Extraction of Beryllium from Quartz

The method that follows is used to separate Beryllium and Aluminum from pure quartz for AMS measurement.

Sample Weighing, Spiking & Blank Preparation

- Determine the amount of quartz and carrier needed for each sample.
- For a batch of 6-11 samples of similar size, prepare 1 process blank.
- Label Jars. Select a Savillex™ jar large enough so you only fill the beaker ¾ full. Estimate space for 5mls HF per gram of sample, the sample itself, and some water. Use the same size beaker for the blank as for the samples. Name the blank with the following format: Blank_1_2007Jul04. (Use 1, 2 etc. if you have more than one blank.)
- Weighing. The analytical balance can weigh up to 200 g. If the Teflon jar + samples wt. will be less than this, you can weigh your sample directly into it. Otherwise, you will have to get your weight by weighing what you remove from the sample jar.
- If you are measuring Al as well, you must record the weight of the beaker.
- To reduce static, wrap Al foil around the beakers.
- If you are not using the entire sample, make sure the sample is well mixed so that the split taken is representative of the entire sample.
- Immediately after weighing the quartz and before removing the Al-foil wrap, cover the sample with MQ-water. Gently squirt around the sides of the jar and add enough water to completely cover the sample.

Weighing Sample

METHOD 1: *Weighing directly into Teflon jar. (For a sample size < 25g and Teflon jar < 180ml.)*

- Place a clean labeled Teflon jar wrapped w/ Al foil on the analytical balance (the Al foil reduces static).
- Tare the balance.
- Add desired amount of sample to the jar. Record the weight to 4 decimal places.
- Remove the jar from the balance and cover with MQ-water.

METHOD 2: *Weight by difference.* (*If the Teflon beaker + Sample will be > 200g.)*

- Wrap the Teflon jar w/ Al foil.
- Weigh the entire sample in its storage container. Record this weight as "Sample + Tare wt.".
- Empty the entire contents of the container into the Teflon jar.
- Weigh the empty container. Record this weight as "Tare wt."
- You will calculate your sample weight – "Sample + Tare" – "Tare".
- Cover the sample w/ MQ-water.

Be very careful not to spill any sample in this transfer, since your sample weight is being determined by weighing the amount removed from the sample container.

Clean your work area between samples!

Adding Carrier (^{9}Be)

Since the natural concentration of ^{10}Be in rock is too low to be detected by AMS we add a known amount of ^{9}Be to each sample.

ALL INITIAL AND FINAL WEIGHTS MUST BE RECORDED IN THE CARRIER LOG BOOK!

Pipette ONLY from the "working" bottle, and never from the "stock" bottle. NEVER RISK CONTAMINATING THE CARRIER!

Refilling the working bottle - Record the initial weight of ***both*** *the working bottle and the stock bottle and confirm that they equal the final weight from the previous use. Remove all the Parafilm™ before weighing the bottles and invert the bottles a few times to homogenize the solution. Pour from the stock bottle into the working bottle and record these new weights. When you are finished spiking all your samples, record the final weight of the "working" bottle.*

- We calculate the amount of carrier added to a sample by weighing the working bottle before and after each addition to a sample rather than directly weighing the amount delivered to the sample.

- Tare the balance.

- Weigh the carrier bottle and confirm that it equals the final weight from the previous use. Record this weight in both the log and your notebook.

- Remove the cap and pipette ^{9}Be carrier into your sample. Use the 100 – 1000µL pipette and MAKE SURE THE PIPET IS SET AT THE CORRECT VOLUME!

- Immediately recap the carrier bottle and reweigh it. Work quickly, but carefully. Do not leave the carrier bottle open longer than necessary. We want to reduce evaporation as much as possible.

- Remove the pipette tip and rinse it out with some MQ-H_2O directly into the sample beaker. This is done to ensure that the entire amount removed from the bottle, which is what we are weighing, is delivered to your sample and no drops were left behind in the tip. Use this method unless you know you can consistently deliver 100% from a pipette. Discard the tip and use a new tip for each sample.

- Reweigh the carrier bottle and record the weight. Calculate the amount of carrier added to your sample as you go along to ensure you have added the amount of carrier you think you have added.

- When finished, check that the cap is screwed on firmly and seal with Parafilm.

- Record all final weights in the Log Book. And, in your notebook, record what carrier you used.

Blanks

The primary use of blank is to correct the sample ^{10}Be concentration for any ^{10}Be contamination occurring during the sample preparation.

As a general rule, prepare 1 blank per 8-10 samples if all samples are of similar size and are spiked with the same amount of carrier and you expect they will go through the exact same column chemistry. If sample weights should vary by a factor of 3, make up 2 blanks, one to represent small samples and one for large samples, or if you know or even just suspect some of your samples will require more column chemistry, prepare an extra blank.

The blanks are treated exactly like a sample. Use the same size Teflon jar as you used for your samples, rinse the sides down w/ MQ-H2O as you did for the quartz, and add the carrier in the exact same manner. Prepare blanks at the same time you weigh out and spike samples.

Sample Dissolution

SAFETY INFORMATION*: You will be using very large volumes of concentrated HF in this step. Follow all safety precautions discussed in the HF/HNO3 leaching step – i.e., closed shoes (no fabric shoes), long pants, lab coat, nitrile or neoprene gloves and eye protection. Do not work alone in the lab while pouring large volumes of HF.* ***The sample may react upon addition of concentrated HF****, so add the HF slowly and use extra caution with a large sample.* ***Leave the lids off completely, or cap loosely, and do not swirl your samples for a few hours****.*

- Add ~5 ml HF per gram of quartz. (Reagent A.C.S. grade is fine)

- The volume of HF added does not need to be exact. Use a comfortably large plastic beaker to measure the HF, making it as easy as possible to pour. (Never use glass w/ HF.)

- Screw the caps on the beakers, loosely at first, to allow for any release of gas if the samples are reactive. After a few hours, tighten the lids.

- If you have the time, just let the samples sit until they're dissolved, rather than putting them on a hotplate. It's the easiest and cleanest way to handle them. You eliminate having to deal with condensation on the lids, and the deposition of silica and fluoride salts on the lid. A 5 gm. sample will dissolve in about a day while a 50 gm. sample will need several days. Swirling them several times/day helps. Make sure the caps are on tightly!

You can speed the dissolution up with heat, but first allow the samples to sit overnight before placing them on the hotplate. You can heat them initially with the lids off and at a very low temperature (~ 50 ºC.) for a few hours so you are sure they won't react violently. Then, put the lids on tightly and turn the heat up to ~150 ºC. It is the combination of heat and pressure that really speeds things up. It is important that you used a large enough jar so there is enough headspace to accommodate the buildup of pressure.

Note: Savillex™ Teflon melts at 270ºC. Keep the temperature below 220ºC.

If you are measuring Al, this is where you would take a split for stable Al measurement. Otherwise, continue with the dry down.

Evaporation & Dry Down w/ Perchloric Acid & HCl

Once the samples have dissolved, or are nearly dissolved, you will evaporate off all the HF. Successive Perchloric Acid ($HClO_4$) evaporations remove fluorides from the sample.

CAUTION: Perchloric acid is a very strong mineral acid and when heated above 150 ºC. becomes a strong oxidizer and can react violently with many oxidizable substances, such as paper and organic solvents, causing fire or explosion. **Do not have any paper or organic solvents in the hood!**

If you dissolved them on the heat, first turn off the hot plate and allow the solutions to cool. There will be drops on the underside of the lid, so be careful not to lose these, especially if the sample hasn't completely dissolved yet. With the lid screwed on tightly you can slightly invert and swirl the jar to remove the drops from the lid. Then, remove the lids and gently rinse them into the jar with MQ-H_2O. Place the lids face down on a clean surface in the hood for storage.

VERY IMPORTANT! Until the sample is completely dissolved, do not spill a drop! If you lose any solution at this point you are preferentially losing ^{9}Be (the carrier). Once the sample has completely dissolved, ^{9}Be and ^{10}Be are in equilibrium, and a spill will not affect the 10/9 ratios.

NOTE: If after the first dry down the sample is not completely dissolved, add more HF (about 10 ml), put the lid on tightly and put it back on the hotplate. If it is really quartz remaining, the combination of a little $HClO_4$, HF, heat and pressure should dissolve it. Sometimes there are minerals that won't dissolve which you'll centrifuge out later.

- Add ~ 0.5 – 1.0 ml conc. $HClO_4$ to each jar, using a disposable pipette. Place on a hotplate at 150 – 200 ºC to evaporate. You can set them on the pancake griddle hotplates overnight at the lower temp. It can take more than 24 hours for a large sample to completely evaporate.

  (Perchloric acid is thick and drippy. Use a disposable pipette and keep a plastic beaker close by to put the pipette in.)

- Dry them down completely, and then add another 0.5 ml conc. $HClO_4$ to each jar. Swirl them to get everything into solution and evaporate to dryness. If needed, use a little MQ-H_2O to rinse the sides down.

- Repeat once more. You will have done a total of 3 additions of $HClO_4$.

Perchloric acid will fume characteristically dense white fumes when the temperature is hot enough.

Chloride Conversion

Successive hydrochloric acid evaporations rids the sample of remaining fluorides and the Perchloric acid almost entirely. Fe, Ti, Al, Be, and other ions are left as chloride salts ready for anion exchange clean up.

- Remove the Teflon jars from the hotplate and cool slightly before adding HCl. HCl tends to splatter when added to a very hot beaker.

- Add ~0.5 ml - 1 ml of conc. HCl

  Use the larger amount for samples with a very large residue. Most samples are fine with 0.5 ml. Rinse down the sides of the beaker with the HCl addition and/or a little MQ-H_2O. The residue should re-dissolve almost instantaneously.

- Evaporate to dryness at ~ 125 - 150 ºC.

- Repeat the HCl addition (using ~ 0.5ml) and evaporation step 2 more times.

- Cool the samples completely. Then add 2 ml concentrated TM grade HCl to each sample. Close the lid, and allow them to dissolve.

  The final solution may be a deep yellow-green color due to $FeCl_3$. Some samples may also have thrown a fine, powdery white precipitate that will not re-dissolve. This is probably TiO_2. No Al or Be is co-precipitated with the Ti and it can be removed by centrifuging before the anion exchange.

Transfer to centrifuge tubes

- Rinse 15-ml centrifuge tubes w/ MQ-H_2O and label them w/ the batch #, sample id, and “anion col.”.

- Transfer the samples to the labeled centrifuge tubes. You can pour it in, or transfer with a transfer pipette. If the sample is thick, sticky and full of residue it is easier with a pipette.

- Add another 2 ml of concentrated TM HCl to the jar as a rinse and transfer to the centrifuge tube. There should be 4 ml in the tube.

Anion Exchange Columns

The anion exchange columns remove Fe^{III} (and some Ti) in the sample.

Equipment and Materials

- 30 ml BioRad Columns packed w/ 7ml AG 1-X8 Resin, 100-200 mesh, chloride form.
- 1.2 N HCl (Trace Metal grade)
- Conc. HCl (Trace Metal grade)
- Teflon beakers for collecting Be fraction **
- 60 ml plastic bottles – rinse with MQ water, label w/ sample id and # and "Anion Eluate".

The anion columns can be reused many times. Inspect the columns before use. When the anion resin gets too old it will take on a darker color and/or contains bubbles in it. If you need to repack columns, follow the procedure for column packing.

** You can reuse the Teflon jars you dissolved your sample in if they are 90 ml or smaller, and if they are clean. Sometimes the digestion leaves black residue behind. This can be wiped out w/ a Kimwipe, but should then be followed by a quick leach w/ some dilute HCl or HNO3 (~5% is fine) on the hotplate. Use a new clean Kimwipe for each jar.

PREPARE SAMPLES:

- Centrifuge the samples for 10-15 minutes at ~ 3500 RPM to remove solids.

STEP 1: COLUMN PRECONDITIONING:

- **Set up the anion columns** in the hood. Place waste collection trays under them, remove the covers, open the stopcocks and allow them to drain. (They are in H_20.) Rinse of the stopcocks before beginning and check that they are clean.

- **Add ~ 30 ml 1.2N HCl to the column**. The volume does not need to be exact. Filling the column to the top equals 25 ml, so fill once plus a little more using the squirt bottle and being careful not to disturb the resin surface.

- **Add ~30 ml concentrated HCl**.

In the meanwhile…

- **Clean disposable pipettes** you will need for transferring your samples from the C-tubes to the columns. Simply rinsing them by sucking up MQ-H_2O a few times should be sufficient, but you can also first suck up a little dilute HCl, and then rinse w/ MQ-H_2O.

STEP 2: LOAD SAMPLES & COLLECT BERYLLIUM FRACTION

- Place the labeled **Teflon beakers** under the columns.

- **Transfer the sample solutions** from the C-tubes to the columns with the disposable pipettes. Drip the solution onto the resin bed being careful not to disrupt the resin surface.

- **Add 20 ml of conc. HCl to each column**, in 2 batches of 10 ml.
  (Use a 10mL-graduated cylinder and fill it from the squirt bottle containing conc. HCl)

STEP 3: COLLECT ANION ELUATE

- Place a labeled **60-ml bottle under each column**.

- **Add 60 ml 1.2N HCl** to each column.

It is not necessary to first measure the 1.2N. You can just use the squirt bottle and fill the headspace above the resin ~2 ½ times (headspace = 25 ml), filling the eluate bottle to the top (which you know is 60 ml.). The resin should return to its original color and the eluate will become clear. Save the eluate just incase!

STEP 4: CLEAN THE COLUMNS

- Place a waste tray under each column and fill the columns with at least 50 ml MQ- H_2O. (Fill to the top 2x)

- Close the stopcocks and fill the columns to the top with MQ-H_2O and put the covers on to prevent the resin from drying out. Rinse the stopcocks with MQ- H_2O.

CATION COLUMNS
Sample Preparation - SULFATE CONVERSION

The cation column separated Al, Be and Ti. The column procedure using 2 ml of resin can handle 3-5 mg of Ti, if the total amount of Al and other metals is less than 3-5 mg. The method easily scales up and the volume of resin can be doubled or tripled.

- Add 1 ml of **0.5M H_2SO_4** to each Be/Al fraction and dry-down. This will take ~ 4-6 hours. NEVER EXCEED THIS VOLUME OF H_2SO_4! The dried residue from this step may turn an alarming dark-brown to black color due to organics which bled from the anion resin. Don't worry - it will disappear over the next couple of steps.

- Add 2 ml **MQ-H_2O containing a trace of 0.5M H_2SO_4. Add a few drops of 2% H_2O_2** from the squirt bottle. The cakes will dissolve and turn an amber/gold color ($TiO[H_2O_2])^{2+}$) if Ti is present. Dry the samples down again. The black charry material will disappear after a while.

- Cool, repeat the **MQ-H_2O w/ trace 0.5M H_2SO_4 and H_2O_2** step , and dry the samples a second time. At the end of this procedure, the samples should end up either as compact white cakes or small, syrupy droplets of involatile H_2SO_4. If they are still discolored, repeat the peroxide/water addition and dry them down a third time.

- Take the samples up in **3-ml of MQ-water, containing a trace of ~2% H_2O_2 and 0.5 M H_2SO_4**.

Note: The samples may not go into solution immediately, but they should within a few hours (unless they are loaded w/ Ti and/or Al, in which case you have a problem!). If necessary you can warm them gently, but don't risk evaporating and concentrating them. Warm them with the covers on. Keeping the acid strength low for column loading gives a sharper elution and cleaner Ti-Be cut. The samples are now in ~0.2M H_2SO_4, ready for loading on the cation exchange columns. They can be stored indefinitely in this form.

Cation Exchange Columns – Separation of Be from Al and Ti

Equipment and Materials

- 11 ml BioRad Columns
- DOWEX-50 X8 200-400# cation exchange resin, presoaked in HCl
- 1.2 N HCl (TM grade)
- 0.2 M H_2SO_4 containing a trace of 2% H_2O_2 (trace ~ 0.015%)
- 0.5 M H_2SO_4 (containing a trace of 2% H_2O_2)
- 4M HCl (TM grade)
- 30 ml HDPE bottle for the Ti fraction.
- 22 ml teflon vials for the Be fraction.
- 8 or 15ml HDPE bottles for the Al fraction.
- Clean disposable pipettes

CALCULATE THE AMOUNT OF EACH ACID YOU WILL NEED BEFORE BEGINNING.

COLUMN SETUP

- Load a column rack with the small (~11 ml volume) Bio-Rad columns with stopcocks. Place waste collection trays under them. Add some water and make sure they are dripping before you add resin. Adding a few drops of methanol first to wet the frit helps, or just tapping the column can help to get the dripping started.

- Using a disposable pipette, add 2 ml of DOWEX-50 X8 200-400# cation exchange resin to each column. (Use presoaked/stripped resin) Fill the column with a little MQ-water and before it drains, slurry in a thin suspension of resin. This will immediately slow the dripping, and you can keep the column full with water while you slowly add more resin to the 2 ml mark. Be very careful not to trap air bubbles.

STRIPPING & CONDITIONING RESIN:

- Strip the resin by filling each column headspace with 4 M HCl (This is ~9 ml, equal to 4-5 resin-bed volumes.) Allow it to drain completely.

- Condition first with ~ 9 ml 1.2 M HCl. Drain completely.

- Next, add ~ 9 ml 0.2 M H_2SO_4 containing a trace of 2% H_2O_2. Drain.

- Remove waste trays and discard acid into waste containers.

ELUTE Ti:

- Place 30 ml rinsed and labeled ("Ti Fraction") bottles under columns.

- Load each sample onto its column using a clean disposable pipette. Ti will form a narrow brown band at the top of each resin bed, and then begin to move down the columns. Allow the sample to run into the resin completely.

- Add 1 ml of 0.5 M H_2SO_4 w/ trace 2% H_2O_2 to each beaker as a rinse. Swirl the beakers to pick up any droplets of sthe original solution left over from the first load. Add the rinse solutions to the columns after they have drained. Allow this to run in completely.

- Add 10 ml (5 bed volumes) of 0.5 M H_2SO_4 w/ trace 2% H_2O_2 to each column. If Ti is present, you can see the Ti band move down the resin and elute from the columns. For samples containing Ti but very little Al, the Ti will elute slower and it may be necessary to add another 1-4ml of 0.5 M H_2SO_4 to completely remove Ti. You can safely elute until the eluate is clear.

NOTE: The drips coming off the column should initially be clear, and then slightly to very yellowing depending on the amount of Ti in the sample. If the drips are immediately yellow, the column is probably overloaded with Al. Take note of this, but continue on. You will probably have to do a second column to clean up the sample.

Make a note in your notebook how many mls is took to elute the Ti, how dark or light, narrow or broad the Ti band is, and when it started dripping yellow.

ELUTE Be:

- Place 22 ml labeled Teflon vials under each column.

- Add 10 ml (5 bed volumes) of 1.2 M HCl ("10%" HCl). This will have to be added in 2 lots. There is no need to allow the first to drain completely before adding the second. Allow it to drain through completely.

  Elute Blanks with 12 ml 1.2 M HCl. With no other ions "pushing" the Be through the column, it takes a little more to get the Be out.

ELUTE Al:

- Place 8 or 15-ml labeled bottles under each column to collect the Al fraction. Elute Al from the columns with 6 ml (~3 bed volumes) of 4M HCl.

Be Recovery

Dry down

- Add ~5 drops of conc. HNO_3 to each Be sample. (You can dry them on the hotplate overnight at < 100 ºC, or in approx. 4 hrs @ 150 ºC.)

- Label and rinse 3x w/ MQ-H_2O 15-ml screw cap centrifuge tubes for each sample.

- Once the Be fractions have dried, cool and remove them from the hotplate. The Be fraction should have contracted to a tiny, clear droplet of concentrated H_2SO_4. Occasionally they will form a small white cake. This usually indicates the presence of either Ti or Al.

Transfer to Centrifuge Tubes

- Pipette 2 ml of 1% HNO_3 into each vial. If pure, the Be fractions will dissolve freely. If they don't, you can warm the vials for a few minutes on the edge of the hotplate with the lid on, or just wait a few hours.

- Carefully pour the solution into a labeled centrifuge tube. Don't worry if a last drop clings to the floor of the Be beaker, but if its large, you can pick it up w/ a pipette.

- Immediately add another 2 ml of 1% HNO_3 into the vial as a rinse, and transfer to the c-tube.

Beryllium Hydroxide $Be(OH)_2$ Precipitations & Washes

You will precipitate the samples two times, and do 4 washes with a pH adjusted water. This step cleans up your sample and gets rid of Boron contamination. You will see your samples get more clear and translucent with each step.

- Add ~ 220 µl NH_4OH to the centrifuge tube, cap it, and mix well on the vortex mixer. You should see the white $Be(OH)_2$ precipitate swirling around. Using a clean pipette tip for each sample, remove ~ 5 µl to check the pH. It should be close to 9. If the pH is below 8.5 add more NH_4OH (add ~30 µl at a time until you reach the correct pH). If you overshoot and the pH is 10, leave it. It's better a little high than low.

- Centrifuge for 15 minutes at 3500 RPM.

- LOOK AT YOUR SAMPLES CAREFULLY AT THIS POINT and compare the sample Be to the blank Be. They should all be the same size. If the samples are larger than the blank, it

indicates Al and you probably need to do a second cation column. If the sample is smaller than the blank, you may have lost Be. But, before you make this assumption, check the pH of the supernatant. If it is just pH 8, try adding more NH_4OH and bring the pH to 9. Centrifuge again. If it is still small, just proceed to the 2nd precipitation. This always improves the clarity and often the size of the precipitate. If the precipitate is too big (indicating that there is probably Al in the Be fraction) go to the section on preparing a sample for a second column.

- Pour supernatants into a rinsed and labeled 30-ml bottle (label w/ sample name and either "Rinse", "wash" or "supernatant". Be careful not to pour out any precipitate.

- Do a 2nd precipitation: Add 100µl of 8N HNO_3 to all your samples. Swirl on the vortex mixer until precipitate has dissolved completely. Bring the volume up to 5-ml with MQ-H_2O. Swirl again. Re-precipitate $Be(OH)_2$ by adding ~100µl TM NH_4OH. Mix well on the vortex mixer.

- Centrifuge for 15 minutes again. Decant supernatant into rinse bottle.

- **IMPORTANT** - After precipitating the $Be(OH)_2$, do not let the samples sit around. Always centrifuge and pour off the supernatant immediately. Impurities in the supernatant may precipitate out of the solution over time defeating the purpose of these precipitation and wash steps.

pH 8 RINSES:

- Bring solution volume up to 5 ml with the pH 8 adjusted H_2O. Swirl on the vortex mixer, centrifuge and decant the supernatant into the rinse bottles. Do 4 pH 8 rinses in total. This step presumably gets rid of any Boron-10 contamination, an isobar of ^{10}Be.

$Be(OH)_2$ Combustion

Transfer Samples to Quartz crucibles

- Set up clean crucibles in the Quartz sleds, and on the sled, write the sample id next to the crucible. If possible, do not fill all 10 positions on the sled, and make a diagram in your notebook with the position of each sample in the sled. The marker gets combusted and disappears, thus leaving one position open in the sled will prevent you from mixing up the samples. There is no identifiable right and left on the sleds.

- Dissolve the $Be(OH)_2$ in the C-tubes with 50 -100 µl8N HNO_3.

- Swirl on the vortex mixer.

- Transfer to the crucible using the 200µl pipette. With another pipette, add another 50 – 100µl 8N HNO_3 as a rinse. Pick this up w/ the same pipette you used for the sample transfer, and add this to the crucible. Use a new pipette tip for each sample.

- After all samples have been transferred to the crucibles, place the sled on ceramic hotplate. Begin with a low temperature, of ~ 100 °C, and over the course of several hours increase the heat to ~ 200-250 °C. You want the sample to dry on the bottom of the crucible and heating it too fast can result in it drying around the sides making it more difficult to get the sample out of the crucible after its been combusted. Once the samples are dry, crank the heat to the highest setting for a few minutes, then turn it off and allow them to cool.

- Put the sled covers on, and transfer the sleds to the furnace. Combust for 60 – 90 min. at 930 °C.

- Once cool enough to handle, remove from the furnace, cover with labeled crucible covers, and store in the flow bench for loading. If you are not going to load them immediately, wrap each crucible in Al foil, label it, and place in a storage box.

LOADING CATHODES for LLNL

Equipment List

- Cathodes
- Drill Rods, #55
- Stainless Spatulas (scrapers)
- Quartz rods
- Niobium powder
- Scooper
- Cathode holder/stand
- Hammer
- Dust Mask

Be extremely careful when working with beryllium metal (oxide form). Beryllium is a known health risk and all precautions must be followed when working with it. Always work in the fume hook, and wear a dust mask.

- **Label Cathodes**
    - Make sure you are using cathodes that have been cleaned, and check each cathode to make sure the hole is centered, and is the correct size. We occasionally get cathodes with holes that are too small. Check this with the drill rod.

    - Label the cathode with the sample number, and full sample name.

- **Clean Drill Rods**
    - If you are starting with new drill rods, you only need to wipe them down with methanol. If you are reusing your drill rods, first wipe them off with methanol. You can rinse them with some water, but dry them off immediately, because they rust easily. Then, clean the ends off with some fine sandpaper (400 or 600 grit). Finally, rinse them off again and wipe them down with methanol.

- **Stainless Spatula's (Scrapers)**
    - These should be cleaned in a 10% nitric solution overnight. They should also get some time in the ultrasonic bath. You can just stick them in the top of the big tank for 30 minutes or so. Pour off the cleaning acid into the 2nd spatula cleaning acid bottle, and then rinse the spatulas thoroughly. Wrap them well with KimWipes and dry them in the oven.

- **Quartz Rods**
    - These are also cleaned in a 10% nitric solution. Follow the spatula instructions. They have their own bottle.

SET UP IN THE HOOD

- Wipe down the hood you will work in thoroughly.

- Set up your tools in the flow bench.

- Set up the cathode holder on a KimWipe.

- Set up the following waste containers.
    - A dirty cup with water for the used crucibles.
    - A clean plastic cup with MQ water for the used spatulas and quartz rods
    - A clean plastic cup (with NO water) for the used drill rods.

- You will need the tiny scoop and niobium powder and a hammer. Wipe off the hammer with some methanol first. Keep a bottle of methanol in the hood for wiping down your surfaces between samples.

LOADING THE CATHODE

- Place the cathode on the holder
- Get the sample from the flow bench.
- Place it on the holder and take the lid off.
- Add 3 level scoops niobium. You can adjust this up or down for larger and smaller samples.
- Using the quartz rod first, gently mix the niobium into the sample. Once the Niobium is mixed in, static is usually not a problem, but before that, the Beryllium can be rather flaky.
- Grind the Niobium and Beryllium together, as you would grind something up with a mortar and pestle.
- Using the stainless steel scraper, scrape together the mixture into the bottom.
- Repeat the quartz rod grinding and scraping a few times.
- When the sample is fully homogenized, use the scraper to collect it into the bottom. You can also tap the crucible on your work surface to get it to collect.
- Carefully tilt the crucible on the edge of the cathode, at a 45 deg. angle or so, and gently tap the crucible, and the cathode with the scraper. This will cause it to pour down onto the cathode. If it doesn't slide right into the hole, simply tap the sides of the cathode. It will.
- Using the drill rod, hammer the sample into the hole. I hammer hard for about 20 taps, them remove the drill rod, take the sample off the holder and tap it a few times on the holder then hammer another 20 pretty hard taps. I repeat the hammer hard followed by tapping a total 3 times, and finish off with about 10 more gentle taps.
- You can gently turn the cathode upside down on the clean KimWipe to check that it isn't going to spill out.

- Store the cathode in a labeled storage vial, and double check that all your labels are correct.
- Wipe down your work area before loading the next cathode.

Preparing Resin and Packing Columns

Resin Preparation

- Soak the resin in concentrated HCl in the designated bottle. After a few hours, decant the HCl into a waste container. Fill the bottle w/ MQ-H2O, shake and decant after the resin has settled. This will take a couple of hours. Do this 2-3 more times several times so that it is no longer strong acid.

Packing columns with resin

- Before filling the columns with resin, fill the column with water and make sure it drips. Usually tapping the column up and down a few times breaks the surface tension and it'll begin to drip. Or, squirt in a few drops of methanol before adding water.

- Then, fill the column with MQ water and using a disposable pipette immediately add some resin from the batch soaking in dilute HCl. The initial resin will settle onto the frit and immediately slow the water dripping through. Keep the water volume full while you add the resin. The resin should settle out gradually and evenly as it is added thus avoiding air bubbles getting trapped in the resin bed. Continue to add resin to the column until the proper volume is reached. If you do get air bubbles, fill the column with some water and suck up the resin with the pipette to re-suspend it and usually it will resettle without bubbles.

Anion columns

- Resin: AG 1-X8 Resin, 100-200 mesh, chloride form

- Fill the anion columns with 7 ml prepared resin. (Note: Resin volume will shrink and swell as different buffer strengths are added.)

- New resin must be thoroughly rinsed with concentrated TM HCl in order to flush out organic material that bleeds from the resin in strong acid. It is visible as orange-brown residue in the eluate. The pre-soak should get rid of this, but still check the eluate when setting up a new column and flush conc. HCl through as needed.

You've overloaded a column! Preparing samples for a 2nd Cation Column

- You still have to precipitate the sample. This is the only way to remove the sulfuric acid. So, do that, and note the volume of ppts.
- Dissolve the BeOH precipitate in a little bit of dilute HCl and transfer it back to a Teflon beaker.
- Then follow the normal procedure for Sulfate Conversion.

DOUBLE CATION COLUMN – (4ml resin)

- Use the larger columns.
- Fill w/ 4 ml resin. Note, that these columns are marked w/ height in cm., and not volume. 4 ml is just under the 6 cm. mark.

STRIPPING & CONDITIONING RESIN:

- ~ 18 ml 4 M HCl (Fill to the top twice.) Allow it to drain completely.
- ~ 18 ml 1.2 M HCl. Drain completely.
- ~ 18 ml 0.2 M H_2SO_4 containing a trace of 2% H_2O_2 . Drain.

Remove waste trays and discard acid into waste containers.

ELUTE Ti:

- Place 30 ml rinsed and labeled ("Ti Fraction") bottles under columns.
- Load each sample onto its column using a clean disposable pipette. YOU STILL LOAD in 3ml. Allow to soak into the resin completely.
- Add 1 ml 0.5 M H_2SO_4 containing a trace of 2% H_2O_2 to each beaker as a rinse and add to the column.
- Add 18 ml of 0.5 M H_2SO_4 w/ trace 2% H_2O_2 to each column. It may be necessary to add a further 4-5 ml of 0.5 M H_2SO_4 to completely remove Ti. Note how much you use.

ELUTE Be:

- Place 22 ml labeled Teflon vials under each column.
- Add exactly 20 ml of 1.2 M HCl ("10%" HCl).

ELUTE Al:

- Place 15-ml bottles under each column to collect the Al fraction. Add 12 ml of 4M HCl.