hand

SPCØØ543702-U

This is the <63 µm fraction.

Will add this to leach test.
Also, add NOP1-501-G-SEP3, because this may be analyzed for U-Th isotopes at UT-Austin

Weigh both into 50 ml, blue capped centrifuge tubes.

[U] = 13.3 ppm NOP1-501-G-SEP3-L — 1.0 M HNO_3 to dissolve

+ powder	13.7571	
empty tube	13.5512	
	0.2059 g	4.12 ml

SPCØØ543702-U DAP 8/10/00

+ powder	13.9530	
empty tube	13.4975	
	0.4555	9.11 ml

Bring first four leachates up to 10 ml with 0.1 M HNO_3.
Weigh: (see empty weights on p. 153)

NOP1-164-SEP1-L Leachate =	17.9780 g	
NOP1-164-SEP4-L Leachate =	18.1182	
NOP1-179-SEP1-L Leachate =	18.1246	*
NOP1-305-L1-SEP4-L Leachate =	17.9705	

* I can see that there are settled solids in here. These solutions contain ~8 ml un-neutralized 0.1 M HNO_3. Note: see p. 159. DAP 8/14/00

Back to opal solutions — see p. 147-148
Pipet solutions - which are all clear - into 30 ml PE bottles.

	Empty bottles →	+ solution 8/11/00
IR-99-1 Opal 1 [U]	8.0289 g	12.0303 g
IR-99-1 Opal 2 [U]	7.9670	12.1026
IR-99-10 Opal Sep 1 [U]	7.9842	12.4152
Blank [U]	7.9646	11.8722

After transfer to bottle, add 250 µl 50% HF to dissolution beaker, swirl, pipet into bottle with transfer pipet. Repeat twice more.
Repeat three rinses in same fashion with 250 µl concentrated HNO_3.
Record weight above.
Nominal Total: 1.25 ml HF, 1.75 HNO_3. This doesn't match weights. ?
Wait: I add water initially - p. 147. OK.

8/11/00

Continue leaching (pp 151-152) for sample info.

NOP1-305-L2-SEP10-L

1.0 M HNO_3 = 1000 µl + 1000 + 500 + 200 + 200 + 200 + 200 No fizz on last.
0.1 M HNO_3 = 1000 µl - Centr - (200 µl 1.0 M - no fizz) + 1500 µl - Centr -
H_2O = 5 ml - Centr.

NOP1-305-L2-SEP4-L

1.0 M HNO_3 = 1000 µl + 500 + 200 + 200 No fizz on last 200.
0.1 M HNO_3 = 2000 µl - Centr - (200 µl 1.0 M - no fizz) + 1500 µl - Centr -
H_2O = 5 ml - Centr.

NOP1-306-SEP4-L

1.0 M HNO_3 = 1000 µl + 1000 + 200 + 200 + 200 No fizz on last 200.
0.1 M HNO_3 = 1500 µl - Centr - (200 µl 1.0 M - no fizz) + 1500 µl - Centr -
H_2O = 5 ml - Centr.

NOP1-434-SEP2-L

1.0 M HNO_3 = 1000 µl + 1000 + 1000 + 500 + 300 + 200 + 200 + 200 No fizz
- Centr - {200 µl 1.0 M (no fizz)} + 1500 µl 0.1 M HNO_3. - Centr -
H_2O = 5 ml Centr.

First centrifugation, marked above by "Centr." 0.1 M HNO_3 to 1st three to get to ~4 ml. Dynac centrifuge, 10 mins., set to 90.

Weigh empty leachate bottles (30 ml PE)

NOP1-305-L2-SEP1-L leachate = 7.9473 g
NOP1-305-L2-SEP4-L Leachate = 7.9631
NOP1-306-SEP4-L Leachate = 8.0050
NOP1-434-SEP2-L Leachate = 7.9894

After 1st centr., each solution has particles floating on it. I don't have time now to re-centrifuge, so leave as is, with leachate in tube over residue.

14:45

8/13/00 DAP

Centrifuge again, 15 mins, "90"
Pipet supernate, then continue as recorded above. All four solns are yellow. Final rinse is H_2O.
After finished, add 250 µl 1.0 M HNO_3 to each residue. No fizz. Leave acid in tube with residue.

Next four powders for leaching - see p. 151-152.

NOP1-434-SEP4-L

1.0 M HNO_3 = 1000 µl + 1000 + 1000 + 300 + 300 + 250 no fizz - Centr -
250 µl, no fizz + 0.1 M HNO_3 2000 - Centr - 5 ml H_2O - Centr.

NOP1-436-L1-SEP1-L

1.0 M HNO_3 = 1000 µl + 1000 + 1000 + 300 + 200 + 250 + 250 no fizz - Centr -
250 µl, no fizz + 0.1 M HNO_3 2000 - Centr - 5 ml H_2O - Centr.

NOP1-436-L1-SEP4-L

1.0 M HNO_3 = 1000 µl + 500 + 500 + 200 + 250 + 250 no fizz - Centr -
250 µl, no fizz + 0.1 M HNO_3 2000 - Centr - 5 ml H_2O - Centr

NOP1-436-L2-SEP2-L

1.0 M HNO_3 = 1000 µl + 500 + 500 + 250 + 250 + 250 no fizz - Centr -
250 µl, no fizz + 0.1 M HNO_3 2000 - Centr - 5 ml H_2O - Centr.

All four: after final, centrifuge, add 250 µl 1.0 M HNO_3. No fizz.

Weigh empty 30 ml PE bottles for leachate:

NOP1-434-SEP4-L = 7.9675 g
NOP1-436-L1-SEP1-L = 7.9648
NOP1-436-L1-SEP4-L = 7.9784
NOP1-436-L2-SEP2-L = 7.9835

Empty bottles for next set of four:

NOP1-436-L2-SEP4-L = 7.9256 g
NOP1-458-SEP1-L = 7.9801
NOP1-458-SEP4-L = 7.9907
NOP1-499-L2-SEP1-L = 7.9905

Of these four, NOP1-434-SEP4-L is the only one with a strong yellow color. The other just have a slight tinge.
Thought: residues look more massive than expected (e.g. NOP1-434-x) based on firing results. Maybe they also lost a lot of water, so carbonate component is less than calculated.
First two sets of four: Put tubes holding residue in 50°C oven.

DAP 8/13/00

~~Next~~ Next four powders for leaching:

NOP1-~~4~~-L2-SEP4-L

1.0 M HNO_3 = 500 µl + 500 + 500 + 250 + 250 + 250, no fizz – Centr –
+ 250 µl 1.0 M HNO_3, no fizz + 2 ml 0.1 M HNO_3 – Centr –
5 ml H_2O – Centr.

NOP1-458-SEP1-L

1.5 ml H_2O + 200 µl 1.0 M HNO_3, no fizz + 250, no fizz – Centr –
+ 250 µl 1.0 M HNO_3, no fizz + 2 ml 0.1 M HNO_3 – Centr –
+ 5 ml H_2O – Centr.

NOP1-458-SEP4-L

1.5 ml H_2O + 300 µl 1.0 M HNO_3, no fizz + 250, no fizz – Centr –
+ 250 µl 1.0 M HNO_3, no fizz + 2 ml 0.1 M HNO_3 – Centr.
+ 5 ml H_2O – Centr.

NOP1-499-L2-SEP1-L

1.0 M HNO_3 = 500 + 500 + 250 + 250 + 250, no fizz – Centr.
+ 250 µl 1.0 M HNO_3, no fizz + 2 ml 0.1 M HNO_3 – Centr.
+ 5 ml H_2O – Centr.

8/14/00 DAP

NOP1-499-L2-SEP3-L

1.0 M HNO_3 = 500 µl + 500 + 300 + 200 + 250 no fizz + ~500 H_2O – Centr –
250 µl no fizz + 2000 0.1 M HNO_3 – Centr – 5 ml H_2O – Centr

NOP1-499-L3-SEP4-L

1.0 M HNO_3 = 500 µl + 500 + 500 + 300 + 200 + 250 no fizz – Centr –
250 µl no fizz + 2000 0.1 M HNO_3 – Centr – 5 ml H_2O – Centr

NOP1-501-G-SEP3-L

1.0 M HNO_3 = 1000 µl + 1000 + 1000 + 1000 + 200 + 250 no fizz + ~5 ml H_2O – Centr –
250 µl ⇒ fizzes + 250 ⇒ fizzes + 250 ⇒ fizzes + 250 no fizz
+ 2000 µl 0.1 M HNO_3 – Centr – 5 ml H_2O – Centr

SPC00543702-U

1.0 M HNO_3 = 1000 µl + 1000 + 1000 + 1000 + 1000 + 1000 + 1000 + 1000 + 1000 + 500 no fizz
– Centr. + 250 µl no fizz + 250 + 0.1 M HNO_3 → 2000.
– Centr – 5 ml H_2O – Centr.

Weigh empty 30 ml PE bottles for leachates

NOP1-499-L2-SEP3-L	=	7.9491 g	Leachate
NOP1-499-L3-SEP4-L	=	7.9899	"
NOP1-501-G-SEP3-L	=	7.9639	"
SPC00543702-U	=	7.9770	"

NOP1-179-SEP1-P-L Leachate Need to get rid of settled solids. Pipet into centrifuge tube & centrifuge 15 mins at "90." Supernate into new bottle.

* Need new 30 ml PE bottle
empty 8.0332 g

Put 1 ml 0.1 M HNO_3 into old leachate bottle, swirl. Much of the remaining residue is stuck to bottle wall. Squirt acid onto residue with pipet. Pipet acid into centrifuge tube. Repeat twice more. Total of 3 ml 0.1 M HNO_3 used to rinse old bottle into c. tube. Centrifuge 15 mins, "90." Add supernate to new leachate bottle.

Want to ensure stability of leachate. Add 0.5 ml 1.0 M HNO_3 to each of the 20 solutions prepared since 8/9/00. This ensures that all but a few will be > 0.05 M HNO_3; most will actually be >0.10 M. A few are guaranteed to be only > ~~0.02 M~~ 0.03 M, which is still OK. DAP 8/14/00

Final weight of leachate solns in bottles DAP 8/14/00

Empty bottle weights recorded on pp. 153, ~~155~~, 156, 157, 159

	Final wt.	empty	soln wt.
NOP1-164-SEP1-L Leachate	18.4932 g	8.0363 g	10.4569 g
NOP1-164-SEP4-L Leachate	18.6349	7.9528	10.6821
NOP1-179-SEP1-P-L Leachate	21.6797	8.0332	13.6465
NOP1-305-L1-SEP4-L Leachate	18.4844	7.9475	10.5369
NOP1-305-L2-SEP1-L Leachate	18.7849	7.9473	10.8376
NOP1-305-L2-SEP4-L Leachate	18.9980	7.9631	11.0349
NOP1-306-SEP4-L Leachate	19.3339	8.0050	11.3289
NOP1-434-SEP2-L Leachate	20.3318	7.9894	12.3424
NOP1-434-SEP4-L Leachate	19.8651	7.9675	11.8976

	Final wt.	empty	soln wt
NOP1-436-L1-SEP1-L Leachate	19.6727 g	7.9648 g	11.7079 g
NOP1-436-L1-SEP4-L Leachate	18.5604	7.9784	10.5820
NOP1-436-L2-SEP2-L Leachate	18.9965	7.9835	11.0130
NOP1-436-L2-SEP4-L Leachate	18.7512	7.9256	10.8256
NOP1-458-SEP1-L Leachate	17.7248	7.9801	9.7447
NOP1-458-SEP4-L Leachate	17.1255	7.9907	9.1348
NOP1-499-L2-SEP1-L Leachate	17.7314	7.9905	9.7409
NOP1-499-L2-SEP3-L Leachate	18.2773	7.9491	10.3282
NOP1-499-L3-SEP4-L Leachate	17.7131	7.9899	9.7232
NOP1-501-G-SEP3-L Leachate	26.3509	7.9639	18.3870
SPC00543702-U Leachate	26.2075	7.9770	18.2305

(3rd column from subtracting 2nd from 1st. Subtraction checked by adding 3rd + 2nd to get 1st.)

Put centrifuge tubes containing residues into 50°C oven at ~ 11:00 AM and 3:00 PM.

Solutions with strongest yellow color: 434-SEP2 and 434-SEP4.
Others: 305-L1-SEP4, 305-L2-SEP1, 305-L2-SEP4, 306-SEP4, 436-L1-SEP1, 499-L3-SEP4
(Moderate color in 305-L2-SEP1, 306-SEP4, 499-L3-SEP4; others are light.)
A few others have a slight tinge of yellow.
Analyze Fe in solns.

8/15/00 Measure weight of centrifuge tubes after removing from 50°C oven at 15:00 (were in oven > 1 day).
This weight will be subtracted from empty tube weight (pp. 145, 151-152, 155).
The net weight is the dry weight of the residue remaining after leaching.

	Final wt centrifuge tube
NOP1-164-SEP1-L	2.1649 g
NOP1-164-SEP4-L	2.0933
NOP1-179-SEP1-P-L	2.0632
NOP1-305-L1-SEP4-L	2.0815
NOP1-305-L2-SEP1-L	13.7096
NOP1-305-L2-SEP4-L	13.6642
NOP1-306-SEP4-L	13.7573
NOP1-434-SEP2-L	13.6137
NOP1-434-SEP4-L	13.7230
NOP1-436-L1-SEP1-L	13.7135
NOP1-436-L1-SEP4-L	13.6976
NOP1-436-L2-SEP2-L	13.5335
NOP1-436-L2-SEP4-L	13.6961
NOP1-458-SEP1-L	13.8101
NOP1-458-SEP4-L	13.8474
NOP1-499-L2-SEP1-L	13.7025
NOP1-499-L2-SEP3-L	13.6962
NOP1-499-L3-SEP4-L	13.7393
NOP1-501-G-SEP3-L	13.5560
SPC00543702-U	13.5925

Entries relevant to this work are continued in notebook #172, on page 13.
DAP 8/16/00

Final Entry:
I have reviewed this notebook. It complies with QAP-001. There is sufficient information for another qualified person to reproduce the activities. R.C. Ray 8/29/00

6/19/2003 DAP

Additional note: The diskettes attached to this notebook were in Macintosh format. I opened the disks on my Windows NT 4.0 PC and converted all files to Windows-readable formats: Excel, Kaleidagraph, and text (for *.30 files). The files were burned to a CD under two directories corresponding to the original Mac diskettes:

"Sci Notebook 127 Disk 1 files" - corresponds to Disk #1, named "Nopal Water Chemistry."

"Sci Notebook 127 Disk 2 files" - corresponds to Disk #2, named "Uranophane W/Pb."

ADDITIONAL INFORMATION FOR SCIENTIFIC NOTEBOOK #: 127

Document Date:	07/26/1995
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Remarks: (computer runs, etc.)	Media contains: Nopal water chemistry; uranophane U/pb; various output files