# STANDARD OPERATING PROCEDURE NO. 25
# STABLE ISOTOPE ANALYSES

| REVISION LOG | | |
|---|---|---|
| Revision Number | Description | Date |
| 25.0 | Original SOP | Nov. 26, 2003 |
| 25.1 | Revisions by Andy Campbell, McLemore rewrites section 2 and 8 | Dec. 2, 2003 |
| 25v2 | Revisions by PJP | 5/19/2004 |
| 25v2 | Finalized by LMK to post on Molycorp project website and to send to George Robinson for lab audit; LMK did not edit this SOP | 3/27/07 |
| 25v3 | Editorial by SKA | 10/23/08 |

## 1.0 PURPOSE AND SCOPE

This standard operating procedure (SOP) provides technical guidance and methods that will be utilized in the preparation and analysis of water and solid material for δD, $\delta^{18}O$, and $\delta^{34}S$ stable isotopes. This procedure gives descriptions of personnel responsibilities, related SOPs, field and laboratory equipment, field sample protocols, sample preparation, sample analysis, and laboratory quality assurance/quality control (QA/QC) and documentation procedures.

## 2.0 RESPONSIBILITIES AND QUALIFICATIONS

The Geological and Hydrological Characterization Study (GHCS) Lead Principal Investigator (PI) or Task 12 and 13 PI's have the overall responsibility for implementing this SOP. They will be responsible for assigning appropriate staff to implement this SOP and for ensuring that the procedures are followed. These laboratory procedures are written for personnel trained in the use of the mass spectrometer and sample preparation equipment.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the

procedures described within this SOP, and receive specific training regarding these procedures, if necessary.

All environmental staff and assay laboratory staff are responsible for reporting deviations from this SOP to the Team Leader, GHCS Lead PI or Task 12 and 13 PI's.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the procedures described within this SOP and receive specific training regarding these procedures, if necessary.

## 3.0 DATA QUALITY OBJECTIVES

This SOP address objectives 1, 2, 3, and 7 in the data quality objectives outline by the GHCS Lead PI. These objectives are listed as follows, respectively:

- Determine how the hydrogeochemistry and water balance dynamics influences rock pile weathering and stability.
- Determine how mineralogy, stratigraphy, and internal structure of the rock piles contribute to weathering and stability.
- Determine if the sequence of host rock hypogene and supergene alteration and weathering provides a basis to predict the effects weathering can have on mine rock stability.
- Determine if pyrite oxidation, moisture content, and microbe populations affects rock pile weathering and stability.

## 4.0 RELATED STANDARD OPERATING PROCEDURES

The procedures for stable isotope sampling and analysis set forth in this SOP are intended for use with the following SOPs:

- SOP No. 2 Sample management
- SOP No. 3 Surveying (GPS)
- SOP No. 4 Taking photographs
- SOP No. 5 Sampling outcrops and drill core (solid)
- SOP No. 6 Drilling, logging, and sampling of subsurface materials (solid)
- SOP No. 8 Sample preparation (solid)
- SOP No. 9 Test pit excavation, logging, and sampling (solid)
- SOP No. 15 Surface water and seep sampling
- SOP No. 16 Ground-water sampling

# 5.0 EQUIPMENT LIST

## 5.1 FIELD EQUIPMENT

The following materials and equipment may be necessary for water and solid material sampling for stable isotope analysis:

- Sample bags and/or water vials
- Bound field logbook
- Sampling site location maps
- Field Sample Forms
- Surveying stakes or flags for marking of grid nodes and/or sampling locations
- Personal protective equipment (PPE) as outlined in the HASP.
- Rock hammer
- Sample labeling and handling materials (e.g., waterproof markers, sample labels, chain of custody [COC] forms, clear plastic tape, custody seals)
- GPS Unit
- Shovel
- Camera, film, and/or digital camera, and photograph forms
- Site Plans, Topographic Maps, or Aerial Photographs

Other site specific materials and equipment may be needed based on field conditions. The field sampling equipment list of this SOP should be used in co-ordinance with those of SOP Numbers 2, 5, 6, 9, 15, and 16.

## 5.2 LABORATORY EQUIPMENT

The following materials and equipment may be necessary for water and solid material stable isotope sample preparation and analysis:

- Sample preparation equipment
  - Ceramic mortar and pestle
  - 1% sodium-hexa-meta-phosphate solution
  - 0.5N NaOH solution for dissolving alunite-jarosite
  - Standard laboratory sieves
  - Ultrasonic cleaner/probe

  - Hot plate with stirring bars
  - 250 and 500 ml beakers
  - Centrifuge
  - 0.4 Nuleopore filter paper
  - Distilled water
  - Graduated cylinder
  - 10N HCl
  - 0.2N NaOH solution to adjust pH
  - Centifuge tubes
  - 0.5N $BaCl_2$ solution
- Finnigan Delta Plus XL Mass Spectrometer
- Computer and Isodat NT software
- Gas bench and autosampler
- Costech Elemental Analyzer and autosampler
- H/Device and autosampler

## 6.0 COLLECTION OF SAMPLES

Sample collection for stable isotope analysis is outlined in SOP No. 2, Sample Management, SOP No. 5, Sampling outcrops and drill core (solid), SOP No. 6, Drilling, logging, and sampling o f subsurface materials (solid), SOP No. 9, Test pit excavation, logging, and sampling (solid), SOP No. 15, Surface water and seep sampling, and SOP No. 16, Ground-water sampling.

## 7.0 FIELD QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES AND SAMPLES

QA/QC samples are designed to help identify potential sources of sample contamination to evaluate potential error introduced by sample collection and handling. In the case of stable isotope analysis, this type of contamination is not applicable. Hence, no field QA/QC samples are collected for stable isotope analysis.

## 8.0 SAMPLE HANDLING

Sample containers and analyses are specified in SOP No. 2, Sample Management. Samples will also be labeled and handled as described in SOP No. 2. Chain of custody forms will be completed.

## 9.0 SAMPLE PREPARATION

### 9.1 Water

### 9.1.1 Water

No sample preparation needed for water samples.

### 9.1.2 Soil Water Extraction

Water can be extracted from soil using a fairly simple cryogenic distillation technique. Water is removed from the soil sample by slow heating of the soil under vacuum while collecting the evolved water by freezing. Water must be removed through this method in order to analyze it for both hydrogen and oxygen isotopes.

- Use clean and dry 100ml and 1000ml round bottom flasks.
- Weigh the 100ml flask in a 250ml beaker and record in logbook.
- Weigh the 1000ml flask balanced in a petri dish and record in logbook (the weights of the petri dish and the 250ml beaker do not matter because we are looking for changes in weight).
- Place plastic funnel in 1000ml flask neck and add soil to flask. Depending on how wet the soil is, you use between 120 grams to 250 grams of soil.
- Remove plastic funnel and record weight of flask and soil under "vessel + sample".
- Quickly take both flasks and attach them to the transfer tube using Apiezon vacuum grease H. Make sure the stopper on the transfer tube is closed.
- Place the 1000ml flask into liquid nitrogen bath in a styrofoam container. Refill with nitrogen as needed. Wait until soil is frozen (approximately 20 minutes for average samples, or until soil looks frosty).
- Attach to vacuum pump with plastic tubing. Open all valves and pump apparatus out to approximately 100 millitorr pressure.
- Close all valves and disconnect apparatus from pump (after disconnected and turned off, make sure to re-open the vacuum pump to atmosphere).
- Remove 1000ml flask from liquid nitrogen and place in heater. Place 100ml flask into liquid nitrogen bath.
- Turn heater on to just below boiling (#5 on our heaters). Refill liquid nitrogen bath as needed to keep the 100ml flask cold, but not let ice build in the neck.
- If water begins to condense on the walls of the transfer tube, chase it through with the heat gun.
- Wait at least 2 hours or until the soil appears dry or baked, and no more water condenses on the walls of the transfer tube (if soil is very dry, you do not necessarily need to wait 2 hours, 1.5 is enough). Wait 20 minutes after you last see water in the transfer tube.
- Make sure the 100ml flask is not frozen to the transfer tube (heat it a little with the heat gun if necessary).
- With a piece of parafilm at hand, open the stopper on the transfer tube to release vacuum. Pull off the 100ml flask and quickly cover with parafilm. Label and set aside to thaw.

- Turn off heater and remove the 1000ml flask. Weigh the dry soil in the flask using the same petri dish as before. Record in logbook under "dry soil".
- Wait for the ice to thaw to water. Once thawed, weigh in the same 250ml beaker used before. Record in logbook under "water".
- Store water in glass bottle with polyseal top.
- To determine % water in sample:
- Determine wet sample weight by subtracting the 1000ml weight from the "vessel+sample" weight.
- Determine dry sample weight by subtracting the 1000ml weight from the "dry soil" weight.
- Determine the water weight by subtracting the 100ml weight from the "water" weight.
- Divide the water weight by the wet sample weight and multiply by 100 for % water.

## 9.2 Solids

### 9.2.1 Sulfides/Sulfates

Sample preparation for sulfides/sulfates is as follows:

- Separate the sulfide/sulfate mineral from the sampled media
- Crush the sulfide/sulfate grains with a mortar and pestle to a fine powder
- Try to have 2 mg **minimum** of sample to 4 mg. If this is not possible, 0.80 mg is needed for analysis.
- Put in sample storage vessel and label.

### 9.2.2 Jarosite/Alunite

The goal is first to obtain a high purity concentrate of alunite/jarosite and clay from samples containing mixtures of the two. Following the separation of contaminant minerals, a wet chemical technique is employed to prepare the alunite/jarosite separate for $\delta^{18}O_{SO4}$ analysis.

#### 9.2.2.1 Mineralogical Separations

**Mineral Separation for stable isotope and $^{40}Ar/^{39}Ar$ radioisotope samples**:

- Hand pick coarse grain fractions of alunite/jarosite from veins or phenocryst replacements.
- Check hand-picked minerals for purity via x-ray diffraction
- Impure samples require alunite/jarosite separation from clay (listed below).
- On pure samples, dissolve alunite/jarosite (listed below).

**Alunite/Jarosite - Clay Separations:**

- Crush approximately 50 g of sample lightly in a ceramic mortar and pestle.

- Sieve, label and store >0.5 mm fraction for future processing (if necessary).
- Weigh 5-20 g of the <0.5 mm fraction into a 250 ml beaker. Add 150-200 ml distilled water and shake slowly in a mechanical shaker overnight – or use a magnetic stirrer.
- Test the pH of the solution with litmus paper. If necessary adjust pH of solution by adding 0.2N NaOH to near neutral (pH 7).
- Let stand after shaking/stirring to see if material flocculates. Add a few ml (always less than 5% by volume) of a 1% sodium-hexa-meta-phosphate solution if material flocculates.
- Use a ultrasonic probe in an ice bath to disperse clay particles for about 15 minutes.
- Separate the > 40 μm by wet sieving. Use a 500 ml beaker and a small glass funnel with a long stem to support a 325 mesh sieve. The sieve should be made of disposable nylon to reduce contamination. Allow the freshly stirred suspension to stand for 40 sec. For each 10 cm of its depth before decanting through sieve.
- Separate the 40 to 10 μm size fraction by taking the suspension of particles from step 7 and transfer to a graduated cylinder, shake, and allow to stand quietly for at least 150 seconds (but not over 200 seconds) for each 1 cm depth. The supernatant liquid is then decanted into a beaker. The sediment portion is brought into suspension again by the addition of distilled water and diluted to a depth of 5 cm. Allow to settle and decant supernatant liquid again after 12 minutes. The sediment is again dispersed a third time with distilled water and allowed to settle for 9 minutes.
- Separation of the 10-2 μm fraction via centrifuge or column settling.
  - Centrifuge technique – The suspension resulting from the 10 μm separation is transferred to centrifuge tubes to a suspension depth of 10 cm. Centrifuge for 4 min. at 750 rpm and decant supernatant liquid into a large flask. The sediment in the bottom of each tube is suspended into a small portion of distilled water and the suspensions of several tubes are transferred into one (if possible). Centrifuge tube at 750 rpm for 3.2 minutes. The processes of resuspension, centrifugation, and decantation are continued until the supernatant suspension is quite clear. The fine material remaining at the bottom of the centrifuge tube is washed into a ceramic evaporating dish lined with plastic wrap and allowed to dry in a vacuum oven at 50 degrees C.
  - Column settling technique – Syphon supernatant liquid after allowing to stand more than 8 hours for each 10 cm of liquid.
- Separation of the 0.2 μm fraction via centrifuge. The apparatus and procedure is identical to step 9 except for speed and time in the centrifuge. The first set of suspensions, brought to a depth of 10 cm is centrifuged for 30 minutes at 2400 rpm. For subsequent centrifuge periods, use 2400 rpm and 25 minutes – 3 centrifugations will suffice.

#### 9.2.2.2 Dissolution of Alunite/Jarosite in NaOH

- Weigh the alunite/jarosite bearing sample and place in a 400 ml beaker. 80 – 120 mg of pure material is a convenient amount that will provide enough sample for all analyses and

replication (if required). Sample should be sized to 120-325 mesh to assure complete dissolution.

- Fill beaker with 150 ml of freshly prepared 0.5N NaOH solution.
- Heat sample on hot plate at 80 degrees C for 3 hours – checking occasionally for dissolution of alunite/jarosite.
- Cool samples for filtering.
- Filter samples using a large diameter 0.4 ml Nucleopore filter paper. Save the filtrate and return it to the beaker after rinsing beaker with distilled water to remove any solid residue. Heat and stir at 80 degrees C.
- Measure the pH of the hot, filtered solution in the beaker and quickly acidify the solution by adding 10N HCl. The object is to make the solution reach a pH of 2.8 to 2.95 rapidly to prevent the precipitation of $Al(OH)_3$ at intermediate pH. If the solution becomes too acidic, back titrate with 0.5N NaOH to 2.8 –2.95.
- Immediately add one squirt of 0.5N $BaCl_2$ solution to precipitate $BaSO_4$. Continue stirring while heating. This will coarsen the precipitate and make final filtration much easier. Add another squirt of 0.5N $BaCl_2$ about 30 minutes after the initial application. Continue heating for 1 – 3 hours.
- Acidify the solution to prevent formation of Al(OH)3 upon cooling. A few drops of 2N HCl is sufficient.
- Before filtering, add a few ml of 10N HCl to each beaker. This will make filtration faster. Filter, dry, weigh, and record yield

# 10.0 PROCEDURES FOR STABLE ISOTOPE ANALYSES

## 10.1 Water

### 10.1.1 Hydrogen

The H-device is used to sample and react water for hydrogen analysis on the mass spec. The reaction is a chromium reduction reaction that takes place in a chromium packed reactor at 850$^{o}$C. The H-device is used in conjunction with the autosampler and the dual inlets on the front of the Delta Plus XP.

**Lab Sequence:**

- The lab has a specific sequence for running hydrogen samples because we make a new correction factor for each run. This is important for your data. Each sequence begins with 2 NM-2 samples, then each OH standard is run once. NM-2 is run once every 10 samples, and again as the last sample of the run. The entire process of running one sample including autosampler time and reaction time is approximately 15 minutes. Please plan your run according to this lab sequence.

**Loading samples:**

- Load anywhere from .5mL to 1mL water into the small H-Device bottles with labels. Do this using a separate insulin syringe for every sample.
- Make sure to write your sample name as well as the lab ID and the bottle number (VERY IMPORTANT) into the H-Device extraction book.
- Write the bottle number on the bottle with a PENCIL.
- Don't forget to load each OH standard and NM-2 each time you run a set of samples. Use only .5mL or less of the OH waters please.

**FIRST on the mass spec:**

- Make sure both inside valves are closed for the dual inlet configuration.
- Switch the air compressor so it is connected to the outside valve and NOT the inside valve.
- Attach the H-Device to the left inlet on the outside dual inlet.
- Attach reference H2 gas to the right side of the inlets, and make sure that gas (HD-1 or HD-2) is selected in the method under Evaluation@H2.

**Putting reference gas into the mass spec:**

- Pump out the right bellows first with the rough pump and then with the turbo pump (when the rough pump is down to $x10^{-3}$), make sure you pump out with the middle valve on the gas inlet open.
- Close off all valves, open the gas so it only expands into the small volume between the first and middle valves, and close the container after a couple seconds. Then open the middle valve and open the inside valves using the computer and let the gas expand into the bellows.
- Close all valves again and make sure to close the middle valve on the gas inlet.
- We want to start the run with the pressure gauge on the right-hand bellows reading approximately 40. To get it to 40, close the pump and the pump to the H-device, then expand the gas from the right bellows into the left bellows as well. Then compress the left bellows as needed to achieve a pressure of 40 in the right-hand bellows. Once this is done, keep the gas in both bellows to use for the zero enrichment of the day.

Set the H-Device up to 850$^{o}$C, increasing only by ~150$^{o}$ every few minutes. Once the device is at temperature, let it sit for at least 10 minutes before starting the run. To pack a reactor, please see Nelson and Dettman, 2001.

**On the computer in the Isodat Acquisition program:**

- Make sure the configuration is on DI+HDevice+Sampler
- Make sure measurement is set on H2
- Start by running the H3 factor; press the H3 button on the toolbar. Choose to run 2 or 3 times, not 5. Make sure to look at the last value used!

- After the H3 factor is finished, choose the number you wish to use for the day. Choose the number closest to the previous value used.
- Run a "zero enrichment" for the day; select the NMT HDzero sequence and highlight one line from the sequence. Press start, and make sure to run only selection.
- Select the NMT HDevice with sampler sequence
- Write all the identifiers you want in the sequence chart
- Make sure the AS Sample number matches each slot you put the sample in
- Make sure in each sample you want to run the AS Method is set to Internal no. 8 and the method is set to NMT HDevice with sampler.met
- When everything else is ready, press START
- When the menu comes up, select to save under the file name: HD00-00-00 (date)
- If you want to measure a selection only, highlight the selection and select measure selection only
- Select whether you want one printout for the entire sequence, or one printout per sample
- When everything else is ready, press OK
- There is a lab sequence that you must follow. This begins each run with 2 NM-2, then each of the OH standards, then 1 NM-2 every 10 samples, and end with NM-2. Use only one bottle of NM-2, but make sure the autosampler knows to go back to this bottle each time.

### 10.1.2 Oxygen

We use an equilibration procedure to measure the isotopes of oxygen in water. This involves using a measured amount of $CO_2$ gas and equilibrating it with a measured amount of water at higher than room temperature. Once the gas and the water are equilibrated, the gas is sampled and measured by the mass spectrometer.

In our lab, sample bottles are put into a heating block which can be used by the autosampler. The autosampler is used to extract the $CO_2$ gas after the equilibration. It is used in conjunction with the Gasbench and the Delta Plus XP. The equilibrated $CO_2$ is measured against a reference gas by continuous flow using the Gasbench.

**Loading Samples:**

- Set heater on autosampler block for 35$^{o}$C.
- Fill an appropriate number of sample vessels with $CO_2$ in He. Invert the sample vessels over the outlet of the $CO_2$ in He tank (it should be equipped with a thin tube that points upright) with the tank and regulator opened such that a slight breeze of gas can be felt coming out of the sample vessel. Let the gas flow into the sample vessel for five seconds, then lift the sample vessel off the tube. Have a cap positioned near the tube, and place the cap on the sample vessel as soon as it has cleared the top of the tube. The gas

needs to be sealed inside the sample vessel, but be careful not to overtighten the cap, as this can cause it to dimple.

- Inject 1 mL of each sample into one of the gas-filled sample vessels – use a separate disposable syringe for each sample.
- Enter the sample number and the lab ID number into the $CO_2$-$H_2O$ extraction book.
- Make sure to label your bottles! Use the printed numbers that can be found in the file drawers (in the third drawer down). These numbers correspond with the autosampler numbers for the heating block. The numbers increase left to right.
- Place sample vessels with gas and water into the autosampler block, replace cover, and allow equilibration to proceed for 24 hours prior to analysis.

**On the computer in the Isodat Acquisition program:**

- Make sure the configuration is on GB+Sampler
- Make sure measurement is set on $CO_2$
- Run several STD on/off measurements; select the NMT GBonlyzero sequence and highlight the NMT GBonly $CO_2$ in He lines from the sequence. Press start, and make sure to run only selection.
- Select the NMT CO2 sequence
- Write all the identifiers you want in the sequence chart
- Make sure the AS Sample number matches each slot you put the sample in
- Make sure in each sample you want to run that the AS Method is set to Internal no. 6 and the method is set to NMT GBonly $CO_2$ in He.met
- When everything else is ready, press START
- When the menu comes up, select to save under the file name: CO200-00-00 (date)
- If you want to measure a selection only, highlight the selection and select measure selection only
- Select whether you want one printout for the entire sequence, or one printout per sample
- When everything is ready, press OK.

## 10.2 Sulfides/Sulfates

### 10.2.1 Sulfides

- Powered sample material is weighed in a tin sample capsule. The weight of material should correspond to an equivalent amount of sulfur as approx. 0.7 mg of pyrite.
- The tin cup is folded to enclose the sample
- Sample cups are placed in the auto sampler of the Costech Elemental Analyzer.
- Two bypass samples should be loaded in front of the samples.

- Two to three different sulfur standards should be run to make a correction curve.
- After loading, vent auto sampler with He and check for leaks.
- Start Mass spec by running SO2 gas in Standard On/Off configuration several times.

#### 10.2.2 Sulfates

Sulfates are analyzed using the same procedure as Sulfides in 9.2.1.

### 10.3 Jarosite/Alunite

These are run for sulfur as per 9.2.2 Sulfates. Procedures for analyzing for δD and $\delta^{18}O$ will be developed during Spring 2004 and included as a revision to this SOP.

## 11.0 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES

### 11.1 δD on Water

Samples are analyzed on a Finnigan Delta Plus XL mass spectrometer in dual inlet mode in combination with a CombiPal autosampler and the H/Device Cr reduction furnace. For each batch of samples (Typically up to 50 total analyses), water satandards OH-1, OH-2, OH-3, OH-4, from the International Atomic Energy Agency, are analyzed. These standards are used to make a calibration curve to assure the accuracy of the analyses. An in-house reference water NM-2 is run once every ten samples to test for drift. One in every 10 samples is run in duplicate to check precision.

### 11.2 $\delta^{18}O$ on Water

Samples are analyzed on a Finnigan Delta Plus XL mass spectrometer in continuous flow mode in combination with the CombiPal autosampler and the Gasbench interface using the $CO_2$-$H_2O$ equilibration method. Standard waters (OH-1 thru -4) are used to define a calibration curve. Instrumental drift is checked by running an in-house reference water (NM-2) every 10 samples. Precision of actual samples is checked by running a duplicate every 10 samples.

### 11.3 $\delta^{34}S$ on Sulfides/Sulfates

Samples are analyzed on a Finnigan Delta Plus XL mass spectrometer using a Costech EA and autosampler. Three solid isotope standards are run each day to calibrate the results. Duplicates are run with each batch of 10 samples.

## 12.0 DOCUMENTATION

Documentation of observations and data acquired in the field and the laboratory will provide information on the activities concluded and also provide a permanent record of field and laboratory activities. The field observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages, and on field

sample forms. Laboratory documentation should occur in the Stable Isotope Laboratory Notebook.

## 12.1 Field Sample Form

A field sample form will be completed at each sample location. Items not applicable to the sampling will be labeled as not applicable (NA). The information on the data sheet includes the following:

- Sample Number
- Project
- Collector
- Field Date
- Location
- Quadrangle
- Scale
- Township, Range, and Section
- Latitude and Longitude
- Waste Rock Pile or Area
- Rock Unit
- Rock Age
- Field Occurrence
- Rock Description
- Rock Structures
- Measured Section
- Sample Type
- Alteration
- Mineralization
- Photo Number
- Remarks
- References

## 12.2 Field Notes

Field notes will also be kept during sampling activities. The following information will be recorded in the bound field logbook using waterproof ink:

- Names of personnel
- Weather conditions
- Date and time of sampling
- Location of sampling, including GPS coordinates
- Description of samples, and analyses to be performed
- Description of QA/QC samples

- Number of samples/sample vials and/or bags
- Sample handling and method of shipment

### 12.3 Laboratory Documents

Sample data will be kept in the Stable Isotopes Laboratory Notebook labeled MOLYCORP, INC. in permanent ink.

## 13.0 REFERENCES

Rye, R.O. and Alpers, C.N., 1997. The stable isotope geochemistry of jarosite. U.S. Geol. Surv. Open-file Rept. 97-88.

Stephen Nelson and David Dettman, 2001, Improving hydrogen isotope ratio measurements for on-line chromium reduction systems, Rapid Communications in Mass Spectrometry; 15: 2301-2306. We keep a copy in the lab.

Wasserman, M.D., Rye, R.O., Bethke, P.M., and Arribas, Jr., A., 1992, Methods for the separation and total stable isotope analysis of alunite: United States Geological Survey, Open-File Report 92-9.