UVM Cosmogenic Laboratory: Be/Al Extraction

Purpose: This method details how we extract Be and Al from purified quartz.

Hazards: The primary hazard associated with this method is contact with or inhalation exposure to concentrated acids (HF, $HCLO_4$, HCl, HNO_3, H_2SO_4) and beryllium.

Personal Protective Gear: Goggles, thin (nitrile) gloves, rubber lab shoes, and lab coat at all times. Add rubber lab smock and face shield when handling HF and $HCLO_4$.

Why Blue and Yellow: We work with samples containing a wide range of different ^{10}Be concentrations. To reduce the chance of contaminating low-level samples with material from high-level samples, we maintain two different sets of lab equipment. Low-level samples are processed on yellow lab equipment. High-level samples are processed on blue lab equipment. It is imperative that these two sets of equipment be kept apart and not traded between the processing stations. All labware is clearly color-coded, either on the item itself or on the storage box. Only take labware from the processing stream in which you are working.

TEN COMMANDMENTS OF THE UVM COSMOGENIC LABORATORY

1.) **Safety is always top priority**
Approach all of your lab work with this in mind! Understand the safety features of the lab and know how to use them, and know what to do in an emergency. Make sure you are totally comfortable with a procedure before you attempt it. Never perform high-hazard parts of the method when you are alone or tired.

2.) **Protect yourself**
Always wear appropriate protection when working in the lab. For high-hazard acids (HF and Perchloric) this means wearing a smock, two pairs of gloves, goggles, and a face shield. Never touch anything in the hood without gloves, and always wear goggles when you are anywhere near active lab work, even if you are just watching. Never have bare skin showing between your smock and gloves when handling acids. Use gauntlets.

3.) **Handle acid with care**
Always use and store acid in an appropriate ventilated space, never on the countertops. Prepare for spills by using a spill tray and having wipes nearby. Exercise extreme caution when measuring and mixing acids; add acid to water, and only mix solutions that are described in the lab manual. Wash everything well before taking it out of the hood.

4.) **Practice appropriate hood etiquette**
Hoods should be used for working, not storage. Never place anything on top of the perforated ventilation grates since it will compromise the hood's ability to protect you from acid fumes. Leave the hoods clean and empty when you finish working; pull down the sash fully and turn out the lights. Wash down hood ducts using red handle for thirty seconds after every batch of samples and wash down the interior of the hood using the DI spray gun after each batch of samples.

5.) **Label, label, label**
This is to protect yourself and others! Make sure that all containers of liquid are clearly labeled. Always use the white boards, hood labels, and unattended operation door signs to alert others about what procedures are taking place.

6.) **Be clean**
Both the meteoric and in situ labs are isotopic clean rooms. Be paranoid! Nothing (except yourself) should be in the lab except what is already there. Do not go in the clean lab if you are dirty from field, construction, or grinding activities. Any surface that is in contact with a sample should be touched with acid-washed surfaces or clean gloves only (i.e. no fingers). Clean all labware and hoods regularly and thoroughly.

7.) **Keep green, blue, and yellow separate**
Green items are for the meteoric lab, blue items are for the high-level side of the in situ lab, and yellow items are for the low-level side of the in situ lab. This is to minimize cross-

contamination. No exceptions! Keep these items stored separately and wash them in their own wash bottles. Wash new tubes only in dedicated wash bottles.

8.) **Plan ahead**
Before you begin a procedure, think about what materials you will need and how long it will take. Do you have all the necessary equipment, disposables, and reagents? Are they out and easily accessible? Do you have enough time to complete the procedure without rushing? Do you know what you need to do? If you notice that the lab is running low on any consumable supplies, make sure to notify Paul before we run out.

9.) **Slow Down**
You should conduct yourself in a careful, deliberate fashion the entire time you are in the lab. Do not rush or take short cuts, since it will compromise your safety, the safety of others, and the integrity of your samples. Take the time to double-check yourself on every step.

10.) **Leave spaces neater than you found them**
This is a shared lab facility. Clean up any spills immediately, put everything back in its place, and wash used labware in a timely fashion. Try to leave all spaces, including the write-up room, how you would like to find them left for you.

SAVING THE EXPENSIVE AND FRAGILE REPIPETTORS

The Eppendorf repipettors are wonderful, time-saving instruments but they are also rather fragile and costly.

If exposed to acid fumes, they will quickly (in a matter of weeks) become inoperable.

These are Eppendorf's recommendations on how to keep these pipettes operating smoothly.

1. Never leave the pipette in the hood with acid present or during acid fuming. Remove the pipette and hang on the outside of the hood. We have installed pipette holders on the outside of the hoods for storage.

2. Always store the pipette vertically; never lie the pipette on its side. Vertical storage keeps any acid on the pipette from seeping into the inner workings.

3. Always store the pipette in open air in the lab so that any acid on the pipette can dissipate.

4. Be careful when inserting tips to press the buttons on the lower sides of the pipette so the tip can slide in with no rotation.

Weighing in Quartz

(Usually done on Tuesday of the week prior to extraction)

Personal Protective Equipment: Thin gloves while setting up. Gloves, goggles, and lab coat while handling Be/Al carrier during the weigh-in process.

For this procedure you will need: *(shown from left to right)*

- Weigh-in cart
- Anti-static device, powered on
- Digestion rack with 12 bottles (labeled in blue or yellow tape with sample name, position letter, and batch number; see description below)
- Ten purity-tested quartz samples in centrifuge tubes
- Four-place balance
- Cylindrical metal anti-static bottle holder to sit on balance
- Milli-Q squirt bottle
- 1 mL variable pipette with a new tip
- Al carrier
- UVM-A reference material solution
- "Xstream" pipette with new 10 mL tip
- Be carrier
- Bottle for Be carrier waste
- Computer with new batch sheet and data recording buddy *(not show)*

1. This is a two-person job. Find a buddy.
2. Use the blank rotation schedule pasted on the inner cover of the batch book to determine the position of your blank(s) and UVM-A reference material, then populate the other positions with your samples. Update the blank rotation schedule by adding your batch number on the appropriate line. If possible, order your samples in such a way that they make the most sense – alphabetical or numerical order will make them easier to keep track of overall.
3. For the blank, use the name "BLK"; if there are two blanks (for low-ratio samples), use BLKX for the second.
4. For the reference material, use the name UVM-A.
5. On each quartz tube, place a small piece of lab tape on the lid with the batch number written on it (BXXX). The tape should be yellow or blue as appropriate.
6. Turn on the balance and the anti-static device.
7. Use the check-weights to test balance and record the masses on the clipboard.
8. Open up the batch template on the lab computer in the corner. Add the batch number for your new batch and save the batch sheet in the folder "in situ batches" using the main lab computer. Enter your samples on the batch sheet and update the date, batch number and carrier fields. Make sure all the information is there, specifically carrier name.
9. In the "min test ICP data" page of the batch sheet Excel workbook, enter all cation concentration data from the quartz test results on the sample tube. Considering these impurities will prevent overloading of the cation and anion resins.
10. Take out bottle A. To remove static from the bottle, wave it through the anti static U-shaped upright for five seconds. Make sure the device is on; the green light should be glowing.
11. **TARE the balance**.
12. Then, mass the empty bottle with lid.
13. Once the balance has stabilized, read the mass to the person sitting at the computer. The person at the computer should enter it and read it back. **Save the sheet after every data entry.**
14. Take the lid off the bottle and set it aside.
15. **TARE the balance** with the empty bottle on it.
16. Wave the quartz tube through the anti static U- shaped upright for five seconds.
17. Pour the quartz into the digestion bottle adding no more than 45 grams (large bottle, low-level samples) or 23 grams (small bottle, high-level samples). If you add too much quartz it will may not digest fully. Record the mass on the batch sheet. **Keep the total cation load < 10,000 μg by adjusting the quartz mass. Using more sample can overload the columns and result in loss of Be and/or poor Be purity.**
18. Calculate the amount of Al carrier needed to get to optimal Al total (PRIME needs 1500 μg; LLNL needs 2500 μg, SUERC can go to 3000 μg but less carrier means higher ratios). The batch sheet does this automatically by multiplying the quartz mass by the Al content of the sample and subtracting that from the desired total (which you can specify in the batch sheet header). Add Al carrier as needed using the adjustable 1 mL pipettor. Record the mass on the batch sheet electronically.

19. **TARE the balance.** Add 250 μg of Be (as carrier) using the x-stream, taking into account the carrier concentration. Record the mass on the batch sheet electronically.
20. When you finish adding the quartz and carriers and have recorded all data, moisten the quartz using the Milli-Q squirt bottle. Add enough water to saturate the quartz, but not so much that there is standing water in the bottom of the bottle. Then replace the lid.
21. Place each bottle as it is filled with sample and carrier into the digestion rack.
22. Repeat for all samples.
23. When it is time to load the blank, add no quartz. Add the full amount of desired Al using the Al carrier and the 1 mL variable pipette, and the standard amount of Be using the Xstream. Record the masses on the batch sheet electronically.
24. When it is time to load the UVM-A reference material, switch to a new pipette tip on the 1 mL variable pipette. Add no quartz; instead, add 1 mL of UVM-A to the bottle and record it in the sample mass field. The UVM-A reference material is pre-spiked with Be and Al, so do not add Be or Al carrier; just put zeroes in the carrier mass fields. When you finish with the UVM-A, discard the pipette tip and put a new one on to do the Al carrier for the remaining samples.
25. Save and print the batch sheet and attach it to a clipboard. Place the appropriate color tape (blue or yellow) across the top and write your name and the batch number on the tape.
26. Clean up by vacuuming the balance chamber to remove all quartz, putting away carrier, disposing of the Be tip as Be waste and the Al tip in trash.

Adding Acid to Quartz

(Usually done on Tuesday of the week prior to extraction, following weigh-in)

Personal Protective Equipment: Double-gloves (thin and thick), rubber smock, goggles, and face shield.

For this procedure you will need: *(shown from left to right)*

- Rack of 12 sample bottles on the small hot plate
- Spill tray
- Clean acid beaker
- Large bottle of HF

1. Take bottle A, place it in the spill tray, and uncap it. Store the cap working side up in the spill tray.
2. Add HF to the sample by decanting from the large bottle to the graduated teflon beaker. WORK IN A SPILL TRAY. Use about 5 times the volume of HF to the mass of sample (for a 20 g sample, add 100 mL of HF; for a 40 g sample, add 200 mL of HF). This should fill the bottle up to its shoulder; do not overfill or it will be very difficult to deal with later.
3. Cap the sample and place it back on the digestion block. DO NOT SQUEEZE THE BOTTLE OR RISK MAKING AN HF SQUIRT GUN.
4. Repeat for all samples from B-L.
5. Let the samples sit in acid for at least an hour with no heat. This allows very fine quartz to dissolve. When samples are digesting, DO NOTHING ELSE IN THE HOOD and make sure the SASH IS ALL THE WAY DOWN.
6. Over the course of several days (see description on next page), the goal is to ramp the samples up to an overnight digestion temperature of 135 °C (or 140 °C for the larger bottles on the low-level side), hot enough to speed digestion but cool enough to prevent boiling.

Quartz Digestion

(Usually done on Tuesday through Friday of the week prior to extraction)

Personal Protective Equipment: Goggles when observing sample activity.

1. Record all temperature increases throughout the digestion on the whiteboard over the hood.
2. Once all acid has been added and you have waited for one hour with the samples cold, bring the hotplate to 65 °C.
3. After an hour or several hours, if there is no reaction, raise the temperature by 10 °C. Wait another several hours, and if there is no reaction, raise the temperature again, at most 10 °C per hour. If at any point, the samples are boiling (more than slowly rising bubbles), turn down the heat and wait. For the first day, take the samples no higher than 90 °C; then let them sit overnight.
4. IMPORTANT: The reaction that dissolves quartz is EXOTHERMIC and can run away causing boiling and venting of hot gas from the bottle top. Violent reaction will occur if you are impatient and ramp too quickly. The most sensitive range is 80 to 115 °C, but the behavior is highly variable. If the samples boil, turn down the hotplate and stay away from the hood.
5. On the second day, continue ramping up the samples. Once samples are above 90 °C, ramp them by no more than 5 °C every few hours. If there is more than a bubble emitted per 30 seconds, do not increase the temperature.
6. On the third and fourth days, ramp the samples to their final temperature (135 °C for the smaller bottles on the high-level side, 140 °C for the larger bottles on the low-level side). They should sit at final temperature over night to ensure complete dissolution.
7. Leave the samples heating until they are all fully dissolved, the liquids will be clear and there will be only a few bubbles on the bottom and sides. There will be no floating material.

Example temperature ramping schedule:

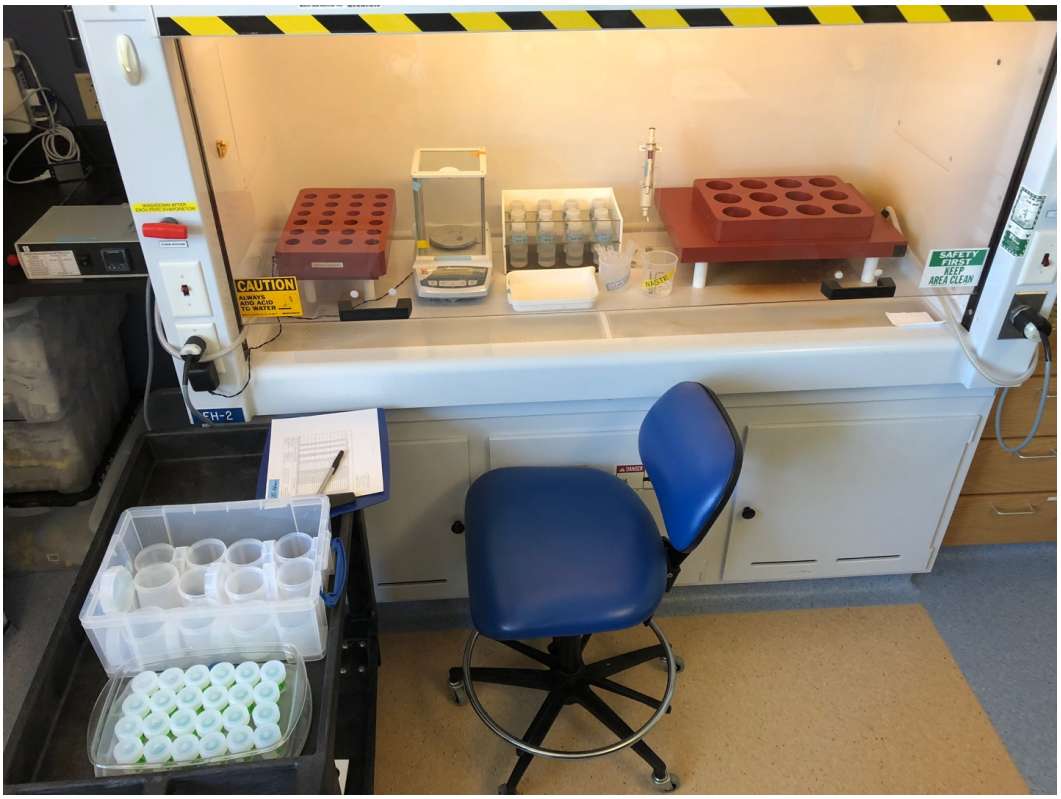
(This is an example only! The behavior of samples is highly variable; some may require much slower and more delicate treatment. Your highest priority is to keep the samples at a low enough temperature such that no visible movement is occurring. If you have any doubt, wait! If the samples seem volatile, work in smaller temperature increments and/or greater spacing). All numbers below are ℃.

Tuesday: 65, 75, 85
Wednesday: 90, 95, 100
Thursday: 105, 110, 115
Friday: 125, 135
(Turn off over the weekend, or allow the automatic shutdown early Monday morning to turn them off for you)

Removing ICP Aliquots

(Usually done on Monday of the extraction week)

Personal Protective Equipment: Thin gloves while setting up. Double-gloves (thin and thick), rubber smock, goggles, and face shield while handling HF, including at the end of the method while you are adding other acids to HF and washing dishes with traces of HF.



For this part of the procedure you will need: *(shown from left to right)*

- Cart pulled up next to the hood for additional storage
- 24 clean aliquot beakers
    - The first set labeled in blue tape with “2 mL”, the batch number, and A-L
    - The second set labeled in blue tape with “4 mL”, the batch number, and A-L
- 12 clean sample evaporation beakers (unlabeled!)
- Batch sheet and pen
- Small hot plate with aliquot drydown block on top
- Three-place balance
- Digested samples still capped and in the digestion block (with the digestion block lid removed)
- Small spill tray
- Large, clean beaker
- 12 new pipette tips for the 5 mL variable pipette, in the clean beaker, tip-down
- Waste beaker with ~50 mL water for diluting and storing used pipette tips
- 5 mL variable pipette on pipette stand
- Large hot plate with sample drydown block on top

1. This is a dangerous job because you are handling large amounts of HF in open beakers. Make sure someone else is around while you are working, and make sure not to block the grills along the front and back of the hood, since they are important for maintaining adequate air flow.
2. Do this method only if you are alert and feel 100%. This is important for safety, and also important because you will be working with unlabeled beakers.
3. Use the check weights (2, 10, 100 g) to verify the performance of the balance and record each check weight result on the clipboard hanging on the wall behind the balance table.
4. NOTE: Accurate weights require patience. Wait for vibration averaging to level out; the snowflake will appear and remain in lower left of balance screen. Only then, take the balance reading.
5. Set up for the first sample. Get the evaporation and ICP beakers, place lids in their respective boxes, and place the beakers in the spill tray. Put on a new pipette tip.
6. Take the first sample out of the digestion block.
7. Holding the bottle over the spill tray,carefully tip the bottle toward its side and roll to remove droplets from the lid. DO NOT SQUEEZE!
8. **MASS** the bottle with LID ON and record.
9. In the spill tray, pour the sample from the bottle into its evaporation beaker. Leave any solids behind even if you need to leave an mL or two of liquid with the solids.
10. Put the 2 mL (4 mL for low level) aliquot beaker on the scale and **TARE**; wait for TARE to stabilize.
11. Remove the tared beaker from the balance and place it back in the spill tray.
12. Check that the 5 mL variable pipettor is set to 2 mL (or 4 mL for low-level samples). Then slowly draw up 2 mL of acid (4 mL for low level) from the evaporation beaker and slowly release it into the ICP beaker.
13. **MASS** the aliquot beaker with the 2 mL aliquot in it (4 mL for low level) and **RECORD** the mass. Put the beaker on the small hotplate in its position in the A-L matrix.
14. Put the 4 mL aliquot beaker (8 mL for low-level samples) on the balance.
15. **TARE** the beaker.
16. Remove the beaker from the balance and place it back in the spill tray.
17. Do the same as before, but use twice as much sample volume (4 mL for high-level samples and 8 mL for low-level samples)
18. **MASS** the solution and **RECORD**. Put the beaker on the hotplate in its position, maintaining two separate A-L matrices.
19. Place the large sample beaker on the large hotplate to dry off in its proper A-L position, which is especially critical because this beaker is unlabeled.
20. When done with a sample, eject the pipette tip in the waste beaker, immersing it in water. Place the empty bottle back in the rack.
21. Repeat for all samples.
22. When finished, remove and put away the balance and 5mL variable pipette. Carry the digestion block with the sample bottles to the wash hood for cleaning. Drain the used pipette tips into the sink in the wash hood, rinse, and put them in the trash. Now, get set up for the next part:

For this part of the procedure you will need:

- Repipette and carousel pipette rack
- Spill tray
- Reservoir container of H_2SO_4, refilled from the larger acid jug if necessary
- Reservoir container of $HClO_4$, refilled from the larger acid jug if necessary
- Large clean acid beaker filled with Milli-Q for rinsing and eventually to store the $HClO_4$ pipette tip
- Dedicated 500 μL H_2SO_4 repipette tip
- Dedicated 25mL $HClO_4$ repipette tip with adapter

23. Snap the H_2SO_4 tip onto the repipette. Then, use the repipette to add 25 μL (dial set to 2.5) of concentrated H_2SO_4 to each ICP aliquot. We add the H_2SO_4 so that when the ICP splits dry down, they stay in single drop of acid and do not dry off (and fly away in a fit of static). You will need to refill the pipette tip part-way through.
24. After you are done using the tip, rinse it in milli-Q water in the "clean acid" beaker by filling/emptying several times. Put the tip back in its storage bag. Carry the wash water to the sink in the wash hood to dump, then bring the empty beaker back.
25. Snap the $HCLO_4$ tip onto the repipette. Then, use the repipette to add 2 mL of $HCLO_4$ to each of the 12 large beakers containing the samples. Release the acid slowly so as to prevent spatter and cross-talk between samples.
26. Eject the $HCLO_4$ tip into the clean acid beaker. Store the tip, the beaker, and the $HCLO_4$ reservoir container in the spill tray under the hood since you will need the same set-up repeatedly tomorrow.
27. Put away the H_2SO_4 tip and reservoir container.
28. In the wash hood, rinse out all the dissolution bottles with DI water to remove any undissolved material and HF.
29. If there is black material in or on the walls of the bottle, use a wipe and ethanol to remove the material. Be gentle, so as not to scratch the bottles. It may help to use a teflon stir stick wrapped in a wipe and soaked in ethanol to clean the deep recesses of the bottle.
30. Place the bottles and lids in a 4 L wash jug and sonicate overnight with 1% HNO_3 to clean.
31. In the wash hood, rinse off all materials used in the hood such as spill trays and pipette stands. Wash the digestion rack the same way and dry on the counter.
32. Set the SMALL hotplate to 120 °C to dry the aliquots off overnight.
33. Set the LARGE hotplate to 150 °C to dry the samples off overnight.

Perchloric Acid Drydowns

(Usually done on Tuesday of the extraction week)

Personal Protective Equipment: Double-gloves (thin and thick), rubber smock, goggles, and face shield while handling $HCLO_4$.

For this part of the procedure you will need:

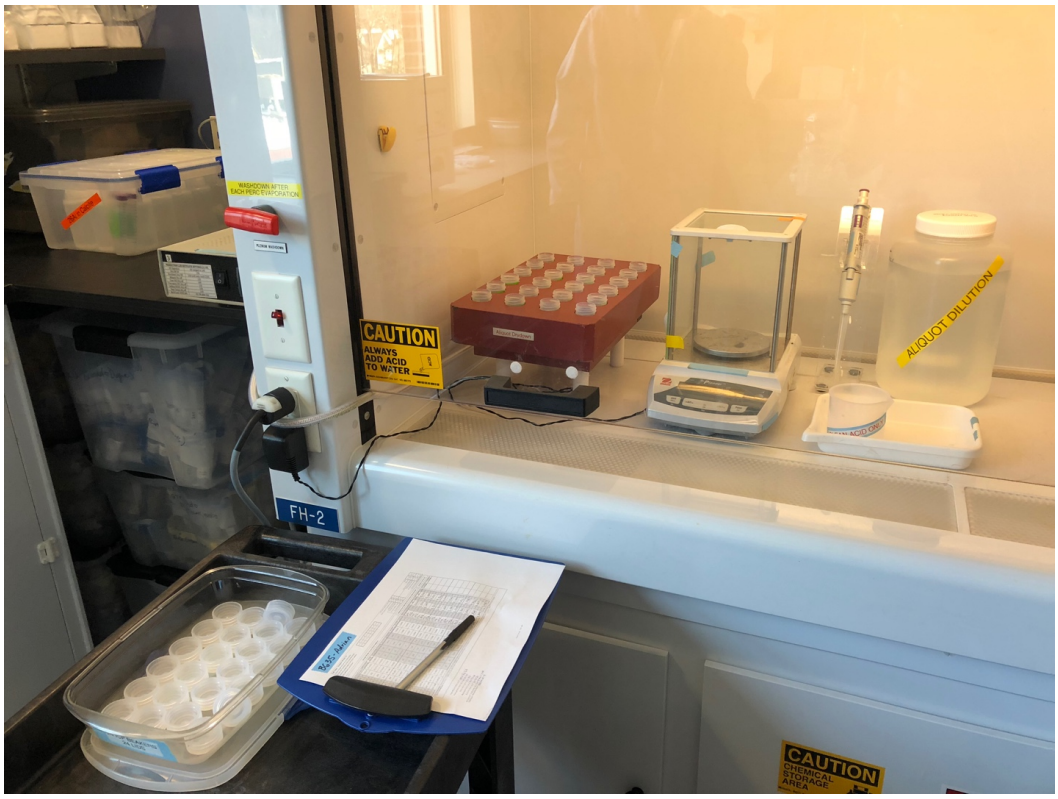
- Repipette and carousel pipette rack
- Spill tray
- Reservoir container of $HClO_4$, refilled from the larger acid jug if necessary
- Large clean acid beaker to store the $HClO_4$ repipette tip
- Dedicated 25 mL $HClO_4$ repipette tip with adapter

1. As soon as you arrive at the lab in the morning, turn the large hot plate with the samples up to 230 °C. It will take about an hour to reach the temperature.
2. You will need to work through the acid addition efficiently, so think about your approach and make sure you have everything you need before beginning. Your goal is to add the acid and get yourself and the labware out of the hood as safely but quickly as possible, since fumes will begin evaporating from the samples.
3. Using the repipette, add 2 mL of $HCLO_4$ to each sample using a circular motion to ensure the cake is fully wetted.
4. Release the tip into the empty clean acid beaker.
5. Place the spill tray with the $HCLO_4$ container, clean acid beaker, and $HCLO_4$ tip under the hood (keep everything together, you will need it again).
6. Remove the pipette rack and pipette and place to the side of the hood.
7. MAKE SURE THE SASH IS ALL THE WAY DOWN. Evaporate the acid until the white fuming totally stops (this will take about two hours).
8. REPEAT the process above (using 2 mL) one more time.
9. Before going home for the evening, repeat a final time but with 4 mL so that each sample is treated for a total of four Perchloric acid dry downs (including the co-evaporation with HF). Switch to the 50 mL $HCLO_4$ pipette tip to do this in order to accommodate the larger volume.
10. After you are done using the $HCLO_4$ repipette tips at the end of the day, rinse in milli-Q water by filling the clean acid beaker with milli-Q and filling/emptying to the sink several times. Rinse the beaker out with mill-Q and cover with parafilm.

Diluting the ICP aliquots

(Usually done on Tuesday of the extraction week)

Personal Protective Equipment: Thin gloves, goggles, and lab coat.

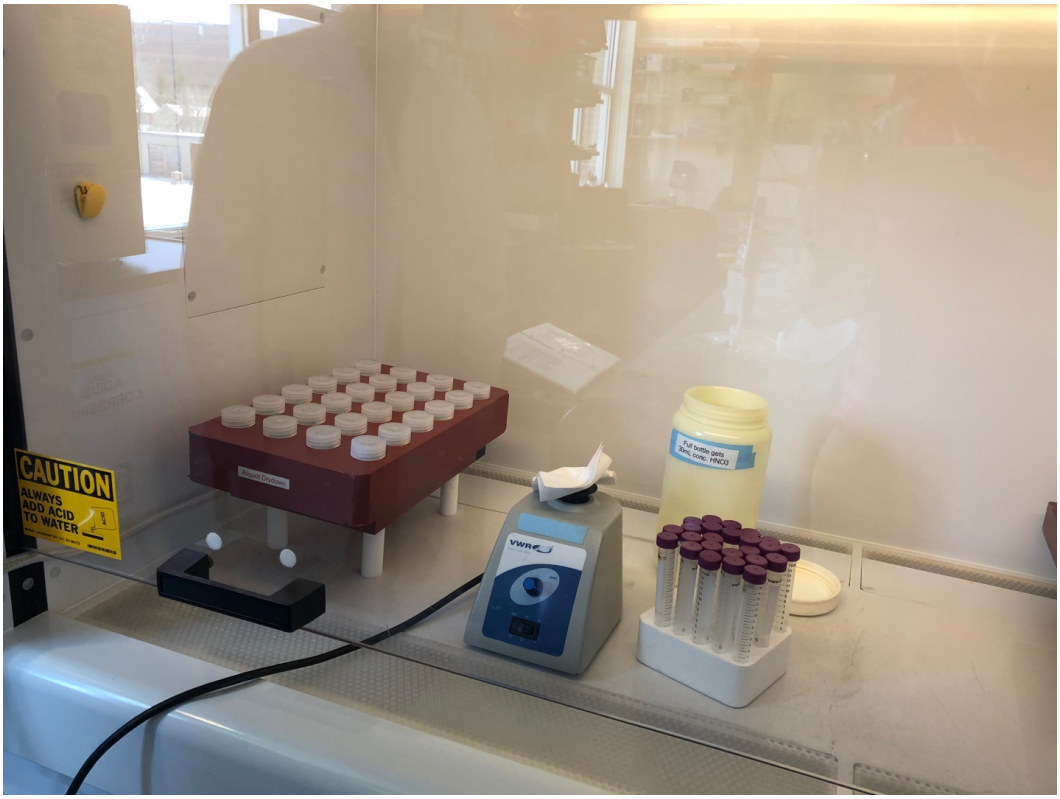


For this part of the procedure you will need: *(shown from left to right)*

- Cart pulled up next to the hood for additional storage
- 24 clean caps for the small aliquot beakers
- Batch sheet and pen
- Small hot plate with aliquot drydown block, holding the aliquots that dried overnight
- Three-place balance
- 5 mL variable pipette, on pipette rack and with a new tip
- Small spill tray
- Small clean acid beaker to hold ~125 mL aliquot dilution solution
- Large jug of aliquot dilution solution

1. Set the small hot plate to 60 °C and allow it to cool for half an hour.
2. Only dilute the aliquots after the initial Perchloric acid has dried off from the large beakers and NO FUMING is occurring.
3. Pour about 125 mL of Ga/Y spiked 0.5% H_2S0_4 into the clean acid beaker.
4. One at a time, remove a beaker from the rack and tare.
5. Place the tared beaker back in the spill tray.
6. Add 5 mL of Ga/Y spiked H_2SO_4. Then move the beaker back to the balance and record the mass on the batch sheet.

7. Cap the beaker and tighten the lid to avoid any evaporation. Place the beaker back in its designated hole on the hot plate
8. Repeat for all beakers.
9. LET THE BEAKERS SIT ON THE HOT PLATE AT 60° C FOR AT LEAST AN HOUR. Using less time could result in incomplete dissolution.



For this part of the procedure you will need: *(shown from left to right)*

- Small hot plate with aliquot drydown block, holding the diluted, capped aliquots that have sat for at least one hour
- Vortex, with small wipe over the rubber foot
- Small (2 L), empty wash jug to hold aliquot beakers after transfer
  Disposable rack with 24 unwashed, unlabeled, purple-capped centrifuge tubes

10. Once the aliquots have sat for an hour and at a time when no acid is fuming from the perchloric drydowns, set up for the next step.
11. One at a time, take an aliquot beaker and make sure the lid is tight. Then vortex for ten seconds.
12. Transfer the label on the beaker to a purple-capped centrifuge tube.
13. Transfer the liquid from the beaker to the same centrifuge tube. Do not rinse the beakers into the tubes because the volume matters.
14. As soon as you are done with a beaker and lid, drop it into the wash jug.
15. Repeat for all beakers.
16. When you are finished, sonicate the aliquot beakers in 1% HNO_3 overnight in the ultrasound.

17. Use a piece of tape to label over the top of the tubes in their rack with the words ALIQUOTS, BATCH-XXX.
18. Put the rack and tubes in the white acid storage cabinet.

Example Tuesday Schedule

You will be working on both the samples themselves as well as the aliquots. Your goal is to intersperse the two procedures such that you are never working in the hood while perchloric acid is fuming from the samples.

9:00AM: Arrive and adjust both hot plates (large one to 230 °C, small one to 60 °C)
9:30-10:30AM: Dilute aliquots by mass and let sit
10:30AM-12:30PM: Perchloric #2 (with #1 being the co-evaporation with HF overnight)
12:30-1:00PM: Transfer aliquots to tubes
1:00-3:00PM: Perchloric #3 (and, for some weeks, mass in the next batch of samples)
5:00PM: Add perchloric #4 before departure

Chloride Conversion

(Usually done on Wednesday morning of the extraction week)

Personal Protective Equipment: Double-glove (thin and thick), goggles, and smock because there is residual perchloric acid on the beaker rims.

For this procedure you will need:

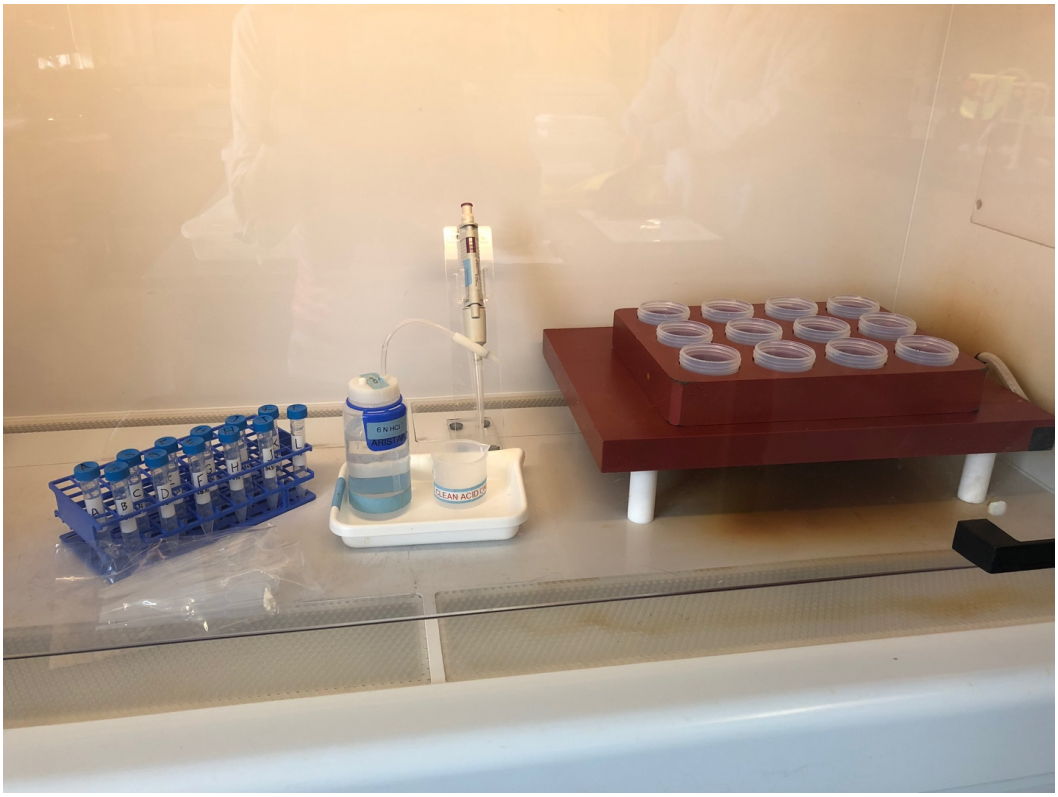
- Large hot plate with samples that have cooled down to the working temperature
- Spill tray
- Reservoir container of HCl, refilled from large acid jug if necessary
- 5 mL variable pipette, on rack and with a new tip

1. As soon as you arrive at the lab, turn the hotplate down to 160 °C and wait until hotplate and samples have cooled.
2. Gently add 2 mL concentrated HCl to each sample using the 5 mL variable pipette. Aim for the bottom edges of the beaker, using a circular motion to help redissolve the cake.
3. One at a time, pick up the sample beaker, bring it toward the front of the hood, and gently swirl so as NOT TO SPLASH. Check and make sure each cake has gone fully back into solution.
4. When you finish, put away the acid and wash the spill tray. Put the pipette and rack to the side since you will use them later, but make sure not to leave the pipette in the hood during the acid evaporation.
5. Wash the outer gloves well in case you touched perchloric acid on the beaker rims.
6. Heat the sample at 160 °C to drive off the HCl, which usually takes about 90 minutes.
7. When samples have completely dried, turn the hot plate down to 60 °C so that you can handle the beakers; this will take about half an hour.

Sample Preparation for Anion Columns

(Usually done on Wednesday of the extraction week)

Personal Protective Equipment: Double-glove (thin and thick), goggles, and smock because there is residual perchloric acid on the beaker rims.



For this procedure you will need: *(shown from left to right)*

- Plastic rack with 12 acid-washed, blue-capped centrifuge tubes, labeled A-L
- Bag of disposable transfer pipettes (opened at the bulb end instead of the tip end)
- Small spill tray
- Squirt bottle of anion column 6N HCl
- Small clean beaker to hold ~65 mL 6N HCl
- 5 mL variable pipette, on pipette rack and with a new tip
- Large hot plate with sample drydown block, holding samples that have been through the chloride conversion and have cooled to 65 °C

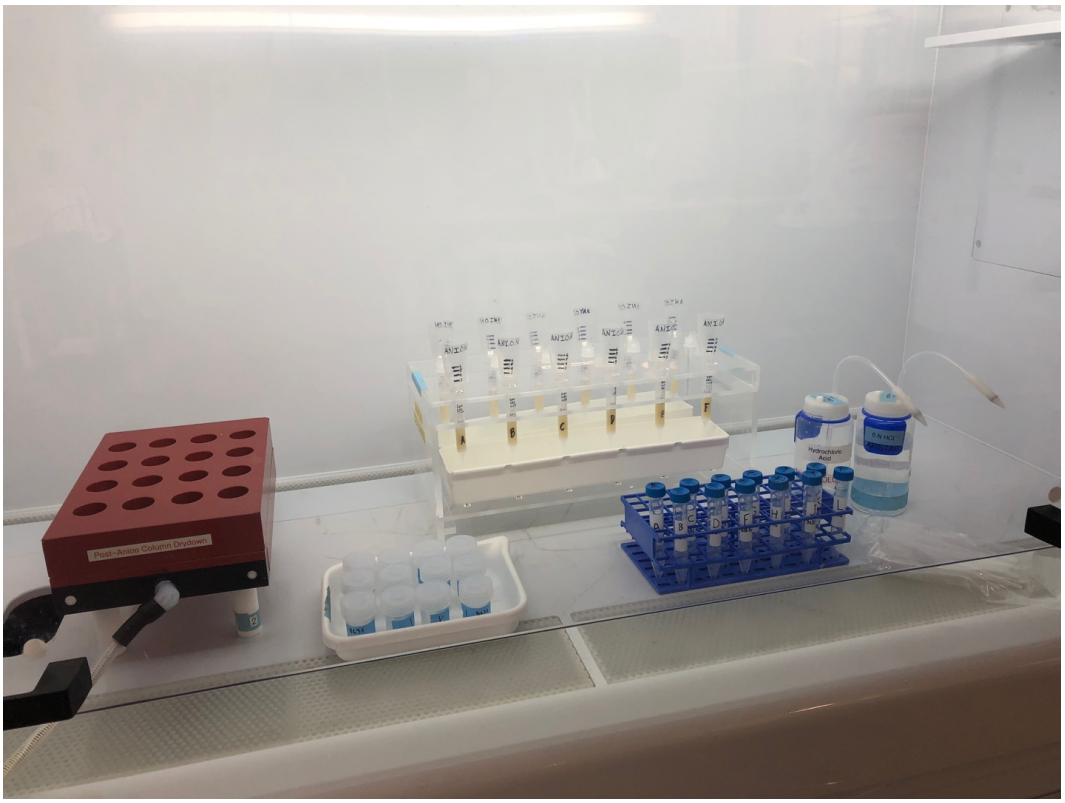
1. Using the 6N HCl Teflon squirt bottle, add about 65 mL of 6 N HCl to the clean acid beaker.
2. Remove the first sample from the hotplate and set on a spill tray.
3. Using the 5 mL variable pipette, add 2.5 mL of 6 N HCl to the sample. Swirl to dissolve.
4. Add a disposable transfer pipette to the beaker and use it to help break up the cake and get sample back in solution. Then, transfer the sample to its labeled, acid-washed 15 mL tube being careful not to drip.

5. Add an additional 2.5 mL of 6N HCl to the sample beaker to serve as a wash. Use the transfer pipette to wash down the sides of the beaker several times. Then, transfer the wash solution to the corresponding sample tube.
6. Repeat in order, B-L for all the remaining samples, **making sure to only have one tube and one beaker at a time in order to avoid mix-ups.** Double check to make sure each tube has 5 mL of solution.
7. Centrifuge the tubes for ten minutes at 3500 rpm.
8. Place the disposable pipettes in Be waste bag. Carry the rack of empty beakers to the wash hood and put it in the sink. Wash the rack and the beakers generously with the DI water spray to get rid of all traces of $HClO_4$, then sonicate the samples in 1% HNO_3 overnight in two separate 4 L wash jugs.
9. When the centrifuge is done, place the tubes back in the rack gently so as not to disturb the plug of material at the bottom and bring the rack to the column hood.
10. Now that you no longer have open samples in the digestion hood, run the hood washdown by turning the red handle and letting the water run for 30 seconds. This removes acid from the hood plenum and lower exhaust where most of the acid has condensed.

Anion columns

(Usually done on Wednesday afternoon of the extraction week)

Personal Protective Equipment: Thin gloves, goggles, and lab coat.



For this procedure you will need: *(shown from left to right)*

- Small hot plate with post-anion column drydown block
- Small spill tray
- 12 anion beakers, labeled in blue tape with A-L
- Anion columns and reservoirs, set up in rack with waste trays
- Plastic rack with 12 samples in tubes, diluted in 6N HCl and centrifuged
- Anion column 1.2N HCl squirt bottle (remixed when empty)
- Anion column 6N HCl squirt bottle (remixed when empty)
- Bag of disposable transfer pipettes (opened at the bulb end instead of the tip end)

1. **NOTE: the bottom cap on the columns must always stay on longer than the top cap or risk injecting air into the columns, destroying them and requiring replacement.**
2. Remove the top caps and place them in the cap bag. Place the anion reservoirs (stored in bags) on the columns. Remove the bottom caps.
3. Let the water in the columns drain out completely.
4. Column Stripping: Take out the 1.2N HCl squirt bottle. Squirt in the 1.2N HCl to the 5 column volume mark on the reservoir. Fill all the columns on one side of the

rack (that will be about 15 mL of acid), then rotate the rack and fill in the remaining columns. This weak acid will ensure the columns are clean.

5. Column Conditioning: Next, add 3 column volumes (9 mL) of 6N HCl to condition the columns. Let this volume drain through.
6. After ALL acid has drained, place the labeled beakers under the correct column. **THIS IS CRITICAL**. If you don't put the beakers under the columns now, you will lose your samples.
7. Sample Loading: Open up the 15 mL tube containing sample A**,** gently suck up the supernatant trying not to disturb the plug of material at the bottom using a new disposable transfer pipette. Try and remove as much liquid as you can from the test tube without drawing up ANY of the plug at the bottom. It will usually take several pulls of liquid to transfer the sample. It is okay to leave several mm of liquid above the plug; much better to lose a bit of sample than to introduce precipitate into the column (which will plug the column, requiring its replacement and resulting in sample loss). Get the pipette down to the frit and gently drip the supernatant into the first column. Once done, leave the pipette in the empty tube.
8. Repeat for all samples and let all samples drain into their respective beakers.
9. Once all samples are transferred, dispose of tubes, pipettes, and caps into Be waste.
10. Sample Elution: When all the samples have COMPLETELY drained in, add another 3 column volumes (9 mL) of 6N HCl to elute the remaining Be and Al into the anion beakers.
11. When the columns finish dripping, place the anion beakers in their rack on the small hotplate.
12. Return the waste bins to the column rack.
13. Column Stripping: Take out the 1.2N HCl squirt bottle. Squirt in the 1.2N HCl to the 5 column volume mark on the reservoir.
14. Column Washing: Fill each column reservoir to top with Milli-Q and let drain completely to waste. Put the bottom cap on first. Fill the column with Milli-Q to the bottom of the textured ring. Put on the top cap.
15. Dump the waste trays into the wash hood sink carefully with the water running.
16. Rinse the waste trays and column rack thoroughly with DI, place the trays back in the column rack, and dry both on the counter.
17. Acid wash the inside of the column reservoirs with the 1% HNO_3 squirt bottle and rinse with copious milli-Q water. Dry in the oven.
18. Put away the column acid bottles under the hood.
19. Now that you have cleaned up, finish the last few steps on the samples:

For this part of the procedure you will need:

- Small hot plate with post-anion column drydown block and open sample beakers
- Small spill tray
- Reservoir container of H_2SO_4, refilled from the larger acid jug if necessary
- 1 mL variable pipette, on rack and with a new tip

20. Add 140 µl of concentrated H_2SO_4 to each sample beaker. This is a CRITICAL step, if you forget it the cation columns the next day will fail.
21. Pipette the acid slowly; H_2SO_4 is very viscous.
22. Dry off at 110 °C overnight.

A Note About Column Acids

There are different acids for anion and cation columns. There is a reason for this. Acid strength is CRITICAL for cation column performance. It is much less critical for anion columns.

For anion columns, it is okay to make more acid and use mixed batches if you run out mid-way through doing columns.

For cation columns, we want to make sure that the same acid solution is used for all samples in a batch. Both storage and wash bottles are clearly labeled with red tape indicating whether they are to be used for cation or anion chemistry. For the cation columns, both the 0.65 M H_2SO_4 and the 1.2 N HCl are mixed in 2 L stock solutions and then transferred to the proper squirt bottles. The stock solutions are enough for exactly three batches. If you mix and match, you will end up with odd amounts of column acids. It is usually a good idea to check your cation acids on Wednesday and remix as necessary so that you are prepared for the following day.

Post-Anion Clean Up
(Only necessary when using new anion columns!)

(Usually done on Thursday morning of the extraction week, if needed)

Personal Protective Equipment: Thin gloves, goggles, and lab coat.

For this procedure you will need:

- Small hot plate with post-anion column drydown block and samples, evaporated down to large beads of H_2SO_4
- Small spill tray
- Reservoir container of Milli-Q water with trace H_2SO_4, remixed as necessary
- Dropper bottle of H_2O_2
- 5 mL variable pipette, on rack and with a new tip

1. Once the post anion drydown is complete, the samples should be completely clear beads of H_2O_2. However, if the anion column resin is new, the beads of acid may look brown and opaque. In that case, additional clean-up is necessary.
2. Add 7 drops of 2% peroxide to each beaker from the dropper bottle.
3. Next, add 2 mL of Milli-Q water with a trace of H_2SO_4 to each beaker using the 5 mL variable pipette. Do this very slowly to avoid splash.
4. Swirl each beaker gently to dissolve the sample. The resulting solution will be yellow to reddish brown in color. Return the sample to the hotplate and evaporate at 110 °C to remove the water. This will take about two hours. As the peroxide evaporates and reacts, the samples will go from red/brown back to clear or nearly clear with little color.
5. Repeat as necessary until the samples are completely clear after the evaporation is complete. This may take up to three times for the first usage of anion column resin.

Cation Columns

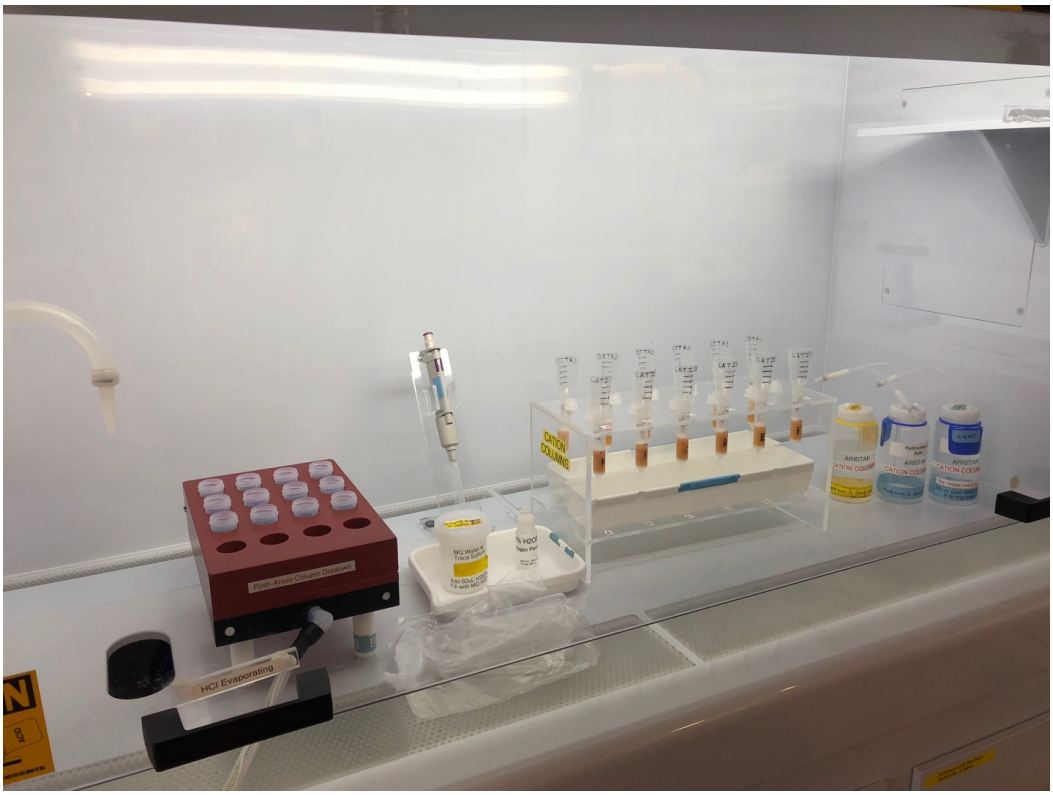
(Usually done on Thursday of the extraction week)

Personal Protective Equipment: Thin gloves, goggles, and lab coat.

NOTE: Capping is key. The bottom cap always needs to be on longer than the top cap so that air is NEVER forced into the resin bed or else…

NOTE: Do not leave the columns dry any longer than absolutely necessary or risk having them get air bubbles and need replacement.

NOTE: Make sure you have enough of all elution solutions to do you WHOLE BATCH before you start. If not, mix new solutions. Solution concentration is absolutely KEY – do dilutions accurately or risk losing your samples.



For this procedure you will need: *(shown from left to right)*

- Small hot plate with post-anion column drydown block
- (Eventually, swap in the post-cation column drydown block)
- Small spill tray
- Trace H_2SO_4 solution
- Dropper bottle with H_2O_2
- Bag of disposable transfer pipettes
- 5mL variable pipette, on pipette rack and with a new tip
- Cation columns and reservoirs, set up in rack with waste trays

- Cation column 0.65M H_2SO_4 squirt bottle (refilled from 2 L stock solution)
- Cation column 1.2N HCl squirt bottle (refilled from 2 L stock solution)
- Cation column 4N HCl squirt bottle (remixed at the beginning of each sample batch)
- Not shown: three sets of 12 beakers (pre-labeled, stored in their boxes until needed)
    - 12 tall beakers, labeled in red tape with the batch number, "Ti", and A-L
    - 12 tall beakers, labeled in blue tape with the batch number, "Be", and A-L
    - 12 short beakers, labeled in orange tape with the batch number, "Al", and A-L

1. Turn down the hotplate to 60 °C.
2. Place the waste bins under the column rack. Take the top cap off, then the bottom cap to drain all the water from your columns. Add the reservoirs. Put the caps in a clean place.
3. Column Conditioning: Condition the columns by filling the reservoir with 5 column volumes of 0.65 M H_2SO_4 (spiked with peroxide) from the squirt bottle. Let the columns drain.
4. Starting with sample "A", dilute the sample in 2 mL Milli-Q water with trace H_2SO_4 and add 7 drops of 2% H_2O_2. The sample will be yellow/orange/red in color. Use a new disposable transfer pipette to make sure the sample is well mixed.
5. CRITICAL – examine the sample in the pipette. Do you see any small, needle like crystals? If so, STOP. Put the sample back in its beaker, get a clean acid washed 15 mL tube, and centrifuge the sample to remove the crystals. They are likely gypsum. After centrifuging, transfer the supernatant only to the column as below. Discard the tube (and the crystals) as Be waste.
6. Loading Sample: Add the sample gently to the frit of its respective column using the pipette. Double check that you are loading the right sample into the right column.
7. Rinsing Sample beakers: Add another 2 mL of Milli-Q water with a trace of H_2SO_4 to the sample beaker. Use the disposable pipette to wash down the walls of the beaker, then add the wash solution to the proper column. DOUBLE CHECK that you are adding the right sample to the right column.
8. Repeat the process of diluting, loading, and washing for all 12 samples.
9. Once the samples have been added, put the empty beakers in a small (2 L) wash jug to sonicate in 1% HNO_3 overnight and dispose of the pipettes as Be waste.
10. Let the solutions drain fully into the columns.
11. Remove the waste trays and put the labeled Ti beakers (tall, 30 mL) under each column. Double check to make sure they are correctly located.
12. Titanium Elution: Use the squirt bottle to slowly add 5.75 column volumes of 0.65 M H2SO4 to the reservoir. Let the columns drip until the liquid is fully drained. **Watch this elution!** The red band of Ti should move through the column and completely elute. If there is any yellow color remaining in the lower tip of the column (this can happen with high Ti samples), decant several mL from the Ti beaker to waste and add another 5 mL of **0.65 M H2SO4 to that column only!** This will complete removal of Ti with minimal loss of Be. Once all columns have drained, remove, cap and store Ti beakers under the hood until yield checks are complete. Remember that you will need to empty and wash these beakers after verifying sample yield and purity.

13. Place the Be beakers (tall, 30 mL) under the column rack in their proper numbered positions. Double check to make sure they are correctly located.
14. Beryllium Elution: Use the squirt bottle to slowly add **1.2 N HCL** to each reservoir. Add 5.5 column volumes of acid. When the samples have completely drained through the columns, transfer the beakers to the drydown block on the hot plate, placing them in the deeper wells in A-L order.
15. Nitric Addition: Add 7 drops of **8 M HNO_3** to each Be sample beaker. This step is rumored to reduce boron in the samples.
    Note: these next few steps shown in gray are only necessary if you will be doing $^{26}Al/^{27}Al$ analyses at PRIME. If you plan to do this (or even think you might want to eventually), execute the Mg removal procedure below. If you are sure you will not be doing $^{26}Al/^{27}Al$ analysis at PRIME, skip the Mg removal process since it is unnecessary and proceed to step #19 below.
16. Following the Be elution, place the waste trays back under the columns.
17. Magnesium Elution: Add 10 column volumes of 1.2N HCl to each column. Note that this will not all fit in the reservoir at one time, so do five, let the solution drain in fully, and do another five. Because you will use so much 1.2N HCl, you will need to refill the squirt bottle from the stock solution several times and will need to remix the stock solution every two batches.
18. Carefully remove the waste trays, carry them to the sink in the wash hood, and put the acid down the drain while running water.
19. Place the Al beakers (shorter, 22 mL) under the column rack in their proper numbered positions. Double check to make sure they are correctly located.
20. Aluminum Elution: Use the squirt bottle to slowly add 4 column volumes (20 mL) of 4 N HCl. When the samples have completely drained through the columns, set all the beakers on the small hotplate in the shallow holes to dry down.
21. Column Stripping: Replace the waste trays. Add 4 additional column volumes of 4 N HCl and let it drain fully. This strong acid will strip any residual Be and Al off the columns and prepare them for the next use.
22. Column Washing: To rinse the columns and remove all acid, fill the reservoir completely with milli-Q water and let it drain.
23. To store the columns: Remove the reservoirs. Cap the bottom first, fill column with water to bottom of textured area, then install the top cap.
24. Dump the waste trays into the wash hood sink carefully with the water running.
25. Rinse the waste trays, column rack, and spill trays thoroughly with DI, place the trays back in the column rack, and dry both on the counter.
26. Acid wash the inside of the column reservoirs with the 1% HNO_3 squirt bottle and rinse with copious milli-Q water. Dry in the oven.
27. Put away the column acid bottles under the hood.
28. Dry the samples down overnight with the hotplate set at 110 °C.

Redissolving

(Usually done on Friday morning of the extraction week)

Personal Protective Equipment: Thin gloves, goggles, and lab coat.

For this part of the procedure you will need:

- Hot plate with 24 dried down Be and Al fractions
- Spill tray
- Reservoir container of 1% HNO_3, remixed for this method
- Repipette and carousel rack
- Dedicated repipette tip for HNO_3
- Lids for sample beakers

1. After drydown overnight, the Be beakers should contain a small, clear bead of liquid (which is H_2SO_4, leftover from the cation column procedure); the Al beakers should contain a white cake.
2. Turn hotplate down to 60 °C and allow to cool for half an hour.
3. Using the repipette, add 8 mL of 1% HNO_3 to each sample beaker. Add the acid very slowly, especially to the short Al beakers, to prevent splash.
4. Tightly cap all beakers and leave them on the hotplate for 10 minutes to redissolve.
5. As the beakers sit, prepare for the next steps:

For this part of the procedure you will need: *(shown from left to right)*

- Small hot plate with post-cation column drydown block and the diluted, capped samples
- Vortex, with small wipe over the rubber foot
- Plastic rack with 24 acid-washed, blue-capped centrifuge tubes
    - 12 tubes labeled in blue tape with A-L, the batch number, "Be", and sample name; also lids labeled A-L
    - 12 tubes labeled in orange tape with A-L, the batch number, "Al", and sample name; also lids labeled A-L
- Disposable rack with 24 unwashed, purple-capped centrifuge tubes
- Large (4 L), empty wash jug for Be beakers (and eventually Ti beakers)
- Small (2 L), empty wash jug for Al beakers

6. One at a time, take out a sample beaker, the corresponding blue sample tube, and a purple tube. Set all three toward the front of the hood (in a separate rack or held in your hand) so that it is clear which you are working on.
7. Make sure the sample beaker is tightly capped, then vortex for ten seconds.
8. Uncap the beaker, and carefully pour the liquid into the blue sample tube, double-checking to make sure that the position letter (A-L) matches. Tighten the blue tube cap.
9. Remove the label from the beaker and put it on the empty purple tube (which will eventually be used for the ICP).
10. Place the empty beaker and lid in the appropriate wash jug.
11. Place both tubes back in their respective racks and get the next two tubes.
12. Proceed through the 12 Be fractions, placing the beakers in the large (4 L) wash jug, which can eventually be used to hold the Ti beakers as well (segregating the beakers by size during washing avoids having to sort them later).
13. Then do the same the 12 Al fractions, placing the beakers in the small (2 L) wash jug.
14. Fill the small wash jug with 1% HNO_3 and sonicate overnight. Wait to fill and sonicate the large wash jug until the yields have been tested on the ICP and the Ti beakers have been added.

Removing yield testing aliquots

(Usually done on Friday morning of the extraction week)

Personal Protective Equipment: Thin gloves, goggles, and lab coat.

NOTE: It is critical to avoid cross-contamination during this procedure. Make sure your pipette body does not touch the edge of the test tube. If it does, wipe down the pipette body with Milli-Q and a wipe.

For this procedure you will need: *(shown from left to right)*

- Box of tips for the 1 mL variable pipette
- 1 mL variable pipette, on carousel rack
- (Swap in 100 µL variable pipette and associated tips when you switch from Be to Al)
- Waste beaker for holding used tips
- Plastic rack with 24 samples in centrifuge tubes
- Vortex, with small wipe over the rubber foot
- Disposable rack with 24 empty, labeled, purple-capped centrifuge tubes
- 5mL variable pipette, on pipette rack and with a new tip
- Small spill tray
- Small clean beaker to hold ~125 mL yield test solution
- Large jug of yield test solution

1. Decant ~125 mL of the yield test solution into the clean acid beaker.
2. In your hand or in a separate rack, hold the first blue-capped Be fraction tube (sample A) and the corresponding empty purple tube.
3. Place a new tip on the 1 mL pipette.
4. Without touching the pipette body to the tube (which could cause cross-contamination), remove a 200 μL aliquot from the Be fraction and squirt it into the purple tube.
5. Eject the used 1 mL tip immediately into the waste beaker so that you cannot inadvertently reuse it.
6. Replace the cap on the blue (sample) tube to ensure that your sample is protected.
7. Using the 5 mL variable pipette, dilute the aliquot in the purple tube with 5 mL of yield test solution (there is no need to replace this pipette tip since it is only touching the dilutant).
8. Replace the cap on the purple (ICP) tube and vortex it for five seconds.
9. Replace both the blue and purple tubes in their respective racks, and bring the next set of tubes to the front of the hood.
10. Repeat for all Be fractions.
11. Once you finish the Be fractions, place the used tips into the Be waste bag. Put away the 1 mL variable pipette and tips.
12. For the Al fractions, repeat the procedure above using the 100 μL pipette and the appropriate tips. From each Al fraction, remove a 50 μL aliquot. Remember to eject the used tip after every sample.
13. When you finish, the Al tips can go in the trash. Put away all pipettes. Wash the spill tray and pipette racks in DI. Label the purple tubes “BXXX Yields” with a piece of tape over the top of the rack.

Precipitating and Washing Hydroxide Jells

(Usually done on Friday afternoon of the extraction week)

Personal Protective Equipment: Thin gloves, goggles, and lab coat.

For this procedure you will need: *(shown from left to right)*

- Plastic rack with 24 samples in centrifuge tubes
- Small spill tray
- Waste beaker
- 30% NH_4OH dropper bottle
- Methyl red dropper bottle
- Milli-Q water squirt bottle
- Vortex

1. Get your first Be fraction (sample A) and bring it toward the front of the hood, either holding it in your hand or standing it in another rack.
2. Add one drop of methyl red (a pH indicator). Try to avoid any drops since they stain the hood deck and spill trays.
3. Using the 30% NH_4OH dropper bottle, add 10 drops to the Be sample tube. Cap, shake and look for the color to change to yellow. If there is no color change, add one more drop, shake again. Keep adding drops one at a time until the color just changes from red to yellow. Now, shake to ensure the sample is well-mixed and stays yellow. Then add one more drop of 30%. Shake sample very well – check color again.

4. Repeat for all Be fractions. They will likely take ~12-15 drops each (you are neutralizing the 1% HNO_3 and also one column volume of 0.65 M H_2SO_4), but it depends on how the 1% HNO_3 was mixed and will vary batch by batch. It will also vary slightly sample to sample, so proceed slowly and make sure not to go over.
5. For the Al fractions, do the same as above but add only 3 drops of 30% to the first tube of Al then titrate dropwise. The Al fractions neutralize more readily because there is no H_2SO_4, only the 1% HNO_3.
6. After the pH titration, let the samples sit for at least an hour, but longer is fine (even numerous days).
7. Put all 24 tubes in the centrifuge, making sure it is balanced. Centrifuge for 10 minutes at 3500 rpm. Unload them carefully back into the rack.
8. **STOP** – if you do not see a pellet/jell in the bottom of the tube, check the pH with pH paper (do this by placing the pH paper on a watch glass and using a disposable transfer pipette to deliver liquid to the paper). **DO NOT** put the paper in the tube. Make sure solution pH is 8 adjusting with the HNO_3 and NH_4OH dropper bottles. Remix and recentrifuge; you should now see a pellet of jell.
9. From each sample tube, decant the liquid into a waste beaker, making sure the jell stays in the bottom of the tube.
10. To each tube, add 8 mL of mill-Q water, vortex for ten seconds, and check and see that the entire pellet is dispersed. This washing removes soluble cations such as Na and Ca. Move through this step efficiently since the water has a slightly lower pH than the solution from which you originally precipitated the jell.
11. Centrifuge again for 10 minutes at 3500 rpm.
12. As above, decant the liquid into a waste beaker, making sure to leave the jell in the bottom of the tube. Decant slowly but thoroughly so that no liquid is left in the tube.
13. If you are not analyzing ^{26}Al, label the Al fractions with the batch number and place them in the Al archive box. Update the archive inventory sheet on the box lid.

Drying Hydroxide Jells into Pellets

(Usually done Monday/Tuesday, or whenever the column hood is available for two days)

Personal Protective Equipment: Thin gloves, goggles, and lab coat.

For this procedure you will need:

- Beryllium and/or aluminum hydroxide gels with liquid poured off
- Small hot plate
- Metal tube drydown blocks (as many as three)
- Spill tray to hold caps

1. You can dry up to three sets of samples (i.e. 36 tubes) at a time.
2. Only dry down when the hood is otherwise unused to prevent contamination. This can usually occur on Monday/Tuesday when the column hood is vacant, or over the weekend if you will be able to cap up the dried pellets on Sunday (before the Monday morning shutdown).
3. Check to see if there is water over the jell that did not come out with decanting. If there is water, use a new disposable pipette for each sample to remove the water (if the water is not removed, the sample will not make a solid pellet but rather will coat the bottom of the tube).
4. Once the water is gone, take off the cap of tube A and place it in the dry block in standard A-L order. Repeat for all other tubes and arrange caps in a spill try upside down in order. NEVER PLACE JELLS IN THE OVEN.
5. Set the hotplate to 65 °C. If you use a higher temperature, the jells will not form pellets. This will take overnight at least. The next day, ramp the hotplate to 98 °C and leave the tubes for at least several hours; all day or overnight is better, especially for Al jells, which are larger and take longer. Use the mirror to **ensure all drops are gone from the tube walls before capping**, otherwise the pellet will become stuck on its way out of the tube.
6. Once samples are dry, cap with the proper cap (check letters) and set next to the window. Make sure the tubes are racked in order and add a piece of tape on the top indicating the batch number and the word “Dried”.

Scanning Batch Sheets

(Usually done on Friday afternoon of the extraction week)

1. Do this only after the batch is complete to ensure that all batch notes on the sheets get scanned.
2. Make sure the name of whoever is running the batch, the batch number, and the date are written in bold sharpie at the top of the batch sheet so they appear legible once scanned.
3. Scan the batch sheet into the computer BEFORE taping it into the Batch Book.
4. Click on the icon labeled, "Click to Scan Batch Sheets." If the One-click tab is not already open, open it and select "PDF."
5. Change the file name to batch_XXX where XXX is your batch number. If you have more than one sheet to scan for the same batch (i.e. if you took notes on a separate page), scan your Batch Sheet as batch_XXXa and any extra pages of notes as batch_XXXb, batch_XXXc, etc.
6. Ensure the scan is being saved to the "Scanned Batch Sheets" folder on the Desktop.
7. Click "Scan" to begin scanning. (NOTE: After many consecutive scans, clicking the "Scan" button will do nothing. If this happens, exit out of MP Navigator EX and re-open the application. Scanning should work after doing this.)
8. When the scan is complete, a "View & Use" window automatically opens. Nothing needs to be done in this window, so you can close it.
9. Check to make sure your batch sheet appears in the "Scanned Batch Sheets" folder on the Desktop. If you rotate the image, you will be prompted to "save changes to your document" upon exiting the program. Click "Yes."
10. In the "Scanned Batch Sheets" folder, highlight the file blue if it is a high-level batch sheet or yellow if it is a low-level batch sheet.
11. Now you may trim the batch sheet and tape it into the respective batch book.

Final Loose Ends

1. After running and verifying yields on the ICP, dump and clean the Ti beakers in the same wash jug as the Be beakers. Do this as soon as possible since all of these beakers will need to be clean and dry for the following week's extraction.
2. Run the aliquots on the ICP (usually done every month or several months) and reduce the data.
3. Only after all data are entered, color the file name for the batch sheet blue or yellow to match the hood in which the samples were run.

ACIDS AND STANDARDS

Principles of making acid and standards

- Concentrations matter, do the same thing every time.
- Add acid to water. Operationally this often means measuring in some water to the container, adding the requisite acid, and measuring in the remaining water.
- Agitate very well after making up the acid and before using
- Always work in the hood and with a spill tray under your vessels.

1. **Ga/Y Spike recipe (DO NOT MAKE THIS UP ALONE, ASK PAUL FOR HELP)**

   This is made up in a 4 liter nalgene wide mouth jar and lasts for many batches (about 30 batches). The concentration of Ga and Y are absolutely crucial and need to be matched to the standards or the internal standard correction will be bogus.

   a. Dump any residual solution from the stock bottle and DO NOT RINSE the bottle.
   b. Add several hundred mL of milliQ water to the volumetric flask.
   c. Use 5ml variable pipette to add 20 mL Aristar Plus conc sulfuric to the water in the volumetric flask.
   d. Mass 40.47 grams of Y standard into a clean, dry 125 mL HDPE bottle. This is 40 mL of standard in 2% nitric at density 1.0117 gms/cc. Pour bottle into the volumetric flask and rinse three times with mQ water into the flask.
   e. Mass 40.47 grams of Ga standard into a clean, dry 125 mL HDPE bottle. This is 40 mL of standard in 2% nitric at density 1.0117 gms/cc. Pour bottle into the volumetric flask and rinse three times with mQ water into the flask.
   f. Top up the volumetric to the 2000 mL line and add to the stock bottle.
   g. Fill the 2000 mL volumetric flask with 2 liters of mQ and add to the stock bottle. This functions as a rinse.
   h. Agitate the stock bottle very well for a minute.
   i. Weak acid wash the volumetric flask twice and rinse carefully with copious mQ water. Let air dry on counter then cap and put away.

2. **Acid recipes**

   a. Anion acid – **1.2 N HCl**, to make 500 mL in teflon wash bottle, add 50 mL of conc Aristar Plus HCl and fill (not precise acid dilution). Remake when needed.

   b. Anion acid – **6 N HCl,** to make 500 mL in teflon wash bottle, add 250 mL of conc Aristar Plus HCl and fill (not precise acid dilution). Remake when needed.

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c. Post anion acid – **trace sulfuric**, make in 250 mL teflon jar, fill jar with mQ water and add 60 ul (yes, that's micro liters) of Aristar Plus conc sulfuric acid. Remake every two batches.

d. Post anion oxidize, **2% Peroxide**, make in 30 mL dropper bottle, 2 mL of 30% peroxide and fill with mQ. Peroxide degrades over time. Replace every few months.

e. Cation acid - **1.2 N HCl,** to make 2000 mL in plastic HDPE wide mouth bottle, add 210 mL of conc Aristar Plus HCl and 1790 mL of mQ water (measure acid and water in 500 mL graduated cylinder). Remake every 3 batches. As needed, use this stock to refill Teflon wash bottles.

f. Cation acid – **0.65 M Sulfuric acid** with peroxide, to make 2000 mL in plastic HDPE wide mouth bottle, add 70 mL of conc Aristar Plus Sulfuric and 1920 mL of mQ water and 10 mL of 30% peroxide (measure acid and peroxide in 50 mL graduated cylinder and water in 500 mL graduated cylinder). Remake every 3 batches. As needed, use this stock to refill Teflon wash bottles.

g. Cation acid – **4 N HCl,** to make 500 mL in teflon wash bottle, add 170 mL of conc Aristar Plus HCl and fill to ring (not precise acid dilution, this dilution can be eyeballed). Remake every batch.

h. Post cation, dry off acid, **8N Nitric**, to fill 30 mL dropper bottle add 15ml mQ water and then 15 mL conc Omnitrace Nitric Acid (not precise acid dilution). Lasts for many, many batches.

i. Precipitation acid – **1% Nitric Acid**, to make in 250 mL teflon jar, fill jar with mQ water and add 3 mL (yes, that's milli liters) of conc Omnitrace Nitric Acid. Remake every batch.

j. Precipitation base – **15% NH4OH** to make 30 mL dropper bottle, add 15 mL of 30% NH4OH and fill to top.

k. Precipitation base – **30% NH4OH,** fill directly from stock solution bottle (not precise acid dilution, this dilution can be eyeballed).

3. Yield solution

a. Add 40 mL conc Aristar Plus Sulfuric and fill with mQ water in 4 liter stock solution jug.

4. Standards

a. **Quartz purity standards.** We make volumetrically a stock solution of 100 ppm cations using 25 mL each of Al, Be, K, Na, Ca, Mg, Fe and Ti SPEX 1000 ppm standards. These eight standards are added to a 250 mL HDPE bottle and 50 mL DI is added. We make the four working standards using the 1% Sulfuric acid solution used for yield tests and varying amounts of the stock solution.

1 ppm, 5 mL stock in 500 mL
2 ppm, 10 mL stock in 500 mL
4 ppm, 20 mL stock in 500 mL
8 ppm, 40 mL stock in 500 mL

b. **Aliquot standards**. These four standards are made and stored in 2000 mL glass volumetric flasks. The idea is to make a lot of standard so it can be used for many batches. Storage in glass prevents evaporation. The internal standard will correct for small amounts of evaporation.

- First, we made up 4 liters of solution to add to aliquots. This contains some sulfuric acid but not the entire amount to matrix match with standards because the aliquots are dried down with some sulfuric acid already and our goal is that the final aliquot dilutions have the same concentration of sulfuric acid as the standards. This aliquot solution is 4 liters (in a widemouth HDPE bottle) of 0.5% Sulfuric Acid. This requires 20 mL of sulfuric acid added with graduated cylinder and 20 mL each of Y and Ga. The Y and Ga are added gravimetrically by pipetting (using 5 mL variable) into the graduated cylinder on the balance. With a density of 1.0117, we add 40.47 g of standards. This solution is remade as needed, about every 30 batches.

- Second, we made up the four standards. We did this by adding some mQ water to each of the four pre-labeled volumetric flasks. Then, we massed the proper amounts of Al and Be standard into a clean, dry 250 mL HDPE bottle as well as proper amounts of Ga and Y standards to match the aliquot dilution solution. Note that Al carrier is in 2% HCl and Be carrier is in 2% Nitric Acid. We poured the standards into the volumetric flask. Then repeatedly rinsed the HDPE bottle with 1% Sulfuric Acid into the volumetric flask. We then topped up the volumetric flask with 1% Sulfuric Acid.

Conc (Be/Al)	Ga (mass std)	Y(mass std)	Be (mass std)	Al (mass std)
1/10 ppm	20.234	20.234	2.023	20.194
2/20 ppm	20.234	20.234	4.047	40.388
4/40 ppm	20.234	20.234	8.094	80.776
8/80 ppm	20.234	20.234	16.187	161.552

c. **Carrier calibration standards.** These are spiked with Ga and Y for ICP internal standardization. We make a blank, 2 and 4 ppm. These are made mass/mass 100 mL at a time. First, we made a stock solution, 500 total mL of 10 ppm Ga and 10 ppm Y in 2% nitric acid. This was done by massing in 5.07 grams of standard (density 1.0117 g/cc). The we made a second solution, just 2% nitric acid (14.3 mL in 500 mL mQ water). The we made 2 and 4 ppm standards by adding 200 ul of Be (1000 ppm, about 0.200 g) and adding 400 ul of Be (about 0.400 g) then adding an mL each of Y and Ga (which were massed. The total mass of the solution was brought to 101.17 (100 mL). See cosmolab start up notebook for original details.

Column Loading

We reuse and regenerate columns. Columns are replaced only if they develop air bubbles or have been compromised in some other fashion. Because of the fritting arrangement, columns themselves are not emptied but disposed of as trash when their time has come.

Loading Anion Columns (done only when columns need replacement)

1. Get out the anion resin (Arcos Dowex 1-8 200-400 mesh). Get out a spill tray, a clean acid beaker, and a disposable pipette.
2. Shake resin to slurry.
3. IF columns are new or dry, place half an mL of ethanol onto each column to wet the frit (if you forget the ethanol, the columns won't drip). Make sure to IMMEDIATELY clean up any ethanol drips. It will destroy the plastic racks by cracking. Let all the ethanol drip through.
4. Load the columns – always load into standing water to reduce the chance of bubbles. Load the first shot of resin into the six columns along the front of the rack. Let the resin settle from suspension. Then, add enough resin to fill the column to the base of the first shoulder. If you add too much resin, pipette it out. Repeat on the back side.
5. After the columns have all been loaded with the same and correct amount of resin, but before the water has drained out, add the upper frit pushing it down through the water until it is uniformly 1-2 mm above the resin bed.

Loading Cation columns (done only when columns need replacement)

1. Set out the column carousel and get out the reference column.
2. Set out the 12 columns that you will use; take out 12 frits and the frit pusher. Mark the 5 mL level on each column using a sharpie. Put a bottom cap on the column.
3. One at a time, fill the column 80% with MilliQ water. Tap the column to dislodge any air bubbles. Then float the frit on the water and use the frit pusher to slowly push the frit down the column until it is flat and square on the base. Repeat for all columns loading them after the frits are in onto the carousel.
4. Get out the CATION resin (Arcos Dowex 50WX8 200-400 mesh). Get out a spill tray, a clean acid beaker and a disposable pipette.
5. Shake resin to slurry.
6. Load the columns with resin – always load into standing water to reduce the chance of bubbles. Add about 2ml of MilliQ water into the column. Then, load the first shot of resin into the columns. Let the resin settle from suspension. Then, add enough resin to fill the column to the 5 mL mark. If you add too much resin, pipette it out. Once all columns are at 5 mL, then add more MilliQ water, resuspend the resin and let it settle out smoothly.
7. After the resin has settled but before the water has drained out, add the upper frit pushing it down through the water until it is uniformly 1-2 mm above the resin bed.

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HF Digestion	60 ramped to 140 (lo), 135 (hi)
Dry Off HF	150
Perchloric Dry Off	230 (will only reach 210)
Aliquot Dry off	120
Conc HCl Dry off	160
Post anion HCl dry off	110
Sulfate/H2O2 dry off	110
Post Cation dry off	110
BeOH and AlOH jells	65; 98 after dry

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