UVM Cosmogenic Laboratory: Meteoric ^{10}Be Extraction

Purpose: This method details the means by which we extract meteoric ^{10}Be (adhered to grains and in grain coatings) using the flux fusion method originally presented by Stone (1998). We have modified this method so that we can process 16 samples at a time, usually 15 samples and a blank. The method is performed in the meteoric laboratory only and uses dedicated sample processing gear and a stand designed to prevent any contact with the flux while it is molten.

Hazards: The primary hazards associated with this method are exposure to very high heat from the torch, as well as the hazards of potential exposure to potassium hydrogen fluoride and perchloric acid. Beryllium is a potent toxin, particularly as an airborne oxide.

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 KHF, HF, and $HCLO_4$. Add leather chaps when using the torch.

Avoiding Contamination: The amount of ^{10}Be in most meteoric samples is orders of magnitude greater than in most *in situ* samples. Therefore, nothing in the meteoric lab should ever leave the room. Under no circumstances should you ever bring something from the meteoric lab into the *in situ* lab.

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.

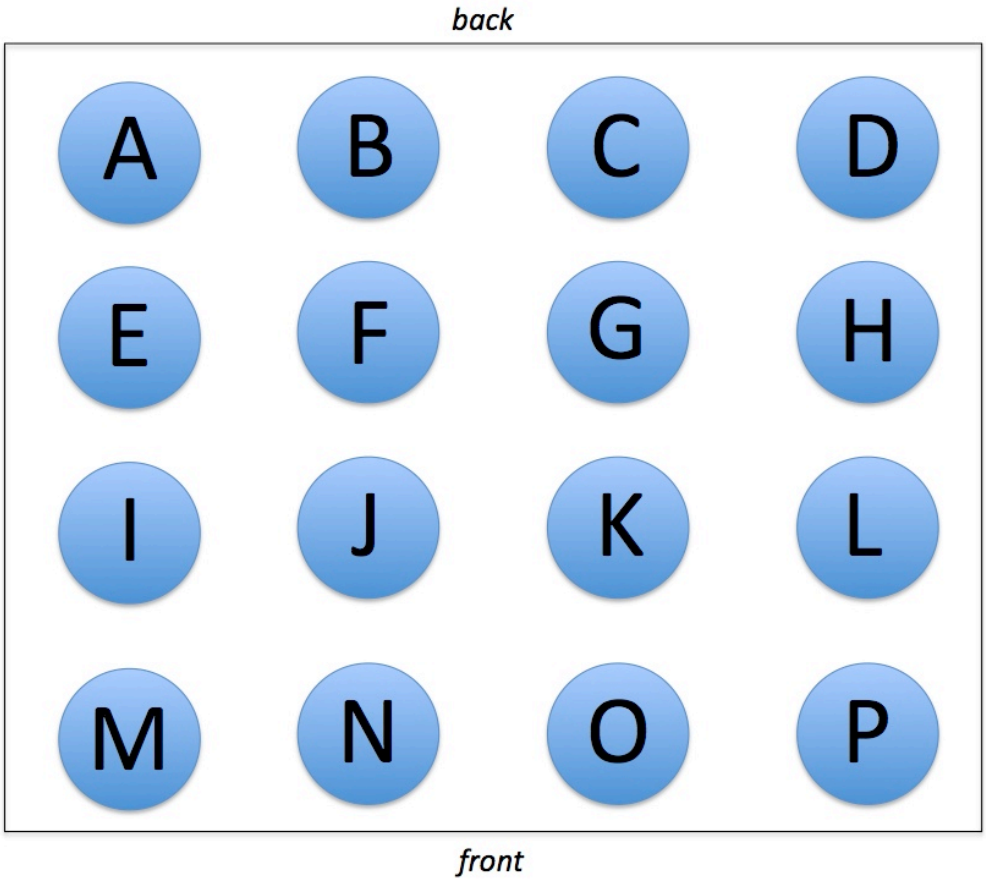
2. Always store the pipette vertically; never lie the pipette on its side. Vertical storage keeps any acid on the pipette from seeping into the inner workings.

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.

Important Notes

1. Cleanliness is imperative, not only for you and your samples, but for everyone who follows. Spilling, splashing, or failing to clean labware properly can contaminate your samples and the samples of everyone who follows you.
2. Sample identification is absolutely key since they will be unlabeled for parts of the process. In order to prevent sample confusion, use the same racking order every time (shown below).
3. The crucibles can ONLY touch clean surfaces since they are leached in water. Setting a crucible down on a non-clean or non-acid washed surface risks contamination. Never touch crucibles with gloved or ungloved fingers; always handle them with cleaned blue clips or Pt-tipped tongs.



Getting Ready

(In advance of your batch)

1. Samples must be fine powders for this method to work properly. If needed, use the SPEX shatter box in the rock room to produce these powders (see lab safety manual for the rock room for specific instructions).
2. Select 15 samples to process. The samples should be selected in the vestibule and then brought into the lab one batch at a time in 20 ml scintillation vials.
3. Note that it is important not to mix different types of samples together; high-level samples should not be extracted in the same batch as low-level samples. This segregation helps minimize the chance of sample cross-talk.
4. Check that there is pressure in the Oxygen tank in the vestibule. It can take several days for a new tank to be delivered, so check well in advance of when you plan to flux.

Preparing the Data Sheet

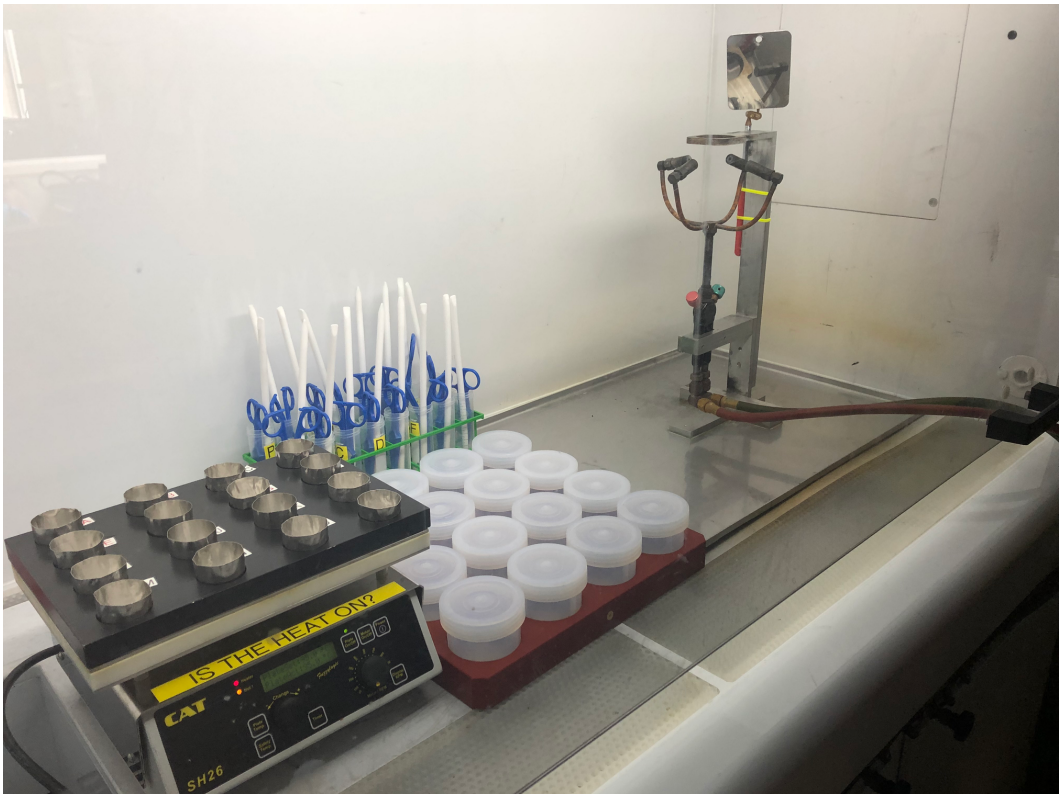
(Usually done on Thursday morning)

1. Open the folder on the desktop entitled “Meteoric Batches”.
2. Open file “Met_TEMPLATE”.
3. Save the file, changing the word “template” to your batch number. The file name format should be “Met_MB#”, where # is your batch number. Save the file in the folder “Meteoric Batches”. To find your batch number, note the last batch processed and add one.
4. Fill in the information at the top of the sheet (name, date, and batch number).
5. Add the name and concentration of the ^{9}Be carrier to the spreadsheet (UVM-SPEX in most cases).
6. Organize your samples in logical order; usually alphabetical or low to high number in a sequence.
7. Add all your samples ID’s to the spreadsheet (in your logical order). Include a blank (named BLK) randomly in your sequence.

Preparing the Lab

(Usually done on Thursday morning)

Personal Protective Equipment: Thin gloves and goggles.



1. Double check that there is pressure in the oxygen tank in the vestibule. Do not start a batch unless there is at least 10,000 kPa on the right-hand guage.
2. Carefully remove the large red hotplate from the hood after first wiping it down. Before removing the hotplate, you will need to disconnect it from the controller. Place the hotplate on the top of the cart. Be careful, the hotplate is very heavy and expensive.
3. Move the fluxing apparatus into the hood. Connect the gas (red) and oxygen (green) lines using the snap connects. Adjust the mirror as necessary so that you will be able to see down into the crucible.
4. Place the red drydown block immediately to the left of the fluxing apparatus.
5. Pre-label the clean, 180 mL Teflon beakers with green tape on their sides using the letters A-P. Arrange the beakers in the drydown block in the proper 4 by 4 matrix.
6. Fill each beaker with 125 mL of Milli-Q water using the large graduated cylinder. Cap each and place it back into the drydown block when finished.
7. Place the small CAT hotplate directly to the left of the drydown block and plug it in. Turn on the hotplate and set it to 95 degrees. Make sure the shaker is off and check to make sure the temperature is increasing.

8. Remove the dark, 16 hole, teflon-coated plate and the stainless plate from their storage bag and place them on the hotplate, stainless plate first. Make sure that you are wearing clean gloves and avoid touching the holes.
9. Bring out the set of tubes labeled A to P and put them in a rack, eight per side. Load each tube with a clean stir stick and blue scissor tong, making sure to avoid touching the working ends of both.

Sample Massing and Carrier Addition

(Usually done on Thursday morning)

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

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

- Beryllium carrier
- Spill tray
- 5 mL variable pipette, with new tip, on pipette rack
- 16 metal spatulas (clean, stored in bag)
- Waste beaker for dirty spatulas
- 15 samples in vials
- Meteoric blank material
- Balance with clean weigh boat

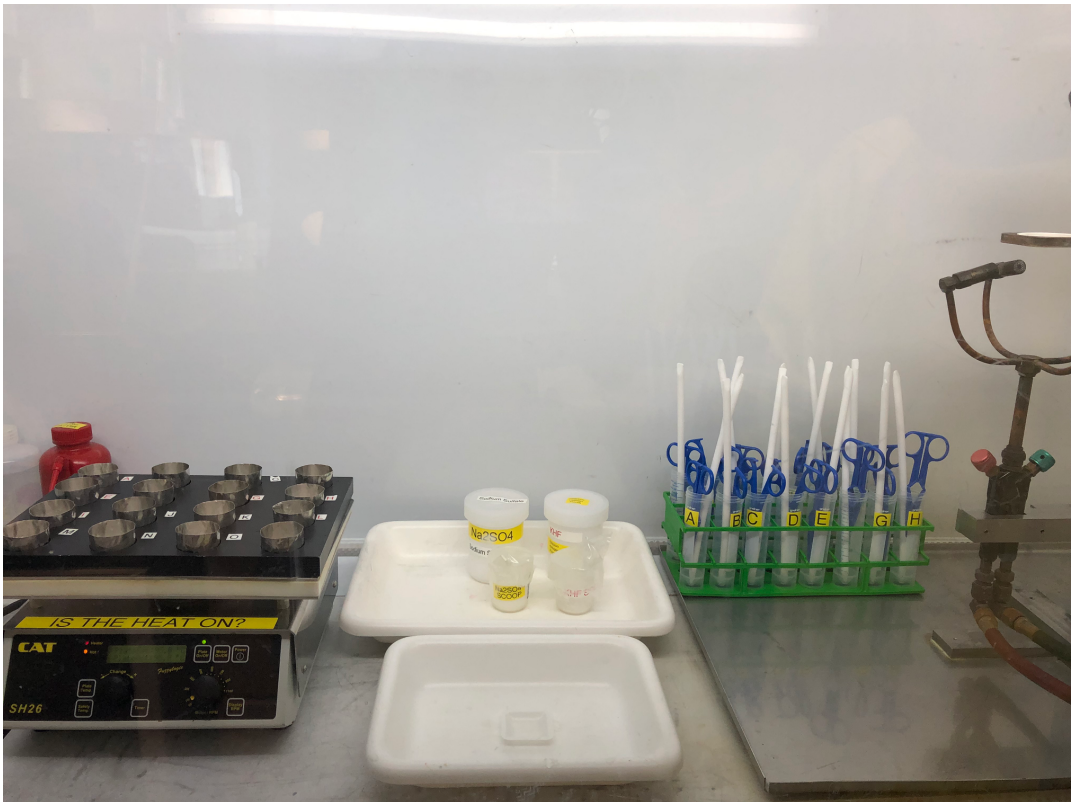
1. Using a set of blue plastic tongs, take out the 16 clean Pt crucibles and put them in the black rack on the CAT hotplate. Use only the blue tongs to transfer crucibles; they should never be touched because even their outer surfaces will be leached into your sample.
2. Open your data sheet on the computer.
3. Turn on the balance and place a new plastic weigh boat on the stage.
4. Starting at A, remove the crucible and place it in the weigh boat on the balance using its dedicated blue tongs. Place the tongs back into the A tube to ensure they stay clean.

5. Close the balance door, let the mass stabilize, and tare.
6. Open the two side doors of the balance. Into the crucible, add 0.5 g of the powdered sample using a clean stainless spatula. Make sure not to spill the powder; adding it in small increments will help.
7. When you have sufficient sample mass, put dirty spatula in waste beaker.
8. Close the balance doors and let the mass stabilize, then record the sample mass in the datasheet. Save the file after each entry.
9. Tare the balance again.
10. Open the top of the balance.
11. Using the 5 mL variable pipette, add the pre-determined amount of Be carrier to the crucible (if using UVM-SPEX carrier, you will likely be adding 1.35 mL, or approximately 400 μg of Be to each sample, but discuss this with Lee or Paul beforehand).
12. Close the top of the balance, let the balance stabilize, and record the mass in the data sheet. Save the data sheet.
13. Using the A blue tongs, carefully place the crucible back on the hotplate in the hood.
14. Repeat steps 4-13 for all samples until you are done.
15. When you do the blank, the procedure is the same. The blank is leached sediment from New Zealand ("WA-65").
16. When you are finished, cap the Be carrier tightly and store it in the Be bin under the hood.
17. Discard the pipette tip into the Be waste bag and place the pipette back into its storage box.
18. Clean the spatulas using ethyl alcohol and wipes. Do not acid wash the spatulas; the acid will corrode them. When clean, place them back in the storage bag.
19. Wash the pipette rack, spill tray, and waste beaker in DI, using the counter sink instead of the hood sink to avoid splashing the samples.
20. Print the data sheet and tape it in the lab batch book.
21. Leave the crucibles heating on the hotplate for approximately 2-3 hours or until completely dried down.

$NaSO_4$ and KHF_2 Addition

(Usually done on Thursday afternoon)

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)*

- CAT hotplate with samples on it
- Large Teflon container of $NaSO_4$
- Small beaker and scoop for $NaSO_4$
- Large Teflon container of KHF_2
- Small beaker and scoop for KHF_2
- Spill tray
- Clean weigh boat
- Rack of tubes holding stir sticks and blue tongs for each sample

1. Leave the hotplate on; keeping the samples warm throughout this process and until you flux will help to drive off any moisture.
2. Note that you will need to move the rack of beakers out of the hood in order to fit everything you need. This is fine; the beakers just contain water at this point.
3. Add 1 level scoop of anhydrous $NaSO_4$ to each crucible using the dedicated scoop.
4. Add 3 level scoops of anhydrous KHF_2 to each crucible using the dedicated scoop.

5. Put parafilm back over the beakers containing the reagent scoops and put them back under the hood, along with the larger reagent containers.
6. Starting with sample A, use its dedicated blue tongs to pick up the crucible and set it on the weigh boat. Continue holding the crucible (with the tongs) with your non-dominant hand throughout the mixing process.
7. Get the dedicated Teflon stir stick, being careful not to touch the working end. Always keep the working end facing upward so that it does not interact with the tube that is holding it.
8. With the sample's dedicated Teflon stir stick, carefully mix the contents into a uniform powder. Work very slowly and carefully so as not to spill, since the powder now contains beryllium; loss of any sample powder could bias your analytical result isotopically. Work gently so as not to deform or puncture the crucible. You want to form a completely homogeneous powder with no lumps.
9. Place the Teflon stir stick back into the corresponding storage tube, working end up so that it stays clean.
10. Using the blue tongs, transfer the crucible back into its hole on the hotplate. Then return the tongs to the corresponding storage tube.
11. Repeat steps 3-7 until all samples have been thoroughly mixed.
12. Use a moist wipe and DI water to clean up any spilled sample material. Rinse the wipe out well in the sink before discarding into Be waste.
13. Use 12 moist wipes to clean off the 12 working (i.e. upward-facing) ends of the stir sticks. Work one at a time so as not to confuse which is which, and place them back into the tubes with the working ends upward.

Sample Fluxing

(Usually done on Friday morning)

Personal Protective Equipment: Double-gloves (thin and thick), rubber smock, goggles, and face shield. Add leather chaps for the entire time the torch is running.

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

- CAT hotplate with homogenized, dried samples on it
- Rack of tubes holding stir sticks and blue tongs for each sample (note that you will not actually be using these, but they need to stay in the hood in order to remain clean)
- Rack of 16 beakers labeled A-P, filled with 125 mL Milli-Q water and capped
- Fluxing apparatus with ceramic ring to hold crucibles
- Black, heat-safe tile
- Pt-tipped tongs (working end suspended in the air)
- Metal spatula (working end suspended in the air)
- Lighter (not shown; keep it in the countertop outside the hood)
- Flashlight (not shown; keep it in the countertop outside the hood)
- Timer (not shown; keep it in the countertop outside the hood)

1. Before putting on your protective equipment, pull the shade down over the exterior window in the write-up room for better visibility of the torch.
2. Fully open the oxygen tank in the vestibule using the large knob at the top of the tank. Do not adjust the white valve regulator! Open only the stainless vertically-oriented valve.
3. Back in the lab, turn on the gas at the wall behind the sink by rotating the valve parallel to the gas line.
4. Turn off the hood blower.
5. Turn off the room light but leave the hood light on (for now).
6. Start with crucible A. Pick up the crucible using the Pt-tipped tongs and set it gently on the ceramic ring, trying not to push it down and to keep it level. Adjust the mirror so you can see right down into the crucible.
7. Set the corresponding A beaker in the spill tray.
8. Turn off the hood light.
9. Turn on the oxygen (the green line) one half turn.
10. Turn on the gas (the red line), increasing it gradually while trying to light the torch heads with the lighter. Your goal is to light the torch with as little gas flow as possible in order to keep the temperature low. Continue until all four torch heads are lit.
11. Pull the sash down completely, and keep it completely closed the entire time the torch is running.
12. Watch the fluxing with a flashlight (from outside the hood), keeping the flame as low as possible. The sample may bubble; if this occurs, try to decrease the torch strength as much as possible without letting the flame extinguish. Eventually, the crucible will start to glow orange; the sample will begin to melt, and become clear at the edges.
13. Once the sample has started to glow, turn up the oxygen and gas incrementally to get more heat. Turn off the flashlight and watch the flux. Continue until all bits of unfluxed material have vanished.
14. Increase the oxygen and gas one more time, getting the flame as hot as you can. Give the sample about 30 seconds at this high heat, until all bubbles have disappeared from the bottom and you can see the crucible bottom through the molten material.
15. Turn the torch off by turning the gas off first and the oxygen off second.
16. Start the timer for one minute. The crucible will cool and eventually the material will solidify. The melt will turn grey and crack, making audible sounds.
17. Turn the hood light back on.
18. When the timer goes off, open the sash.
19. Open the lid of the beaker. You are now about to quench the still-hot crucible in the water. Your primary goal is to do this safely, always keeping the crucible's opening facing the back wall of the hood.
20. Use the Pt–tipped tongs to lift the crucible off the stand, grasping it on its left side. You may need to use the metal spatula to delicately help the ceramic ring off the crucible (make sure it doesn't fall!).
21. Rotate your hand (and the tongs holding the crucible) 90° so that the place where the tongs grip the crucible is toward you. Then tilt the crucible so that the opening is toward the back of the hood. Lower the crucible into the beaker of water so that it fills with

water, but do not submerge the tongs (which will cause cross-contamination between samples).

22. Once the crucible has filled with water, gently release it from the tongs and allow it to drop down to the bottom of the beaker. It should be right-side up and completely submerged.
23. Cap the beaker and set it back in the rack.
24. If the Pt-tipped tongs touched the water, clean them with a wipe and Milli-Q water.
25. Repeat steps 2-24 for all samples. Note that when you do the blank, it will likely behave with more volatility than the samples; flux it as gently and slowly as possible.
26. When finished, turn off the gas and oxygen fully, both at the hood controls and at their primary controls (the wall valve and the tank valve, respectively).
27. Remove the teflon-coated crucible holder and stainless steel plate from the CAT hotplate. Wash both with 1% HNO_3 from the squirt bottle and then copious amounts of Milli-Q water. Dry them in the oven.
28. Wipe down the CAT hotplate with a few damp wipes, then put it back under the sink.
29. Using wipes and the 1% HNO_3 squirt bottle, clean the entire fluxing stand to remove any splatter. Then do the same wipe down with Milli-Q water from the squirt bottle to remove any nitric. Dispose of the HNO_3 wipes as Be waste.
30. Disconnect the oxygen and gas hoses, then put the fluxing apparatus back under the sink.
31. Turn the hood blower back on.
32. Remove the test tube rack holding the stirrers and tongs from the hood. Then, use the DI spray head to wash down the sides and deck of the hood thoroughly in order to wash away any spattered sample material. Take the green squeegee from its hanging place over the sink and use it to wipe down the hood and hood walls. Put the test tube rack back into the hood to keep the tools clean.
33. Place the hotplate in the hood and connect it. Make sure the hotplate is at least two inches away from the side and back walls of the hood.
34. Set the rack full of beakers on the hotplate. Turn on the hotplate at the controller and on the hood face and set it to 95°C.
35. On your way out, double-check that the gas and oxygen are off.
36. Leach the samples for at least overnight, and ideally several days (e.g. over the weekend). Only Be and K fluorides are water soluble; the leaching allows them to dissolve from the fusion cake and enter the solution.

Fusion Cake Removal

(Usually done on Monday morning)

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)*

- Bin filled with DI water for holding dirty crucibles
- Large wash jug for dirty dishes
- Rack of tubes holding stir sticks and blue tongs for each sample
- Spill tray
- Beakers sitting in rack on hot plate

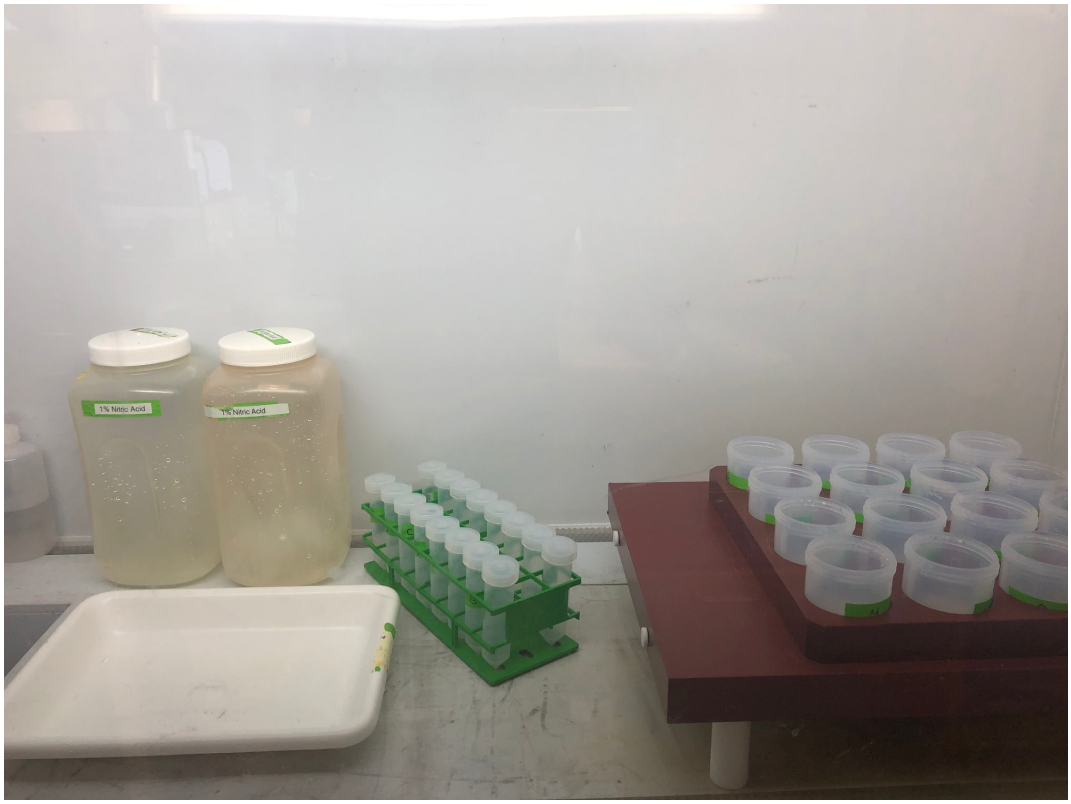
1. Turn off the hotplate and let samples cool for at least an hour until they are at room temperature (or, on a Monday, the hotplate will shut off automatically at 5:00AM).
2. Remove sample A from the hot plate and place it in the spill tray. Tighten its lid, then turn the beaker on an angle to remove the condensate from the lid.
3. Open the beaker and place the lid into the large wash jug.
4. Take the blue tongs for sample A and clamp them onto the side of the crucible, which is submerged in solution. Hold the tongs as you work.
5. Take the stir stick for sample A, remembering not to touch the working (upward) end.

6. Use the working end of the stir stick to very gently dislodge the fusion cake from the crucible and into the Teflon beaker. Work very gently to avoid splashing and/or damaging the crucibles. Tip the crucible to dump the cake scrapings into the beaker.
7. Holding the crucible with the tongs, look into it and gently revisit any pieces of the fusion cake that are still stuck to the crucible walls. When dislodged, tip those pieces into the beaker. Repeat as necessary.
8. When you are done and the crucible is completely cleaned out, place it in the dirty crucible wash bin that is filled with water. Place the stir stick and blue tongs into the wash jug.
9. Place the beaker for sample A back on the hotplate in the standard matrix order.
10. Repeat steps 2-9 until all 16 beakers have been done.
11. Fill the wash jug with 1% HNO_3 and place it in the sonicator (but do not start the sonicator yet, more dishes will be coming soon).
12. Rinse the crucibles several times in DI water, taking care not to splash into the open sample beakers.
13. Gently place the crucibles into the dedicated small wash jug for 10% NaOH. They must not stack, otherwise they won't get clean. Put a layer of seven on the bottom level, then one of the large white drilled watch glasses, seven more crucibles, the second watch glass, and two crucibles on the top. Ensure that all are facing upward and free to fill with solution. Carefully fill the wash jug with 10% NaOH (stored under the hood) and place it in the sonicator overnight.
14. Wash the rack of centrifuge tubes used for holding the stir sticks and tongs thoroughly with the DI spray, then place the rack and tubes on the bottom shelf of the oven (not in a bin, because it has not been acid washed) to dry.
15. Turn the hot plate on and set it to 165°C.
16. Take out the reference beaker marked with the target evaporation level. Your goal is to reduce the volume of the liquid to about 20-30 mL. Any less and precipitate will form; any more and you won't have sufficient capacity for the next steps.
17. The evaporation will take around three hours. You can ignore it for the first 1.5-2 hours; after that, keep a close eye on the samples and check them regularly against the reference beaker.
18. Once they are at the desired volume, turn off the hotplate and let it cool to room temperature.

Transferring to Centrifuge Tubes

(Usually done on Monday afternoon)

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)*

- Two large wash jugs for beakers
- Spill tray
- Rack of 16 teflon centrifuge tubes labeled A-P
- Beakers sitting in rack on hot plate

1. Make sure the samples have cooled completely before beginning this step!
2. Uncap the centrifuge tube for sample A.
3. Pick up beaker A and swirl gently to break up cake. Then pour the contents, cake and all, into the corresponding tube using one smooth motion. If needed, pour a bit of liquid back from the centrifuge tube and use that to resuspend any remaining material in the beaker, then pour back into the tube. At the end of this step, the beaker should be clean of all liquid and solids.
4. Cap up the tube and place it into the rack.
5. Place the beaker back into its slot on the hotplate (for now).
6. Repeat steps 2-5 for all 16 samples.

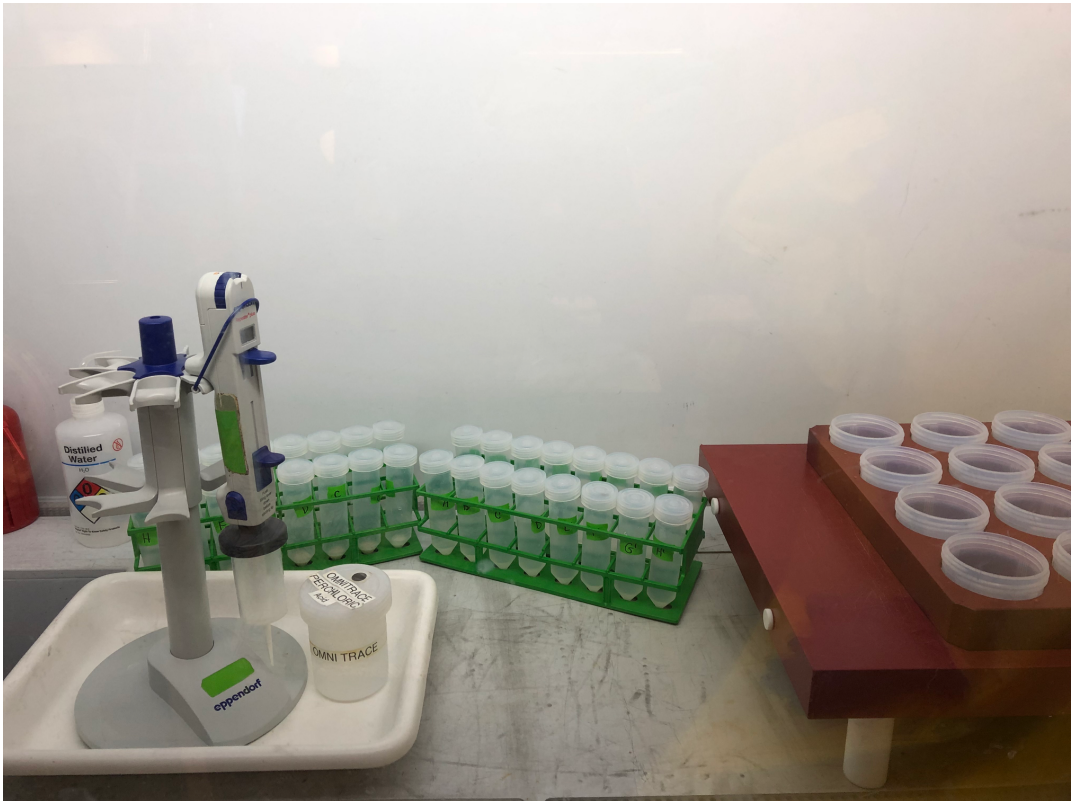
7. Your tubes may have slightly different amounts of liquid. Find the one with the largest volume, and determine what that volume is (e.g. 30 mL). Carefully use the Milli-Q squirt bottle to top all 16 tubes up to the same level so that the centrifuge balances.
8. You can leave the tubes capped and in the hood overnight, waiting until the following day to do the perchlorate precipitation.
9. Take the rack of beakers and put it in the sink. Spray the whole set-up (rack and beakers) with DI in order to remove traces of HF before washing.
10. Place the beakers into two wash jugs.
11. One of these wash jugs will not fit into the ultrasound tonight; it will need to wait until tomorrow night. For now, do not fill it with acid; just set it in the sink outside the hood.
12. Fill the other wash jug with 1% HNO_3 and add it to the sonicator.
13. Top off and start the sonicator.
14. Spray down the red beaker rack one more time, and place it on the cart to dry.

<u>Monday night dishes:</u>
Large jug full of beaker lids, stir sticks, and tongs
Small jug with crucibles in NaOH
Large jug (one of two) full of beakers

Perchlorate Precipitation

(Usually done on Tuesday)

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)*

- Repeater pipette, on rack, with dedicated 50mL tip
- Perchloric acid in spill tray
- Rack of 16 teflon centrifuge tubes labeled A-P containing your samples
- Rack of 16 teflon centrifuge tubes labeled A'-P'
- Hot plate with 16 clean 120mL beakers, unlabeled

1. Carefully load your 16 sample tubes into the centrifuge. Centrifuge at 3500 RPM for 5 minutes. Then remove them gently so as not to disturb the solids at the bottom, and bring them back into the hood.
2. Working one at a time, pour the liquid sample into the empty tube with the sample letter (i.e. A to A'). Leave the solid fusion cake remnants behind. Pay very careful attention to matching letters.
3. Set the rack of tubes with the fusion cake remnants toward the back of the hood or in the sink; you will deal with disposal later.
4. Place the rack of A'-P' samples in a large spill tray.

5. Uncap each of your sample tubes (A'-P'), laying the cap face-up next to its tube alongside the rack and in the spill tray.
6. Use the repeater pipette to deliver 5mL of $HClO_4$ to each tube. Do the first 6 tubes, then top off the reservoir, then do the remaining 10 to avoid having leftover acid at the end. A white precipitate will form as the acid goes into the tube.
7. Cap the tubes tightly and invert each several times to mix. Make sure the sash is far down for protection.
8. Let precipitate settle for 15 minutes.
9. Remove the caps and add an additional 3mL of $HCLO_4$ to each sample, as before. Pipette in slowly, since the solution level will be nearing the top of the tube. With this smaller volume, it will not be necessary to refill the reservoir.
10. Cap and shake the tubes as before.
11. Let the precipitate settle for another 15 minutes.
12. Carefully load your 16 sample tubes into the centrifuge. Centrifuge at 3500 RPM for 5 minutes. Then remove them gently so as not to disturb the solids at the bottom, and bring them back into the hood.
13. One at a time, bring one of the empty 120mL beakers into a spill tray and pour the solution from the centrifuge tube into the beaker, leaving all of the precipitate behind. Place the beaker back into its proper slot in the A-P matrix, double-checking to ensure you are on the correct sample.
14. You now have a few clean-up steps to do (see below). Do the clean-up before turning on the hot plate so that $HCLO_4$ is not fuming while you are working in the hood.

For this part of the procedure you will need:

- Dedicated waste container for fusion cake solids
- Dedicated waste container for $KClO_4$ precipitate
- Two large wash jugs for Teflon centrifuge tubes
- NaOH and HNO_3 jugs and corresponding liquid storage containers for crucible washing

1. Dispose of the fusion cake waste. To each tube (A-P) add a small amount of DI water, cap, shake, and pour the slurry into the dedicated waste container for fusion cake solids. Repeat as necessary to get all solids out of the tubes. Place the Teflon tubes and their lids into a wash jug as you finish each.
2. Dispose of the $KClO_4$ waste. To each tube (A'-P') add a small amount of DI water, cap, shake, and pour the slurry into the dedicated waste container for $KClO_4$ waste. Repeat as necessary to get all solids out of the tubes. Place the Teflon tubes and their lids into a second wash jug.
3. For both of the above processes, if the waste container becomes too full, you can decant the liquid into the sink; just make sure that all solids have settled and remain in the container.
4. Fill both wash jugs of centrifuge tubes and add them to the sonicator, after removing the dishes from the previous night.

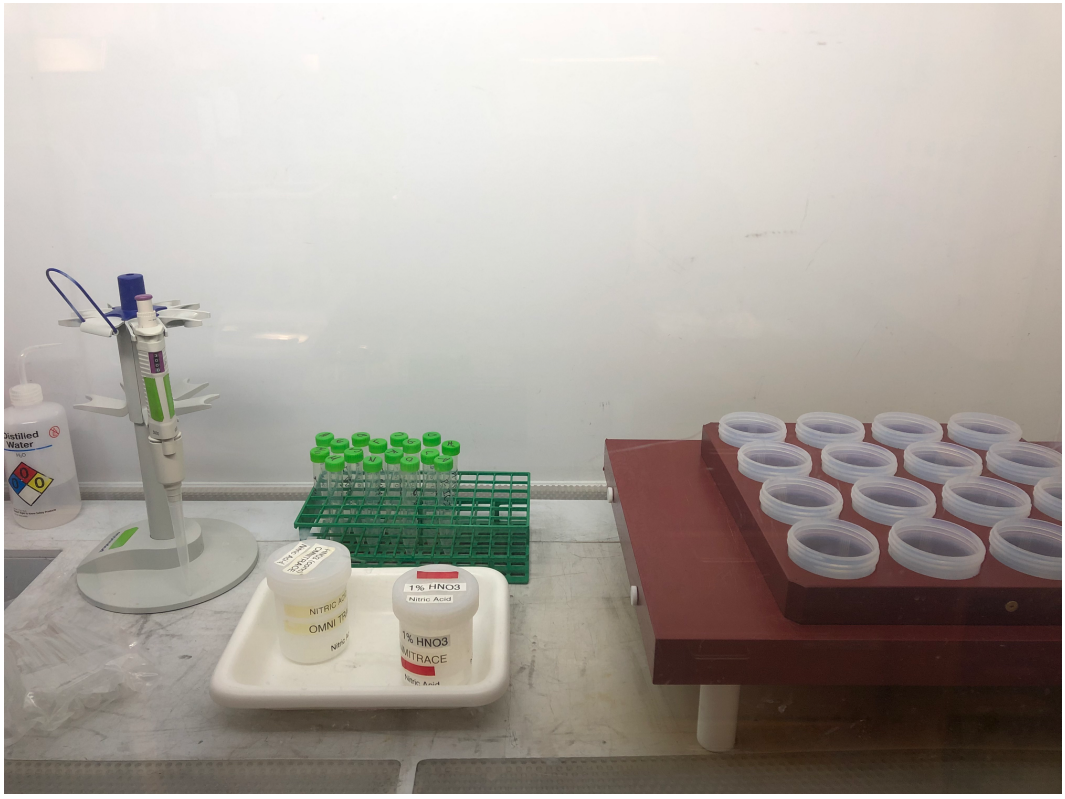
5. This is a good time to do your dishes from the day before: stir sticks, tongs, beaker lids, and half of the large beakers. Use the normal acid washing procedure.
6. Next, continue washing the crucibles. Remove the dedicated container of 10% NaOH and crucibles from the sonicator and bring it into the hood. Carefully pour the base back into the storage container from where it came, working slowly to avoid pouring out the crucibles.
7. When little to no base is left, pour the crucibles and the Teflon watch glasses into the dishwashing colander. Wash each with DI water, and use some damp wipes to encourage any bits of adhered fusion cake to come off the sides of the crucible. Wash thoroughly to remove all traces of base, since the crucibles will be going into acid next.
8. Repeat the crucible washing procedure, this time using 10% HNO_3. Place the crucibles into the dedicated crucible acid washing jug, making sure they are all upright and not nested. Then pour in 10% HNO_3 from the storage container under the hood.
9. Overnight, sonicate the two large wash jugs of Teflon tubes as well as the small jug with the crucibles.
10. Wipe down the deck of the hood using the green squeegee.
11. Wipe down the inside of the centrifuge and wash out the inner and outer baskets to ensure all traces of acid are gone.
12. Only once you are finished with clean-up, turn the hotplate to 230°C. The perchloric acid will evaporate off overnight.

<u>Tuesday night dishes</u>
Two large wash jugs full of Teflon centrifuge tubes
Small wash jug with crucibles in 10% HNO_3

Redissolution

(Usually done on Wednesday morning)

Personal Protective Equipment: Double-gloves (thin and thick), rubber smock, goggles, and face shield (because there is still perchloric condensate on the rims of the beakers)



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

- 5 mL variable pipette, on rack, with new tip
- Bag of disposable transfer pipettes (one per sample)
- Rack of 16 new, acid-washed, 15 mL centrifuge tubes labeled with the position letter (A-P), the batch number, and the sample name
- Container of trace-metal grade HNO_3 (i.e. NOT dish-washing acid!)
- Container of 1% HNO_3 (remix as needed)
- Spill tray
- Hotplate with 16 samples that have dried down overnight

1. Set the hotplate to 60°C and let it cool.
2. Meanwhile, mix more 1% HNO_3 if necessary. Make sure to use trace metal grade acid and not dishwashing acid, since this will be added to your samples.
3. Take your first sample (A) and set it in the spill tray. Then, using the variable pipette, add 12 mL of 1% HNO_3 (i.e. three shots of 4 mL).
4. Pick up the beaker and gently swirl the liquid until the cake of white material is fully dissolved; this may take a few minutes. Be careful to avoid touching the rim of the

beaker, since little beads of $HCLO_4$ condensate will remain there after the evaporation the previous night.

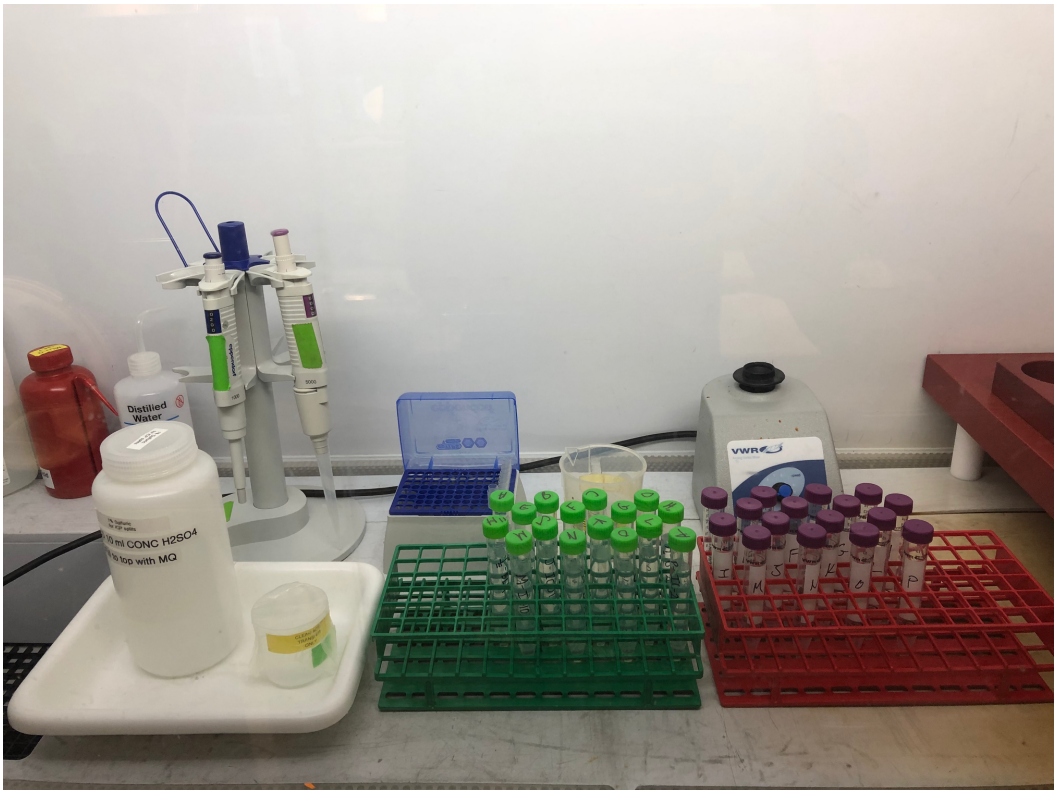
5. Once the material is back in solution, use a new transfer pipette to transfer the liquid into the corresponding green-capped entrifuge tube.
6. Repeat steps 3-5 for the remaining samples, until all tubes have been filled.
7. Turn the hot plate off.
8. Rinse all the used beakers in DI to get rid of the $HCLO_4$ around the rims. Then load them into two large wash jugs with 1% HNO_3. Sonicate overnight.
9. This is a good time to do dishes from the previous night; wash the Teflon centrifuge tubes using the standard procedure.
10. Continue washing the crucibles. Remove the dedicated container of 10% HNO_3 and crucibles from the sonicator and bring it into the hood. Carefully pour the acid back into the storage container from where it came, working slowly to avoid pouring out the crucibles.
11. Dump the crucibles into the colander. From now on, never touch them with your fingers. Instead, use a clean set of blue tongs.
12. Handling them only with tongs, triple-rinse each crucible in Milli-Q, then place it in the dedicated crucible drying bin in the oven.

<u>Wednesday night dishes</u>
One large wash jug full of 180 mL leaching beakers (leftover from Monday)
Two large wash jugs full of 120 mL perchloric evaporation beakers

Removing Yield Test Aliquots

(Usually done on Wednesday morning)

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



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

- 1mL pipette on rack (no tip yet)
- 5mL variable pipette, on pipette rack and with a new tip
- Spill tray
- Bottle of 1% H_2SO_4 yield test solution
- Clean acid beaker for holding yield test solution
- Box of tips for the 1 mL variable pipette
- Waste beaker for holding used tips
- Plastic rack with 16 samples in centrifuge tubes
- Vortex, with small wipe over the rubber foot
- Disposable rack with 16 empty, labeled (A-P), purple-capped centrifuge tubes

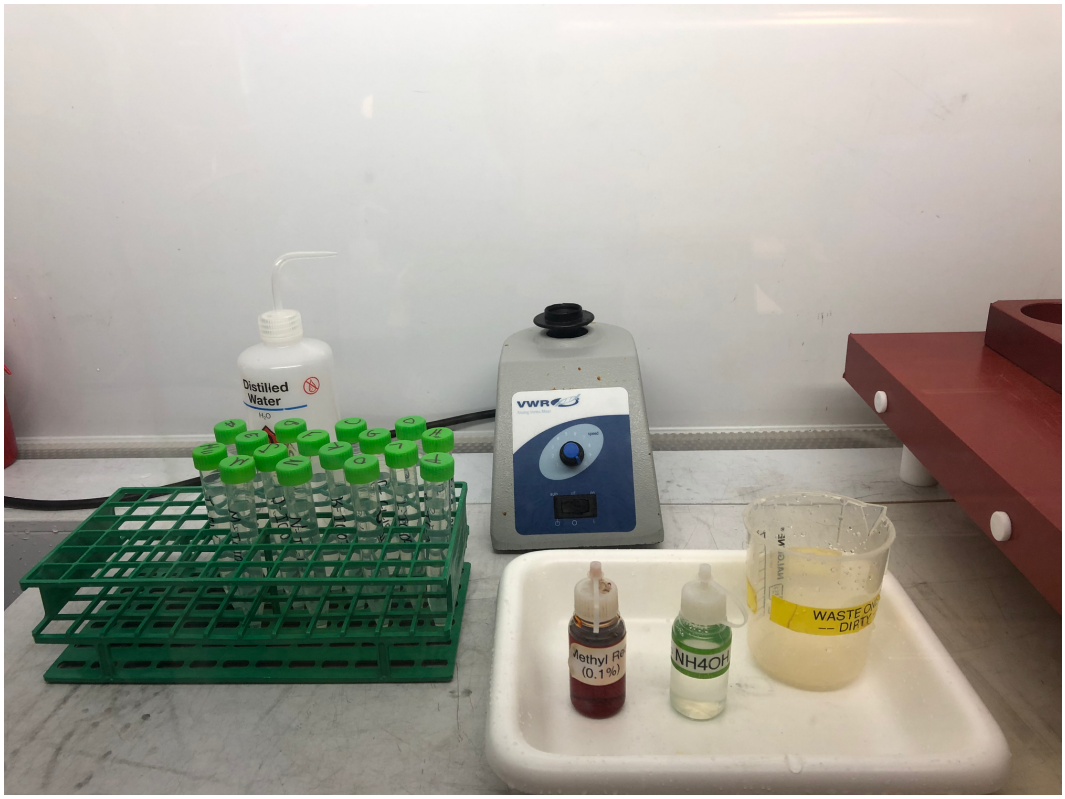
1. Carefully pour ~85 mL of yield test solution from the bottle and into the clean acid beaker.
2. In your hand or in a separate rack, hold the first green-capped sample tube (sample A) and the corresponding empty purple tube.
3. Place a new tip on the 1 mL pipette.

4. Without touching the pipette body to the tube (which could cause cross-contamination), remove a 200 μL aliquot from the sample 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 green (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 green and purple tubes in their respective racks, and bring the next set of tubes to the front of the hood.
10. Repeat for all samples.
11. Once you finish, place the used tips into the Be waste bag.
12. Put away all pipettes. Wash the spill tray and pipette racks in DI. Wash the clean acid beaker with Milli-Q and cover the top with parafilm.
13. Label the purple tubes “MBXXX Yields” with a piece of tape over the top of the rack.

Precipitating and Washing Hydroxide Jells

(Usually done on Wednesday afternoon)

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



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

- Milli-Q water squirt bottle
- Plastic rack with 16 samples in centrifuge tubes
- Vortex
- Small spill tray
- Methyl red dropper bottle
- 30% NH_4OH dropper bottle
- Waste beaker

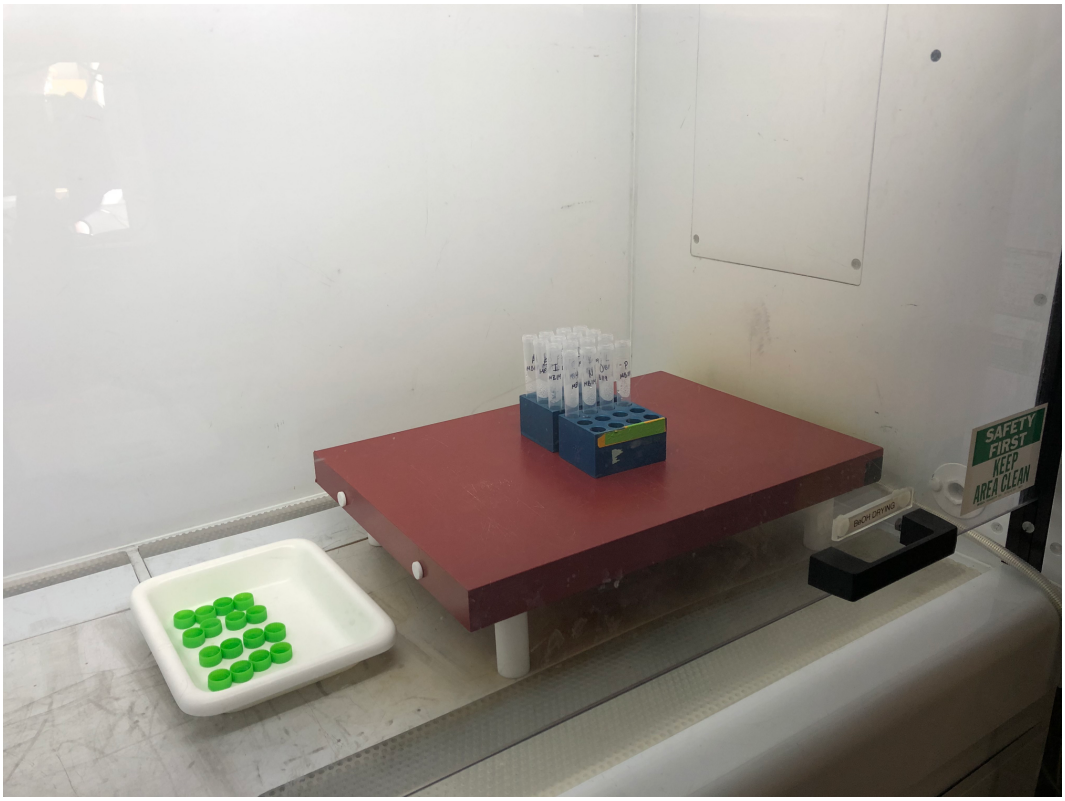
1. Get your first sample (A) and bring it toward the front of the hood, either holding it in your hand or standing it in another rack.
2. Add one drop of methyl red (a pH indicator). Try to avoid any spillage since it stains the hood deck and spill trays.
3. Using the 30% NH_4OH dropper bottle, add 3 drops to the sample tube. Cap, shake, and look for the color to change to green. If there is no color change, add one more drop and shake again. Keep adding drops one at a time until the color just changes from red to green. After the color change, add one more drop of 30% and shake a final time.

4. Repeat for each sample. They will likely take ~4-5 drops each, 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. After the pH titration, let the samples sit for at least an hour.
6. Put all 16 tubes in the centrifuge, making sure it is balanced. Centrifuge for 10 minutes at 3500 rpm. Unload them carefully back into the rack.
7. **STOP** – if you do not see a gel in the bottom of the tube, do not proceed! 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 that the solution pH is 8, adjusting with the HNO_3 and NH_4OH dropper bottles as needed. Remix and recentrifuge; you should now see the gel. If not, seek help from Paul or Lee.
8. From each sample tube, decant the liquid into a waste beaker, making sure the jell stays in the bottom of the tube.
9. To each tube, add 10 mL of mill-Q water, vortex for ten seconds, and check and see that the entire gel is dispersed. Move through this step efficiently since the water has a slightly lower pH than the solution from which you originally precipitated the gel.
10. Centrifuge again for 10 minutes at 3500 rpm.
11. As above, decant the liquid into a waste beaker, making sure to leave the gel in the bottom of the tube. Decant slowly but thoroughly so that no liquid is left in the tube.

Drying Hydroxide Jells into Pellets

(Usually done the following week, 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: *(shown from left to right)*

- Large hot plate
- Spill tray to hold caps
- Metal tube drydown blocks
- Beryllium hydroxide gels with liquid poured off

1. Do not dry your gels until you have analyzed the yield/purity aliquots on the ICP and have concluded that the samples are high-yield and pure.
2. Only do this process when the hood is otherwise unused to prevent contamination.
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). Put any used pipettes into the beryllium waste bag.
4. Once the water is gone, take off the cap of tube A and place it in the dry block in standard A-P order. Repeat for all other tubes and arrange the caps in a spill try in order, with their working sides facing upward.
5. Set the hotplate to 65°C and let them sit overnight.

6. **6.** The next day, increase the hotplate temperature to 98°C and leave the tubes for at least several hours; all day or overnight is better. 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.
7. Once samples are dry, cap them with the proper cap (double check that the letters match in order to avoid cross-contamination). Make sure the tubes are racked in order and label them with a piece of tape saying “Batch MBXXX, Dried Be Gels”.