

UVM Cosmogenic Laboratory – *Meteoritic ¹⁰Be Extraction*

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

Purpose: This method details the means by which we extract meteoritic ¹⁰Be, 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.

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 (use nitrile under neoprene when adding flux and fluxing and using Perchloric acid), goggles, face shield (when fluxing), rubber gown (when fluxing and handling Perchloric acid), rubber lab shoes.

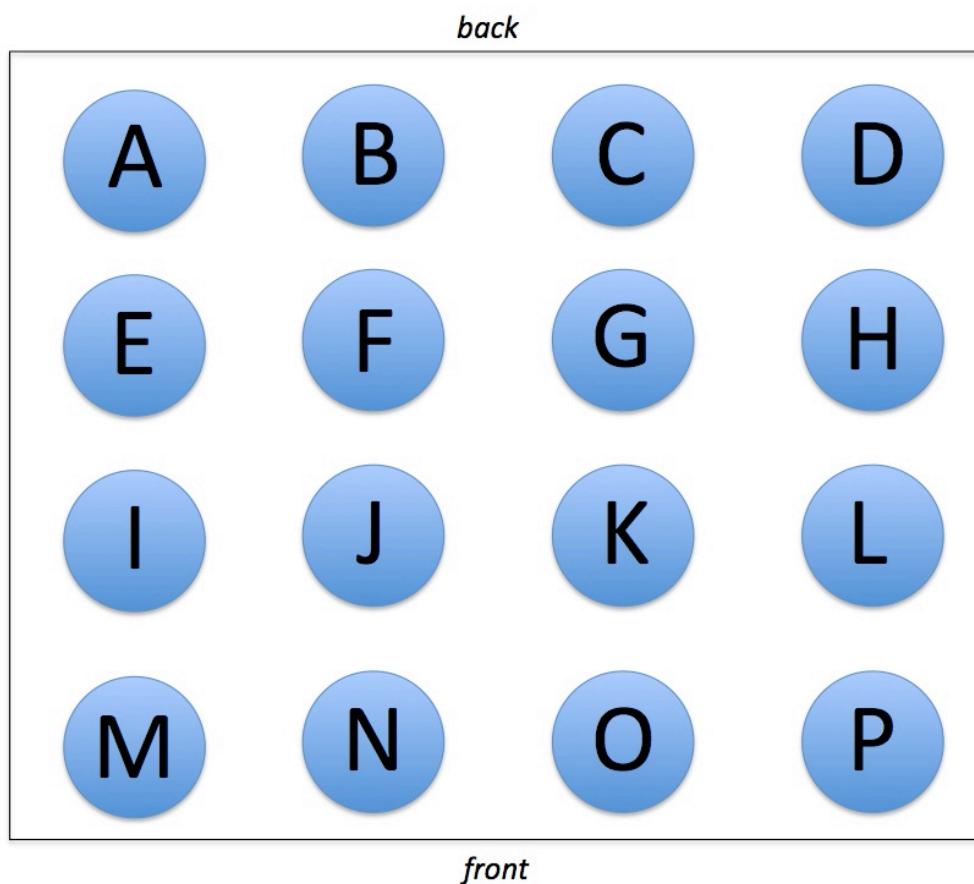
Decontamination procedures before leaving the Lab:

- 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.

Important Notes

1. Cleanliness is imperative, not only for you and your samples but for everyone who follows. Failure to clean labwear 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 and on the lab web site is an **ILLUSTRATED GUIDE** to performing this method. **BEFORE** doing the method, check it out!

Super-cleaning for low-level samples (this is a two day process)

If running low-level samples after running high-level samples (or if you are a paranoid person), you will want to more aggressively clean the Pt crucibles and teflon before starting. Do this by placing each crucible in a 180 ml teflon beaker and adding the >10% NaOH solution until the crucible is immersed. Cap and heat on hotplate at 98C overnight. Drain the NaOH back into the

storage container. Rinse (milli-Q) the beakers and crucibles and wipe the crucibles with ecowipe to remove all softened residue. Fill the beaker and crucible with 10% Nitric Acid (stored under hood), cap, and heat overnight. Rinse well with milli-Q and dry in slotted tray on top shelf of oven. Before fluxing, wash down the inside of the hood with 1% nitric and DI sprayer. For very low-level samples, you may wish to use all acid-washed disposable tubes.

Getting ready

Select 15 samples to test and bring them into the meteoric lab. They must be fine powders or the method will not work. Use the SPEX shatterbox to produce these powders (see lab safety manual for rock 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.

DAY 1. Load and Flux

Preparing your data sheet [10 minutes]

1. Turn on keyboard and mouse. Open the optical port on the mouse. Press the button on the right side of the keyboard once.
2. Open the folder on the desktop entitled "Meteoric Batches".
3. Open file "meteoric_template".
4. Save the file (select type .xlsx) remembering to 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. Organize your samples in logical order. This is a key step.
7. Add all your samples ID's to the spreadsheet (in your logical order) beginning with the blank, which is called **BLK_MB#**.

Preparing the lab [10 minutes]

1. Carefully remove the hotplate from the hood after first wiping it down with ecowipe and DI 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 very expensive (\$3000).
2. Place the sink plug into the hood. Tape the nozzle.
3. Place the CAT hotplate in the hood and plug it in. It is stored under the sink. Turn on the hotplate. Set to 95 degrees. Make sure the shaker is off and CHECK to make sure the hotplate is WARM.
4. Put on CLEAN, thin gloves. Remove the dark, 16 hole, teflon-coated plate and the stainless plate from their storage bags and place them on the hotplate, stainless plate first. Make sure that you are wearing CLEAN gloves and try and avoid touch the holes. It is stored in bottom drawer in plastic bag.
5. 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.
6. Bring out the bag of stainless spatulas as well as a 500 ml beaker to hold them once they are dirty. Set the spatulas and beaker on the counter near the balance.
7. Move the fluxing apparatus into the hood. This is a two-person operation. Carefully remove, roll and save the plastic bag which 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]

1. Bring out 16 clean Pt crucibles and put them on the telfon-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. Make sure that you are wearing CLEAN gloves. 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. Set it in the hood in a small spill tray. Also bring out the liquid Be waste container and set it in the hood on the same spill tray. You need to decide which carrier to use. For low-level samples, such as volcanic rocks and Greenland in ice sediment, use beryl carrier.
4. FOR HIGH LEVEL CARRIER (most samples), bring out the repeat pipettor and a new 2.5 ml tip (they are both in a drawer labeled repeat pipettor). FOR LOW level carrier, 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 300 ul (HIGH LEVEL) and 500 to 600 ul (LOW LEVEL, depends on carrier concentration, aim for 300 ug of Be in each sample). Shot 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.
5. One at a time, starting at "A" remove a crucible and place it on the balance using its the 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.
6. 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.
7. Beginning with the Blank, 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.
8. Record the sample mass and double check the sample name on the spreadsheet on the computer. SAVE THE FILE after each sample.
9. TARE the balance. Remove the pipette from the hood. Open the top of the balance. Pipette 300 ul of carrier into the sample using the repipettor upper lever.
10. 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.
11. Place the sample back on the hotplate in the hood.
12. Repeat the above steps (5 to 9) for all samples until you are done.
13. Double check that the temperature on the hotplate is set to 95°C and that the *power* is on and that *plate* is on and WARM.

14. Leave the crucibles heating on the hotplate for approximately 2 hours to dry down.
15. Print the data sheet and tape it in the lab batch book.
16. Turn the mouse off by sliding the cover over the optical path and turn off the keyboard by holding the power button down for a few seconds until it blinks.

NaSO₄ and KHF₂ addition and sample fluxing: [~3 hours]

1. 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 parafilm beakers also stored under the hood.
2. Pre-label 180 ml Teflon beakers with green tape on the upper sides. Use the letters A-P and arrange the beakers 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 on the counter. Use the labeled comparison beaker stored in the bottom drawer to get the water height right.
3. Place a clean tile in the tray (they are stored in the lower drawer in a plastic bag). Place a clean plastic weighboat on the tile. Start with sample A. Use its dedicated blue tongs to pick up the crucible and set it 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 NaSO₄ using the dedicated scoop. The mass of NaSO₄ amount should ~ equal the sample weight. Do this in the hood while the crucible is sitting on the weighboat.
5. For a 0.5 gram sample, add 3 level scoops of anhydrous KHF₂ to (~5 times the sample weight) using the large scoop. Do this in the hood while the crucible is sitting on the weigh boat.
6. Mix the contents of the crucible thoroughly using the dedicated teflon stir stick. 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 cap 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.
8. 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.
9. Set out Pt tipped tongs. Set out a clean stainless spatula on the edge of the fluxing stand so the tip is suspended in air. Turn OFF the hood blower and light. Turn off the room light. (switches are on upper left of hood face).
10. Set a clean alumina ring on the fluxing stand.
11. 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.
12. Bring over the proper beaker (the one labeled A) and set it in the spill tray.
13. 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.
14. PULL the sash ALL THE WAY DOWN.
 15. 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.
 16. When the fluxing is done, turn off the gas then the oxygen and let the crucible cool until the flux has solidified. It will turn grey and may crack. This takes about 60 seconds. Once the flux has solidified, open the sash enough to take the lid off the beaker.
 17. Use the Pt-tipped tongs to lift the crucible off the 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. It will hiss. Try not to put the tong tips in the water. Cap the beaker tightly and set it back on the counter. To make this work, grasp the left side of the crucible when removing it from the torch stand.
 18. 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.
 19. Repeat steps 11-19 for all 16 samples.
 20. When all samples are completed, remove the telfon-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 also under the sink.
 21. 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 DI water from the squirt bottle to remove any nitric. Dispose the wipes as Be waste in the waste bag.
 22. Make sure both GAS VALVES ON THE HOOD ARE OFF. Disconnect the gas hoses. Cover the fluxing stand with its plastic bag and move it back under the sink.
 23. Turn off the gas at the wall and turn off the oxygen at the tank. Open the gas and oxygen valves ON THE HOOD.
 24. 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. 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.
 25. 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.

26. 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.
27. Turn the hood blower back on before leaving lab

Day 2. Extract and purify

Fusion Cake Removal [1 .5 hr including clean up]

1. Turn off the hotplate and let samples cool for an hour or more. They contain HF. If in a hurry, remove the samples and order them on the hood deck in the standard 4 by 4 matrix. Let samples cool 15 minutes. 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.
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.
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 up side 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. 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. When you are done, and the crucible is completely cleaned out, place it in the wash bin that is filled with water. Place the stir stick and blue tongs in a 4 liter cleaning jug.
7. Place the beaker for sample "A" back on the hotplate in the standard matrix order.
8. Place the crucible in the washbin under water.
9. Drop the stir stick and blue tongs into a 4 liter washing jug.
10. Repeat steps 3 to 8 until all 16 beakers have been done.
11. When the hotplate is full, turn it on and set to 165 degrees. The idea is to reduce the volume of the liquid to between 10 and 40 ml but not to let the solutions go to dryness. This dry off will take 2 to 3 hours. Keep checking on levels using the labeled beaker as a guide.
12. Rinse the crucibles several times with DI water being careful to minimize splash. Carefully place them upright in into 2-liter nalgene in layers starting with 7 on the bottom, three teflon watch glasses, 7 more, three more watch glasses, and then two more. The idea here is to prevent any 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. This step softens any remaining flux on the crucible walls.
13. Fill the stir stick jug with DI water, add 60 ml concentrated nitric acid, and set on the ultrasound to sonicate for the day.

Perchlorate Precipitation

[~3 hours]

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'.
2. Put spill tray into hood with Milli-Q squirt bottle in side 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. Use the wash bottle to add an ml or two of Milli-Q water to the beaker, swirl and pour again.
3. In hood, bring up the tube volume to 30 ml using Milli-Q squirt bottle.
4. Centrifuge at 2500 RPM for 5 minutes. Gently remove the tubes from the centrifuge so as not to disturb them.
5. In hood, rinse each Teflon beaker with DI and place in a pair of 4 liter wash jugs.
6. 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.
7. Put on yellow rubber smock and double glove.
8. 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!**
9. Bring out the repipettor and use a NEW 50 ml reservoir used for Perchloric acid. Place the container and the repipettor on the square spill tray.
10. Find another spill tray and place the green rack in it. Uncap one row of tubes laying the cap next to each, top down on the spill tray.
11. Set the vortexer in the hood. Turn the hotplate side ways to get more room.
12. Fill the repipettor and deliver 5 ml of perchloric acid to each 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 vortex thoroughly, invert again and vortex one more time. Repeat for all samples. Make sure the sash is WAY down for protection.
13. Let precipitate settle for 20 minutes. Then remove caps and add 5 more ml of perchloric acid to each sample. Cap tubes tightly, invert several times, and vortex thoroughly, invert again and vortex one more time. Let precipitate settle for another 20 minutes.
14. Centrifuge each at 2500 RPM for 5 minutes.
15. 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.
16. Add 5 ml of conc HNO_3 to each beaker and place on the hotplate, uncovered, in the white beaker-stand. Set hotplate to 230 C.
17. Allow samples to dry down to a white cake overnight.
18. Inside the hood, rinse the KClO_4 waste from tubes used in the previous step into a waste container. Triple DI rinse the tubes.

Day 3: Beating the Boron

1. For all of these steps work back to front across rows because acid will start fuming immediately.
2. Do this early in the morning. Keep the hotplate at 230. Add 5 ml conc HNO₃ to each sample using the repeat pipettor and the dedicated HNO₃ tip. Evaporate off until completely dry and then some – at least two hours
3. Once HNO₃ is completely dried off, add 5 ml conc HF to each sample using the repeat pipettor and the dedicated HF tip. Evaporate off until completely dry and more...at least 5 hours. The final cake looks different at this point – fluffy. That's OK. The AMS folks will thank you for doing this step well.
4. In the evening, add 10 ml of Perchloric acid using the repeat pipettor. Let this dry off overnight.

Day 4: Recover and Precipitate BeOH

Be Recovery

1. Set the hotplate to 60C and let it cool until the hotplate temp is 60C, about an hour.
2. Inside Hood, add 20 ml of 1% OMNITRACE HNO₃ to each beaker (NOT THE WASHING NITRIC!). The 1% OMNITRACE HNO₃ is found under the hood. The cakes should dissolve within about 15 minutes.
3. Label 16 of the ACID-CLEANED disposable 50 ml tubes from A-P 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 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 one by one. 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 (no more!) for good measure.
7. Let the tubes sit for an hour; a very small amount of precipitate (Be(OH)₃) will form. It's almost impossible to see.
8. Centrifuge at 3000 for 5 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 enough, the gels should stick in the bottom of the tube.
10. Add 10 ml of MILLI-Q and re-suspend the gel using the vortexer.
11. Transfer each to a clean, labeled 15 ml tube. Rinse the 50 ml tube into the 15 ml tube once with an ml of MILLI-Q.
12. Centrifuge again this time at 3000 rpm, and pour off DI into waste beaker.

13. At this point, add 1% HNO₃ (in MQ water) to the 8 ml level on the tube. Do this carefully to get the same volume in each tube. Use the repeat pipettor to add the acid.
14. CAP and VORTEX each sample very well so it is completely mixed and the gel is dissolved.

ICP Splits for Be yield and purity test

1. Take out the 1 ml variable pipettor and a box of tips.
2. Take out and rack up and label 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. Take a 0.2 ml aliquot from each sample and place it in the appropriate purple cap tube. Do this OPENING ONLY ONE tube a time. This a STEP where MENTAL CONCENTRATION is key to avoid cross-contamination.
5. Once there is solution in every purple tube, use the repipettor to add 5 ml of weak (1.0 M H₂SO₄) to each purple tube. VORTEX WELL!

Final Precip and Wash

1. Add one drop of methyl red to each sample tube from the dropper bottle.
2. 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 3 and then go one by one. 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 (no more!) for good measure.
3. Let the tubes sit for an hour; a very small amount of precipitate (Be(OH)₃) will form. It's almost impossible to see.
4. Centrifuge at 3000 or greater for 5 minutes. There should be a small amount of Be(OH)₂ gel at the bottom of each tube.
5. Carefully, very carefully decant the supernate into a waste beaker. If centrifuged enough, the gels should stick in the bottom of the tube.
6. Add 10 ml of MILLI-Q and re-suspend the gel using the vortexer.
7. Centrifuge again, the Be gel will reappear at the bottom
8. Pour off DI into waste beaker and cap tube.
9. Repeat for all. When done, pour the water in the waster beaker down the sink.

Drying gels

1. Do this when nothing else is going on in the hood.
2. Place each sample in the dry down block on big hotplate. Take off the caps and arrange in a spill tray, top down.
3. Set the hotplate to 75C until the gel is dry. This is key, slow cool drying gives great pellets.
4. If after the gel is dry, there is condensate on the tube walls, increase heat to 100C.
5. When dry, recap using the correct caps, and set aside for packing.

Cleaning Procedures:

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

1. Individually rinse the 16 crucibles with Mill-Q and make sure that all remaining fusion material is removed. If needed, use wipe to clean the crucible. 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 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.
2. 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 – using the same technique as before to make sure the crucibles don't nest. Place bottle in the ultrasound and sonicate at least overnight.
3. Remove the jar from the ultrasound, and pour the 10% Nitric into its storage bin 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 crucibles in their tray on the top shelf of the drying oven.
4. 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 $KClO_4$, 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. 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 tube box.

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, pipetette 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 bucket under the hood. Cap and store in waste bin under hood.