# UVM Cosmogenic Laboratory – ***Meteoric $^{10}$Be Extraction***

*(Modified from Stone's method (Stone, 1998) by Reusser and Bierman in October and December, 2007 and January-June, 2009, late 2010, late 2011, late 2012)*

**Purpose:** This method details the means by which we extract meteoric $^{10}$Be, that adhered to grains and in grain coatings, using the flux fusion methods 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. We use cation columns to reduce boron levels in sediments.

**Hazards:** The primary hazards associated with this method are exposure to very high heat from the torch, crucible and molten flux 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. Strive at all times to keep beryllium in the beaker and handle all Be waste with care.

**Personal Protective Gear:** Gloves (double glove, light nitrile under heavier neoprene, when adding flux, fluxing, dealing with water leachate (contains HF), and using Perchloric acid), goggles, face shield (when fluxing and using perchloric), rubber gown (when fluxing and handling Perchloric acid), rubber lab shoes and tyvek sleeves to protect the area between gloves and lab smocks from exposure.

**Decontamination procedures before leaving the Lab:**
-Rinse outer Neoprene gloves with DI water and hang them in the hood on the clip
-Remove inner gloves and place in trash.
-Hang smock in corner and goggles on hooks before exiting the lab
-Wash hands thoroughly before leaving the vestibule.

**Safety Signage:** For the safety of people both inside and outside the lab, it is imperative that proper signage be used both on the hood itself (plexiglass labels on Velcro), on the lab door, and on the vestibule door. Please read and implement signage instructions provided in the detailed methods that follow.

# 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.*

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 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 only (i.e. no fingers). Clean all lab ware 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 tunes 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?*

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 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 lab ware 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.**

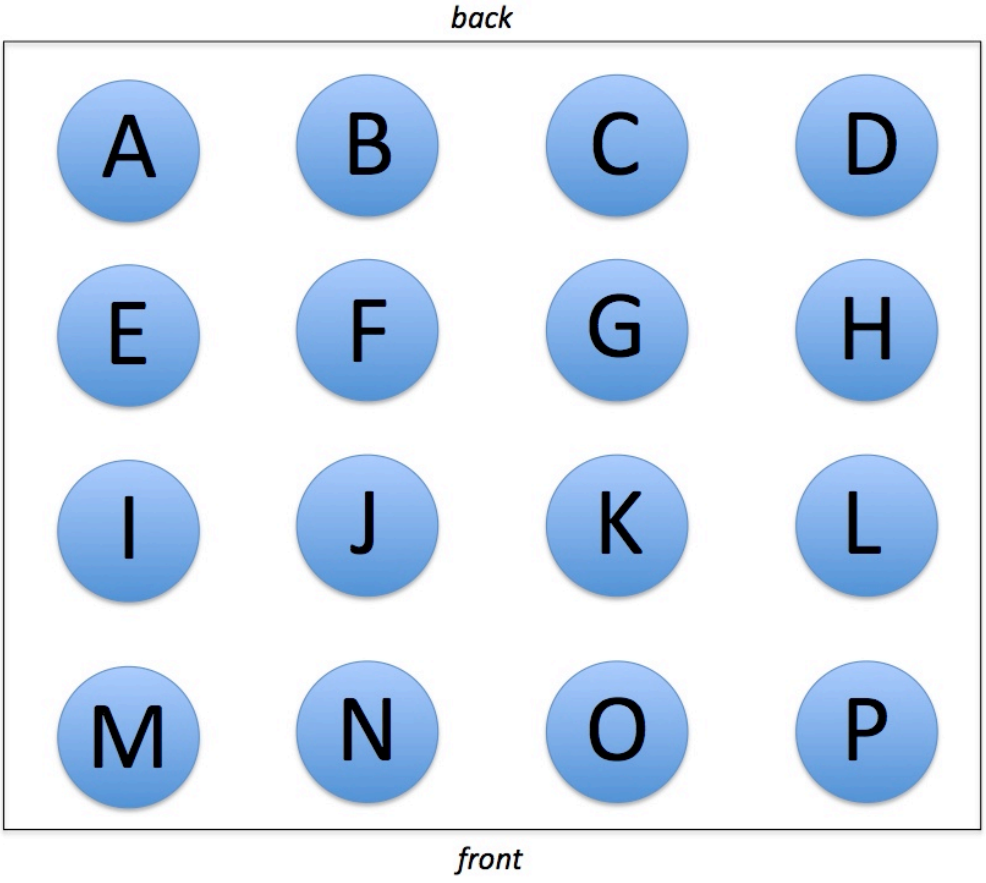
2. **Always store the pipette vertically; never lie the pipette on its side. Vertical storage keeps any acid on the pipette from seeping into the inner workings. We have installed pipette holders on the outside of the hoods.**

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. Failure to clean lab wear properly or spillage can contaminate your samples and the samples of everyone who follows you!
2. Sample identification is absolutely key. In order to prevent sample confusion, use the same racking order every time.
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. In addition, never touch crucibles with gloved or ungloved fingers. Always handle them with cleaned blue clips or Pt tipped tongs.




4. On the lab computer is an **ILLUSTRATED GUIDE** to performing this method. **BEFORE** doing the method, check it out!

## Getting ready

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 using the plastic case.

Samples must be fine powders or the method will not work. Use the SPEX shatter box to produce these powders (see lab safety manual for the rock room for specific instructions).

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.

We use low-level beryl carrier exclusively.

Make sure to initial each step on the batch sheet as it is completed.

If you need to interrupt the process at any point, make sure the beakers are covered or the samples are in closed tubes and the hotplate is off.

Check that there is pressure in the Oxygen tank in the Vestibule.

# *DAY 1. Load and Flux*

**Preparing your data sheet** **[10 minutes]**

*This is an important step since it records critical data for later calculations and arranges samples in an order that makes sense to you and is thus less likely to cause confusion later.*

1. Turn on the mouse.
2. Open the folder on the desktop entitled "Meteoric Batches".
3. Open file "Met_TEMPLATE".
4. Save the file (select type *.xlsx*) remembering to the change 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.
5. Fill in the information at the top of the sheet (name, date, and batch).
6. **IMPORTANT** – add the name of your carrier to the spreadsheet.
7. Organize your samples in logical order; usually alphabetical or low to high number in sequence.
8. Add all your samples ID's to the spreadsheet (in your logical order) beginning with the blank, which is called **BLK_MB#.** Look at the previous batch and move the blank one position higher than in the previous batch. It is important to rotate the blank between positions in case there is a bias in our process, such as in the column step.

**Preparing the lab** **[10 minutes]**

*This step sets up the hood for successful drying of the samples and carrier as well as safe and efficient fluxing of the samples.*

1. CHECK that there is pressure in the oxygen tank in the vestibule. Do not start a batch unless there is pressure in the tank.
2. Carefully remove the large red hotplate from the hood after first wiping it down with ecowipe and 1% HNO3 followed by MQ water (from the wash bottle) INSIDE the hood. Dispose of the wipes in the trash. Before removing the hotplate, you will need to disconnect it from the controller (at the back). Place the hotplate on the top of the cart. BE CAREFUL, the hotplate is very heavy and expensive ($3000).
3. Place the sink plug into the hood. Tape the nozzle to prevent DI water from dripping on your samples.
4. Place the CAT hotplate in the hood and plug it in. It is stored under the sink. CRITICAL – the sink plug can be unsteady unless the hotplate is placed correctly on it. To MAKE THE HOTPLATE STEADY, center the left feet of the CAT hotplate on the sink plug. Turn on the hotplate. Set to 95 degrees. Make sure the shaker is off and CHECK to make sure the hotplate is WARM.
5. Put on CLEAN, thin gloves. Remove the dark, 16 hole, teflon-coated plate and the stainless plate from their storage container and place them on the hotplate, stainless plate first. Make sure that you are wearing CLEAN gloves and avoid touching the holes. These items are stored in the bottom drawer.
6. Bring out the rack of tubes labeled A to P. Load each tube with a clean stir stick and blue scissor tong. Make sure labels are visible on each tube, i.e. facing outward on the rack.
7. Bring out the bag of stainless spatulas as well as a 500 ml WASTE beaker to hold them once they are dirty. Set the spatulas and beaker on the counter near the balance.
8. Move the fluxing apparatus into the hood. This is a two-person operation. Carefully remove, roll and save the plastic bag that covered the fluxing apparatus. Check that the gas and oxygen valves on the hood face are OFF. Connect the gas and oxygen lines using the snap connects.

**Loading your samples [1 hour]**

*This is a critical step where samples are massed and carrier is added. Take particular care to ensure the right sample ends up in the right crucible and that data are saved after every entry into the spread sheet.*

1. Make sure that you are wearing CLEAN gloves. Bring out 16 clean Pt crucibles and put them on the teflon-coated dry down plate that sits on the stainless plate that sits on the CAT hotplate. They are stored in the oven on the top shelf. From here on, use blue tongs to transfer crucibles; they cannot be touched from this point on.
2. Turn on the balance. Place a new plastic weigh boat on the balance and tare.
3. Bring out the Be carrier from below the hood. It's in a small savillex jar. Top up from the large (125 ml stock bottle). Set it in the hood in a small spill tray. Also bring out the liquid Be waste container (250 ml HDPE bottle with waste accumulation label) and set it in the hood on the same spill tray. We use only beryl carrier.
4. Use the 10 ml tip. Set the pipettor on the pipette carousel making sure the tip does not touch anything. Fill the reservoir with Be carrier trying to avoid air bubbles. Adjust the pipette so that the display reads and 800 ul (LOW LEVEL, depends on carrier concentration, aim for 300-350 ug of Be in each sample). BE CAREFUL, PIPETOR SOMETIMES READS THE WRONG VALUE – check the shot value on each tip and adjust if needed.
5. Shoot the first shot into the waste container being careful NOT to touch the tip to the side of the container. Set the pipettor on the pipette carousel making sure the tip does not touch anything. DO NOT LAY THE REPIPETTOR DOWN on its side; use the rack.
6. One at a time, starting at "A", remove a crucible and place it on the weigh boat on the balance using its dedicated blue scissor tong. CLOSE the balance. Let the mass stabilize. TARE the balance. Wait 10 seconds. The balance should read ZERO. If it does not, tare again.
7. The blank is leached sediment from New Zealand (WA-65) and is located in a 50ml tube in the bottom right had drawer labeled as meteoric blank.
8. Weigh in approximately ½ gram of each pulverized sample using a clean stainless spatula. Try very hard not to spill the powder. Close the balance doors and let the mass stabilize. Put dirty spatula in waste bin.
9. Record the sample mass and double check the sample name on the spreadsheet on the computer. SAVE THE FILE after each sample.
10. TARE the balance. Remove the pipette from the hood. Open the top of the balance. Pipette about 300 ug of carrier into the sample using the repipettor upper lever.
11. Record the carrier mass on the spreadsheet on the computer. DOUBLE check that the mass is associated with the right sample name on the spreadsheet on the computer. SAVE THE FILE after each sample.
12. Place the sample back on the hotplate in the hood.
13. Repeat the above steps (6 to 11) for all samples until you are done.

14. Double check that the temperature on the hotplate is set to 95$^{o}$C and that the *power* is on and that *plate* is on and WARM.
15. Leave the crucibles heating on the hotplate for approximately 2 hours to dry down.
16. CLEAN UP – shoot any excess carrier into the liquid Be waste container, expel the pipette tip to a Be waste bag, clean spatulas using a wipe to remove solids, wash with DI, spray with 1% Nitric, rinse with Milli-Q. DO NOT put spatulas in sonicators. It will destroy them.
17. Print the data sheet and tape it in the lab batch book. Check off, date and initial completed steps on work sheets.

**NaSO4 and KHF2 addition [~1 hour]**

*This step involves the addition of fluxing reagents to the samples and mixing. Thorough mixing is important for proper fluxing. Careful mixing and attention to detail (using the right stir sticks) prevents sample cross talk.*

1. Pre-label the clean, 180 ml Teflon beakers with green tape on the upper sides. Use the letters A-P and arrange the beakers on the counter in exactly the same 4 by 4 matrix as the samples. Rinse each with Milli-Q water and fill with ~120 ml of Milli-Q water. Cap and set back on the counter. Use the labeled comparison beaker stored in the bottom drawer to get the water height right.
2. SHUT OFF THE CAT HOTPLATE. Inside the hood set out a medium spill tray. Bring out the small savillex jars of NaSO4 and KHF2 from under the hood and set them in the spill tray. Bring out the reagent scoops in their parafilmed beakers also stored under the hood. If you need to refill the beakers, reagents are under the hood.
3. Place a clean tile in the tray (they are stored in the lower drawer in a plastic bag). Place a clean plastic weigh boat on the tile. Start with sample A. Use its dedicated blue tongs to pick up the crucible and set it on the weigh boat, which is on the tile. With the sample's dedicated Teflon stir stick, carefully crush the cake at the bottom of the crucible into a uniform powder. Work slowly so as not to spill since the powder now contains beryllium, a toxin. Loss of any sample powder could bias your analytical result.
4. For a 0.5 gram sample, add 1 level scoop of anhydrous $NaSO_4$ using the dedicated scoop. The mass of $NaSO_4$ should ~ equal the sample weight. Do this in the hood while the crucible in sitting on the weigh boat.
5. For a 0.5 gram sample, add 3 level scoops of anhydrous $KHF_2$ (~5 times the sample weight) using the large scoop. Do this in the hood while the crucible in sitting on the weigh boat.
6. Mix the contents of the crucible thoroughly using the dedicated teflon stir stick. Get rid of all lumps but do this GENTLY so as not to deform the crucibles or lose sample. Hold the side of the crucible with blue locking clamps while mixing.
7. Working from A-P, repeat steps 4, 5, and 6 until reagents have been added to all samples and all samples have been mixed. Then re-parafilm the reagent beakers and place them back under the hood. Place the scoops and small nalgene beakers back in their respective bags. Use a moist wipe and DI water to clean up any spilled reagent. Place the wipe off to the side of the hood to be rinsed out later.

**Sample fluxing** **[~2 hours]**

*This step melts the sample along with flux over a very hot natural gas flame that is augmented with oxygen. This is a high hazard step that requires absolute attention to detail and safety*

1. Don all the safety gear including the yellow smock, face shield, and leather chaps. Make sure to double glove.
2. Check that the gas valves on the hood face are OFF. Turn on the oxygen in the vestibule on FULL using the large knob at the top of the tank. Turn on the gas at the wall using the stopcock. Put out the spill tray for beakers with water, a tile in the front left corner of the fluxing device and set out Pt tipped tongs and a clean stainless spatula on the edge of the tile or fluxing stand so the tip is suspended in air. Put out the flashlight on the counter and a lighter in the hood.
3. Turn OFF the hood blower. Turn off the room light. (switches are on upper left of hood face).
4. Set a clean ceramic washer on the fluxing stand.
5. Start with crucible A. Pick up the crucible and set it gently on the ring trying not to push it down and to keep it level. Adjust the mirror so you can see right down into the crucible.
6. Bring over the proper beaker (the one labeled A) and set it in the spill tray and OPEN it, setting it upside down next to the beaker in the spill tray.
7. Turn OFF the hood light.
8. Have one person ready with a lighter (standing to the right) and another sitting in front of the gas controls. Turn on the oxygen one half turn. The sitting person slowly brings up the gas while the other person tries to light the burner. Continue until all four burners are lit on low.
9. PULL the sash ALL THE WAY DOWN.
10. Watch the fluxing with a flashlight keeping the flame as low as possible. The sample may bubble and the crucible will start to glow orange. Once the bubbling has stopped, the sample will begin to melt, and become clear at the edges. Turn up the gas to get more heat and turn off the flashlight and watch the flux. Flux for a minute or so after all black bits that swim around the sample have vanished.
11. Take out the timer and set it to 60 seconds. When the fluxing is done, turn off the GAS FIRST then the oxygen and START the timer. In 60 seconds, the crucible will cool so that the flux has solidified. The melt will turn grey and may crack (listen closely, you should hear it cracking). Turn ON the hood light. This is a very important safety step – you **MUST WAIT A MINUTE** or risk splattering hot, molten KHF when the crucible is dunked into the water.
12. After a minute of cooling, after the flux has solidified and the timer has sounded, open the sash and use the Pt–tipped tongs to lift the crucible off the stand. To make this work, grasp the left side of the crucible when removing it from the torch stand. The ring may come with it. If that's the case, use the spatula to gently tap the ring off the crucible. CRITICAL - Slowly place the crucible into the beaker filled with water. MAKE SURE CRUCIBLE IS FACING REAR OF THE HOOD WHEN DUNKED so that any splatter will be directed toward the back of the hood. It will hiss upon immersion.

Try not to put the tong tips in the water. Cap the beaker **TIGHTLY** and set it back on the counter.

13. If wet or dirty (because you have touched the water) wipe the Pt-tipped tongs well between each flux with a clean wipe and Milli-Q water (from the squirt bottle) to avoid cross-talk between samples.
14. Repeat steps 11-19 for all 16 samples.
15. When all samples are completed, remove the teflon-coated crucible holder from the CAT hotplate and set aside in the hood. Then remove the hotplate and place it back in its hiding space under the sink. Remove the sink plug and place it under the sink.
16. Take out several medium size wipes and the 1% nitric rinse bottle and wipe off 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 the wipes as Be waste in the waste bag.
17. Make sure both GAS VALVES ON THE HOOD ARE OFF. Disconnect the gas hoses. Cover the fluxing stand after it is cool with its plastic bag and move it back under the sink.
18. **Turn off the gas at the wall and turn off the oxygen at the tank. THIS IS A KEY SAFETY STEP.**
19. Remove the test tube rack from the hood. Turn on the DI water for the spray gun and remove the spray gun from its holder on the outside of the hood. Use the spray gun to wash down the sides and deck of the hood thoroughly in order to wash away any spattered Be or flux. Take the green squeegee from its hanging place over the sink and use it to wipe down the hood and hood walls. Rehang the squeegee.
20. Place the hotplate back in the hood and reconnect it. MAKE SURE HOTPLATE IS NOT TOUCHING ANYTHING INCLUDING HOOD WALL AND WASH BOTTLES. Place tube rack back in hood.
21. Check that the caps on the beakers are tight and set them on the hotplate. Turn on the hotplate at the controller and on the hood face. Set the hotplate to sub-boiling (95 C) and leave it on overnight. Only Be and K fluorides are water soluble. The overnight leaching allows them to dissolve from the fusion cake and enter the solution.
22. **Turn the hood blower back on before leaving lab.**
23. As you leave the lab, place the unattended operation sign for **Be in Water with HF** on both the hood face and the glass exterior wall of the vestibule.

# *Day 2. Extract and purify*

**Fusion Cake Removal [1.5 hr including clean up]**

*This step removes the fusion cake from the crucible by crushing the cake. The crushed cake will more effectively leach Be into the water surrounding the cake as it is heated to reduce the volume. Note that the water contains HF from the KHF flux and so should be treated with great care; it's hazardous!*

1. Turn off the hotplate and let samples cool for an hour or more until they are at room temperature. They contain HF so working when they are hot is not an option because of the reactivity of working with hot HF. Much of the Be in the fusion cake should have by now leached into the Milli-Q water in which it leached over night. Double glove for this step and wear full protective gear including face shield.
2. If you need more workspace in the hood, place the sink plug in.
3. Set out the labeled CRUCIBLE wash bin and fill 2/3 full with DI water and a washing jug to hold the spatulas and tongs.
4. Starting from the back (sample A), tighten its lid, turn the beaker on an angle to reduce the amount of condensate on the lid.
5. Take the stir stick dedicated to sample "A". Pull it out and use a wetted wipe to clean off the end that was used to stir the sample. Then, flip the stir stick upside down and use the clean end for the next step.
6. Open the beaker and use the blue tongs for that beaker to hold the crucible. Take the dedicated stir stick you have just cleaned and use it to dislodge and scrape the cake from the crucible into the teflon beaker. Do this working within the beaker and do it gently so as not to splash. Any water you splash is laced with beryllium and fluoride. MAKE SURE that the cake is broken up as finely as possible to increase yield. Tip the crucible to nudge the cake scrapings into the beaker filled with water. The idea is to completely remove the cake from the crucible and deposit it within the beaker without deforming the crucible.
7. When you are done, and the crucible is completely cleaned out, place it in the dirty crucible wash bin that is filled with water. The bin fits nicely in the sink. Place the stir stick, blue tongs, and beaker lids in a 4 liter cleaning jug.
8. Place the beaker for sample "A" back on the hotplate in the standard matrix order.
9. Repeat steps 3 to 8 until all 16 beakers have been done.
10. When the hotplate is full, turn it on and set to 165 degrees. The idea is to reduce the volume of the liquid to about 20 ml; try to be within a few mls of this volume. This dry off will take 2 to 3 hours. Keep checking on levels using the labeled beaker as a guide. When the largest sample has evaporated to about 20 ml, turn off the hotplate and let them cool at least an hour.
11. To clean the crucibles, rinse the crucibles several times with DI water being careful to minimize splash. Carefully place them upright in into 2-liter nalgene bottle LABLED FOR CRUCIBLES ONLY (NaOH) in layers starting with 7 crucibles on the bottom, a drilled teflon watch glass, 7 more crucibles, a drilled teflon watch glasses, and then two more. **USE THE Pt tongs to do this and you will get it right the first**

**time! The idea here is to prevent any crucible nesting.** Carefully fill the nalgene with 10% NaOH (stored under the hood and reused) and place it in heated ultrasound for the rest of the day or even better, over night. This step softens any remaining flux on the crucible walls.

12. Fill the wash jug (that contains lids, blue tongs, and stir sticks) with DI water, add 60 ml concentrated nitric acid, and set on the ultrasound to sonicate for the day or overnight.

**Perchlorate Precipitation [~3 hours]**

*This step uses Perchloric acid to precipitate any potassium still left in solution after the fluxing and water leaching step. This is an important sample purification step.*

1. Use a full wrap of green tape to label each of the 32 Teflon 50 ml centrifuge tubes. Label 16 tubes A-P and another 16 tubes A' – P'. Keep the caps on for now.
2. Use two green tube racks to line up the tubes so that both sets are in alphabetical order.
3. Put on yellow rubber smock, double glove, put on the sleeves, and put on the face shield.
4. Put spill tray into hood with Milli-Q squirt bottle inside it. Pick up beaker A and swirl gently to break up cake. Pour the contents, cake and all, into the tube labeled 'A' using one smooth motion. If needed, pour a bit of liquid back from the centrifuge tube and use that to police up any remaining material in the beaker. At the end of this step, the beaker should be clean of all liquid and solids.
5. Repeat for all 16 samples.
6. Make sure all the tubes contain between 35 and 40 ml of liquid. If they contain less, make a note to that effect on the batch sheet and add DI water until there is about 40 ml of liquid in the tube.
7. Centrifuge half the tubes at a time at 2500 RPM for 5 minutes. Gently remove the tubes from the centrifuge so as not to disturb the solids at the bottom of the tube.
8. While the samples are spinning, in the hood, rinse each Teflon beaker with DI water and place in a pair of 4 liter wash jugs for later cleaning.
9. Inside hood, decant the supernatant from centrifuged tubes into the new tubes of the same letter but bearing the apostrophe. Do this in such a way that you can see the cake and stop decanting if the cake begins to move out of the tube. Decant all 16 tubes in sequence starting with A and paying careful attention to matching letters. You may need to leave a little bit of liquid behind. That's OK – better to leave some liquid than to carry over solids to your sample.
10. Bring out the labeled Perchloric acid 240 ml container and use the large Perchloric bottle to bring the 240 ml container to the fill line (about 170 ml). Do this very carefully and in a spill tray. **Immediately close the lids on both the jar and bottle before doing anything else!**
11. Bring out the repipettor and the 50 ml reservoir used for Perchloric acid. Place the container on the square spill tray as well as a clean acid beaker with MQ water for rinsing pipette tip before disposal.
12. Place the carousel in the hood to hold the pipettor.
13. Find another spill tray and place the green rack in it. Uncap one tube at a time, laying the cap next to it, top down on the spill tray.
14. Fill the repipettor and deliver 5 ml of perchloric acid to the sample in its test tube to precipitate $KClO_4$. Be careful of drips. Drips indicate an overfull reservoir or a reservoir that needs replacement. A white precipitate will form as the acid goes into the tube. Cap tube tightly, invert several times, and shake thoroughly, invert again and shake one more time. Repeat for all samples. Make sure the sash is WAY down for protection.

15. Let precipitate settle for 20 minutes. Then remove caps and add three more ml of perchloric acid VERY SLOWLY to each sample watching carefully to see if more precipitate forms. If more precipitate forms, add another ml. Cap tubes tightly, invert several times, and shake thoroughly, invert again and shake one more time. Let precipitate settle for another 20 minutes. CAUTION – these TUBES ARE VERY FULL. Be careful.
16. Centrifuge in two batches at 2500 RPM for 5 minutes.
17. While the samples are spinning, rinse the KHF fusion cakes (in the first set of tubes) into their proper waste container. DI rinse the tubes and sonicate in 1% Nitric overnight. It is OK to drain the liquid from the waste container into the sink and retain the solids.
18. Replace the tubes in order into a pair of green racks in the hood.
19. Take out the box containing 16 of the 120 ml beakers. There should be no lids and the beakers should not be labeled.
20. **This is a critical step** – be careful as you are pouring Perchloric acid into unlabeled beakers. Inside the hood, decant supernatant into Teflon beakers starting with A and continuing to P. Use the standard matrix to keep track of what's where. It is better to leave a little liquid in the tubes that to pour solids into your sample.
21. Use the repipettor to ADD 5 ml of conc OMNITRACE Nitric Acid (HNO3) to each beaker and place on the hotplate, uncovered, in the beaker rack. THIS ACID ADDITION IS IMPORTANT for reducing boron! Note well; this is NOT the washing acid but the high purity acid stored in the teflon container under the hood.
22. Inside the hood, there is clean up to do. Rinse the $KClO_4$ waste from tubes used in the previous step into the labeled waste container. DI rinse the tubes and sonicate in 1% Nitric overnight. It is OK to drain the liquid from the waste container into the sink and retain the solids.
23. Small amounts of Perchloric acid are sometimes present of the test tubes and have damaged the centrifuge. To prevent further damage, after each batch, remove the tube holders and baskets from the centrifuge and rinse them all very well with DI water. Wipe down the inside of the centrifuge with a DI saturated wipe and then dry with a second wipe.
24. Set hotplate to 230 C. Allow samples to dry down to a white cake overnight. Put the *Perchloric acid evaporating* unattended operation label on the hood and on the glass wall of the vestibule.
25. Stay out of the lab while the Perchloric acid is fuming since walking back and forth in front of the hood will cause turbulence that could draw fumes into the room.

## *Day 3: Recover and Precipitate BeOH*

**Be Recovery** **[2 hours]**

*This step gets the samples back into solution from which BeOH can be precipitated leaving various soluble cations* **in solution.**

1. Set the hotplate to 60C and let it cool until the hotplate temp is 60C, about an hour.
2. Inside the hood, use the repipettor to add 20 ml of 1% OMNITRACE HNO3 to each beaker (NOT THE WASHING NITRIC!). The 1% OMNITRACE HNO3 is found under the hood. Wait until cakes are dissolved (usually within about 15 minutes). **USE clean 1% ACID only, NOT concentrated acid.** TURN HOTPLATE OFF.
3. Label 16 of the ACID-CLEANED disposable 50 ml tubes from A-P PLUS the BATCH NUMBER on the side of the tube and place in rack. Set out a spill tray into the hood.
4. Pour the sample solution from each beaker into the corresponding tube, rinsing the beaker once with about a ml DI. Final volume should be 20-25 ml in each 50 ml tube. Try to pour over the side of the rim where there are not many or even any Perchloric acid droplets.
5. Add two drops of methyl red to each tube from the dropper bottle.
6. Start with the first sample. Slowly, titrate drops of 30% NH4OH into the tube mixing as you go until solution just turns from red to yellow. Start with 10 and then go two by two. When you get close to the color change, you can switch to 15% NH4OH. The color change is sudden. Once the color has turned yellow, add 2 drops of 15% to ensure the sample is at or above pH 8 since the indicator changes at about pH 6.
7. Let the tubes sit for AT LEAST AN HOUR, overnight to several days is best; a very small amount of precipitate ($Be(OH)_3$) will form. It's almost impossible to see.
8. Centrifuge at 2500 rpm for 10 minutes. There should be a small amount of $Be(OH)_2$ gel at the bottom of each tube.
9. Carefully, very carefully decant the supernate into a waste beaker. If centrifuged well enough and handled gently, the gels will stick in the bottom of the tube.
10. Add 10 ml of MILLI-Q water and re-suspend the gel using the vortexer. This step removes additional K and Na which are water soluble and improves column performance.
11. Centrifuge at 2500 for 10 minutes. There should be a small amount of clear $Be(OH)_2$ gel at the bottom of each tube. *Carefully decant liquid making sure not to lose the gel.* Store samples under the hood in containment in anticipation of cation columns.

**Cation Columns [2.5 hours]**

*The use of cation columns is critical for Boron removal and to improve overall sample purity and thus AMS beam currents.*

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: Make sure you have enough of all elution solutions to do you WHOLE BATCH before you start. Discard any solution remaining in wash bottles from previous columation. Dump down the hood sink with running water. Solution concentration is absolutely KEY – do dilutions accurately or risk losing your samples.

1. Take out, rack up, and label cap and tube (using marker with sample position, batch number, and sample name) 16, 15-ml ACID WASHED, GREEN CAPPED tubes, one for each sample. DO NOT USE TAPE or the tubes will not insert in the rack. These tubes will be the tubes to which your samples are eluted.
2. Take out, rack up, and label (with sample position and batch number) 16, 15-ml TEFLON tubes, one for each sample. These tubes will be the tubes into which the boron fraction of your samples will be eluted. USE tape to label these teflon tube – but make sure that you use a small piece of tape and wrap it at the top of the tube just below the threads.
3. Remake acid for column cleaning, column conditioning, and sample elution each time. The acid is stored in Teflon 500 ml squirt bottles, which are labeled and include recipes for the diluted acids. There are two of these squirt bottles, one for 2 N HCl and the other for 0.3 N HCl. ACID VOLUMES ARE CRITICAL FOR PROPER COLUMN PERFORMANCE.
4. **MIXING ACID** For 2 N, use 80 ml of conc (Aristar) HCl and 420 ml of MQ water. To do this, add a few cm of water to the bottle, then use a small graduated cylinder to measure the acid, add to the squirt bottle, and then top up the squirt bottle to 500 ml which is the bottom of the yellow band (see mark on bottle).
5. **MIXING ACID** For 0.3 N, use 12.5 ml of conc (Aristar) HCl and 487.5 mls of MQ water. To do this, add a few cm of water to the bottle, then use a the 5 ml variable pipette to measure acid from the teflon container, add to the squirt bottle, and then top up the squirt bottle to 500 ml which is the bottom of the yellow band (see mark on bottle).
6. The column racks are stored on the bottom shelf of the cart. There are three parts to the racks: the column rack, holders for 50 ml waste tubes, and holders for 15 ml sample tubes.
7. PUT THE SINK PLUG IN. Move all rack parts into the hood.
8. There is a white board above the hood. Use it to indicate exactly what steps in the following process have been completed. It's a great way to make sure all the steps are done and done in the right order.
9. **UNCAPPING Columns**. Place the 50 ml rack and its waste tubes under the columns. Pull top caps from columns, then pull bottom caps. Put the top and bottom caps into their designated clean plastic bag.

10. Remove the reservoirs from their bag and place the lettered reservoir on its column.
11. Let the MQ water in the columns drain to waste (into the 50ml waste tubes)
12. **CLEANING COLUMNS** Add 6 column volumes (which is 9 ml) of 2 N HCl to each column. Add this acid with the squirt bottle up to the clear line on the reservoir which is labeled 6 CV. Let acid drain to waste in the 50 ml waste tubes.
13. **CONDITION COLUMNS** Add 6 column volumes (which is 9 ml) of 0.3 N HCl to each column. Add this acid with the squirt bottle up to the clear line on the reservoir which is labeled 6 CV. Let acid drain to waste in the 50 ml waste tubes.
14. **REDISOLVE** Samples. Pull out the rack of 50 ml tubes holding the sample jells. One at a time, open a tube and add 2 ml of 0.3 N HCl using the repipettor and a designated tip. Cap and repeat for all tubes. Use the disposable pipette sucking up and gently releasing the solution to ensure complete dissolution while minimizing spatter on the sides of the tubes.
15. **LOADING** Samples. Use the dedicated disposable pipette that is now in the 50 ml sample tube. The pipette that was used to suck up the sample and expel it back into the tube several times to ensure complete dissolution of the jell. Now, suck up the sample and add the sample gently to the frit of its respective column. Repeat for all columns. Let all samples DRAIN FULLY to the waste tubes.
16. **Remove the rack with the waste tubes** and replace with the rack containing the boron fraction, teflon, 15 ml tubes. Make sure the right tubes are in the right slots.
17. **BORON FRACTION** To elute the boron, add 6 column volumes (which is 9 ml) of 0.3 N HCl to each column. Add this acid with the squirt bottle up to the clear line on the reservoir which is labeled 6 CV. Let acid drain to labeled 15 ml acid washed teflon tubes. Save these tubes until the yield test is complete and then dispose of the fluid and wash the tube overnight in 1% Nitric.
18. **After the boron fraction has eluted** (the columns have drained), swap tubes, capping and racking the tubes containing the boron fraction and replacing them with the acid washed, green sample tubes. DOUBLE CHECK that the right tube is in the right location on the rack.
19. **ADD ELUTION ACID**. Add 6 column volumes (which is 9 ml) of 2 N HCl to each column. Add this acid with the squirt bottle up to the clear line on the reservoir which is labeled 6 CV. Let acid drain completely into the 15 ml, acid washed, green tubes.
20. **REMOVE SAMPLE RACK** and cap tubes containing 9 ml of 2N acid in which your PRECIOUS SAMPLES are dissolved. Cap the tubes with their lettered caps. Set these aside.
21. **CLEANING COLUMNS**. Replace the waste tube rack. Add 6 column volumes (which is 9 ml) of 2 N HCl to each column. Add this acid with the squirt bottle up to the clear line on the reservoir which is labeled 6 CV. Let acid drain to waste.
22. Empty the waste tubes into the sink.
23. **WASHING COLUMNS** Fill reservoirs with MQ water from the squirt bottle and let drain to waste.
24. **Place bottom caps on the columns,** fill the columns to the knurled section with MilliQ water, and then place the top caps on the columns.
25. Remove the columns from the rack and store them in their small foam rack on the shelf.

26. Remove the waste tube rack and empty each tube into the sink with flowing water after removing the sink plug. Rinse the tubes WELL with 1% nitric and then with MQ water several times inside and out and set in the oven to dry.
27. Once the yield tests are completed (and the Be is accounted for), drain the boron fraction tubes to waste and wash them by sonicating in a wash jug overnight in 1% Nitric Acid.

## ICP Splits for Be yield and purity test

*This is a critical step that ensures the process has worked correctly and makes sure that any targets we send to the accelerator have sufficient Be in them to produce good currents.*

1. Take out the 1 ml variable pipettor and a box of 1000 ul tips. Take out 2 green racks for 15 ml tubes.
2. Take out and rack up and label (with the matrix letter) 16, 15-ml tubes, one for each sample. These should be purple not acid-washed tubes.
3. **USE A NEW TIP FOR EACH SAMPLE.**
4. Set the 1 ml variable pipettor to 0.200 ml. Open green tube labeled A. Take an aliquot from the first sample (A) and place it in the appropriate purple cap tube (also A). This a step for which MENTAL CONCENTRATION is key to avoid cross-contamination. OPEN ONLY ONE tube a time. DISCARD the tip to Be waste. Cap the green tube. Move the sample that has been aliquoted to the other green rack.
5. Once there is solution in the purple tube, use the repipettor to add 5 ml of weak (1% H2SO4) to the purple tube. CAP the purple tube.
6. Repeat for all samples.
7. VORTEX WELL all the purple tubes.
8. Once yields are run (and the Be is accounted for) and the samples are declared, CLEAN, then dump the “boron fraction” and proceed.

**Final Precip and Wash**

*This step further cleans the Be jell by rejecting elements that will not precipitate. Washing removes soluble elements including Cl that interfere with AMS measurement.*

1. WAIT FOR YEILD TEST results before doing this step.
2. Add one drop of methyl red to each sample tube from the dropper bottle.
3. Start with the first sample. Slowly, titrate drops of 30% NH4OH into the tube mixing as you go until solution just turns from red to yellow. Start with 15 drops and then go two by two. When you get close switch to 15% NH4OH. The color change is sudden, so try no to over add the base. Once the color has turned yellow, add 2 drops to ensure that the pH is close to 8.
4. Let the tubes sit for AT LEAST an hour – better to let them sit over night or for a few days; a very small amount of precipitate ($Be(OH)_3$) will form. It's almost impossible to see.
5. Centrifuge at 3000 or greater for 10 minutes. There should be a small but clearly visible amount of $Be(OH)_2$ gel at the bottom of each tube.
6. Carefully decant the supernate into a waste beaker. If centrifuged well, the jell will stick in the bottom of the tube.
7. Add 10 ml of MILLI-Q and re-suspend the gel using the VORTEXER. VORTEXING is a CRITICAL STEP. If missed, samples won't be washed.
8. Centrifuge again, the Be gel will reappear at the bottom
9. CAREFULLY Pour off DI into waste beaker and cap tube.
10. Repeat for all. When done, pour the water in the waste beaker down the sink and wash the beaker with 1% Nitric Acid.
11. The samples now need to be brought up (redissolved) in 5 ml 1% OMNITRACE HNO3. This is CRITICAL STEP as the Cl (in the HCl) persists even after washing and interferes with AMS measurements. Dispense using the repipettor into each tube and then VORTEX to ensure the jell dissolves fully.
12. Precipitate and spin each sample as above (steps 3-5).
13. Wash each sample as above (steps 7-9). The final jell should be small and white/grey and perhaps 2 or 3 mm tall in the tip of the tube. Store in water.

**Drying gels**

*This step prepares the samples for packing into cathodes.*

1. Do this when nothing else is going on in the hood.
2. If there is any water above the jell in the tubes, use a disposable pipette (one for each sample) to remove all the free the water. This ensures that the material will dry into a nice pellet.
3. Place each sample in the dry down block on big hotplate. Take off the caps (check to make sure they are labeled by letter) and arrange in a spill tray, top down.
4. Set the hotplate to 65C until the jells are dry – usually over night. This is key, slow cool drying gives great pellets.
5. Once the gel is dry, increase heat to 98C and leave for at least several hours.
6. Use the mirror to make sure there is NO condensate remaining on the tube walls.
7. When dry, cool, recap using the correct caps, and set aside in a rack for packing.
8. Label the rack with the batch number and store under the hood.

# *Cleaning Procedures:*

*Platinum Crucibles (re-use) – Done following cake scrapping on the 2nd day:*

1. Individually rinse the 16 crucibles with Mill-Q and makes sure that all remaining fusion material is removed. If needed, use wipe to clean the crucible. If a crucible is bent, try to gently reshape it into round.
2. Gently place the crucibles upright in the labeled 2 liter NaOH-wash bottle. Seven crucibles will fit in one layer. Place a watch glass (with drilled holes) on top of the first layer and add the next 7 crucibles followed by another watch glass. Place the remaining 2 on top. Gently fill the bottle with the >10% NaOH mix that is stored under the hood. Place on the heated ultrasound for at least 6 hours – more is OK if there is not another batch starting.
3. Remove the 2-liter jar with crucibles from the ultrasound. Decant NaOH into the storage bottle. Rinse the crucibles individually and check again that they are clean of all flux material. Wipe and scrape more if needed. Place them in clean 2-liter Nalgene filled with 10% Nitric (which is also stored under the hood) – using the same technique as before to make sure the crucibles don't nest. Place bottle in the ultrasound and sonicate at least overnight.
4. Remove the jar from the ultrasound, and pour the 10% Nitric Acid back into its storage bottle and return to under the hood. Rinse each crucible several times with Milli-Q water and place inverted in the labeled crucible drying tray. Place the tray on the top shelf of the drying oven.
5. When everything is dry and if the crucibles are not being used, lid the drying tray and leave on the top shelf of the oven.

*Teflon Beakers and Lids (re-use) – Done following the 2nd day water evaporation:*

1. After use, rinse the beakers and lids into the sink with DI water and make sure they are clean of any and all contaminants and solids.
2. Place beakers and lids in a 4 liter jug sitting up or sideways (not inverted) and fill with DI water. Add 60 ml of conc Nitric acid and cap in the hood. Place in the ultrasound overnight and sonicate.
3. Remove the jug from the ultrasound, open in the hood and drain into a colander placed in the hood sink. Rinse each beaker several times with Milli-Q water and place upright on a drying tray in the oven or use wet (but the beakers MUST BE ACID FREE or else the water extraction won't work, you will get Fe and Al in the samples as these can be leached).

*Teflon Stir Sticks and Blue Tongs (re-use) – Done after cake scrapping on 2nd day:*

1. Place stir sticks and blue tongs in a 4 liter jug and fill with DI water. Add 60 ml of conc. Nitric acid and cap in the hood. Place in the ultrasound overnight and sonicate.

2. Remove the jug from the ultrasound, open in the hood and drain into a colander placed in the hood sink. Rinse each stir stick and set of blue tong several times with Milli-Q water and place in a drying tray in the oven.
3. When the items are dry, return them to their storage vessels in the drawer.

*Teflon 50 ml tubes (re-use) – Done following the Perchloric Precipitation on 2nd Day:*

1. After removing the fusion cakes and KClO4, rinse the tubes into the sink to make sure all remaining material is removed. Fill tubes with DI water and place them vertically in a 4 liter jug half way filled with DI water. When full, top off with DI water. Add the lids. Add 60 ml of conc. Nitric acid and cap tightly in the hood. Shake well to make sure acid is well mixed. Place in the ultrasound overnight and sonicate.
2. Remove the jug from the ultrasound, open in the hood and drain into a colander placed in the hood sink. Rinse each tube individually, inside and out, several times with Milli-Q water and place in racks in the oven to dry. Place the caps in a tray to dry.
3. When the tubes are dry, cap them return them to their storage box.

*Plastic 50 and 15 ml tubes (use once and dispose as Be Waste)*

1. Find and open several bags of green-topped tubes. Rinse and then fill each tube with DI water and drop them vertically in a 4 liter jug that is DEDICATED FOR NEW TUBE CLEANING. Do not force more tubes into the jug than will easily fit. Fill with DI water. Add the lids. Add 60 ml of conc. Nitric acid and cap tightly in the hood. Shake well to make sure acid is well mixed. Place in the ultrasound overnight and sonicate.
2. Remove the jug from the ultrasound, open in the hood and drain into a colander placed in the hood sink. Rinse each tube individually, inside and out, several times with Milli-Q water and place in racks in the oven to dry. Place the caps in a tray to dry.
3. When the tubes are dry, cap them and place them in the ACID WASHED 15 ml or 50 ml tube box as appropriate.

## *Waste Disposal:*

**Excess diluted acid** – flush down the drain with copious water.

**1% Nitric acid washing solutions** – flush down the drain with copious water.

**Be-contaminated gloves, pipette tips, and wipes** – place in Be-waste bag, seal, and retain in waste bin under the hood. Do not place in trash.

**Potassium Perchlorate remaining after centrifuge step** – wash into labeled waste bucket, cap, and store in waste bin under hood.

**Fusion Cakes** – wash into labeled waste bucked under the hood. Cap and store in waste bin under hood.