Rock Crushing and Sieving - Rock Room, Delehanty 108

(Takes approximately 30 minutes per sample)

Personal Protective Equipment: Dust mask, shatterproof safety glasses, hearing protection.

Notes before beginning:

- The Rock Room and equipment is a communal area shared by multiple lab groups. Make sure you reserve space in the room ahead of time by writing your name on the schedule pinned to the front door.
- After using equipment in the Rock Room, fill out the use log for that machine (they are typically pinned to the alcove doors). In the “Group” space, put “Cosmo Lab”.
- Your rock sample will be pulverized after this process. If you need to make any final observations (for example, measuring the sample thickness) or take photos of the intact rock sample, now is the time to do so!
- The Cosmo Lab has two sieves (mesh size 250 and 850 microns) that are stored in the mineral separation lab. You will need these for the rock crushing process. Make sure to take these back up to the mineral separation lab when finished.
- **IF YOU ARE WORKING WITH SEDIMENT, you will only do steps #12-20 and #26**

Instructions:

1. Turn on the exhaust fan. On the left-hand side of the alcove, you will see a dial labelled “Exhaust Fan”. Turn this dial to the “on” position and wait for about a minute. You will eventually hear fans.
2. Open the double doors to the jaw crusher and disc mill alcove and inspect both machines. The jaw crusher is on the right side of the alcove and has a large opening at the top that allows users to drop rock samples in. The disc mill is on the left side and has a smaller chute on the left-most side of the machine that lets users pour crushed rock

into the central chamber. Open the disc mill by lifting the lid up, rotating both handles on the center chamber, and swinging the chamber open on its hinges. If visible crushed rock fragments or rock flour (fine dust) are present in either machine, clean them before use. There are several brushes and a dustpan in the Rock Room drawers to help with this. Check the plastic catch tray under the jaw crusher for rock fragments and check the pull-out catch drawer in the disc mill for any sediment. For deep cleaning, there is a compressed air hose hanging on the right-hand side of the alcove. To turn on air, turn the yellow handle to be in-line with the hose. Always wear a mask and eye protection when cleaning.

3. Position the plastic catch tray under the jaw crusher.
4. Dust mask, ear, and eye protection must now be on! Turn the jaw crusher on. Look for a metal utility box on the right wall next to the jaw crusher. There is a lever with labelled "on" and "off" positions. Flip this lever to the "on" position. Next, look for a big red button and pull until you feel it click into position. The jaw crusher will start making a loud sound as the heavy plates within start to move back and forth.
5. Carefully drop a rock sample into the jaw crusher. Do not lower your fingers below the opening of the jaw crusher, drop your sample from a few inches above the opening. You will hear the sample getting crushed by the plates within, and the shattered pieces will fall to the catch tray below.
6. If your sample is in multiple fragments, continue dropping the pieces into the jaw crusher until they have all passed through.
7. Turn off the jaw crusher by pressing the big red button **AND** turn off the power supply to the machine using the breaker.
   Important: If the machine is not switched off in both places, it becomes a major safety hazard. Take time to ensure you know both switches are off.
8. Inspect your crushed sample. Most of the pieces should be gravel to small pebble in size. If you have a few larger pieces, use a hammer to break them down to a smaller size.
9. Move to the disc mill. Make sure the handles that lock the plates in place are secured and tightened.
10. When you are satisfied with the size of the disc mill gap, close the disc plate cover and carefully begin dropping your crushed rock fragments into the sample chute on the left side of the disc mill. Do this slowly, as the disc mill can jam up if too much material is poured in at once.
    a. In the event that the mill jams up (it stops rotating and makes a straining sound), press the red button to stop the machine AND flip the lever to cut power to the machine. Wait a minute or two for the discs to cool down, and the turn the spacing knob counterclockwise a bit to slightly widen the disc gap. Turn the

machine back on and the jam should clear up. Turn the spacing knob clockwise to bring the gap back down to the previous spacing.

11. After you have poured all of your crushed rock fragments into the disc mill, turn off the mill and wait for the discs to stop spinning **AND** turn off the power supply using the breaker.
    Important: If the machine is not switched off in both places, it becomes a major safety hazard. Take time to ensure you know both switches are off.
12. Remove the sample catch tray from the bottom of the disc mill and take it over to the sieve shaker.

13. Make sure that the 250 and 850 micron sieves are loaded on the sieve shaker (the 850 sieve must be on top of the 250 sieve) with a catch tray on the bottom. If needed, use a wrench to adjust the height of the sieve stack so that the top of the 850 micron sieve is level with the top of the sieve shaker (there should be a drawer labelled “tools” in the Rock Room with wrenches).
14. Pour your sample into sieve stack and put the lid (a metal disk with a cork in the middle) on top.
15. Lower the sieve shaker arm so that it rests on top of the sieve shaker lid.
16. Turn the sieve shaker on, make sure it is set for at least one minute, and start the machine. This will make a loud banging sound; close the alcove door to muffle the sound.
17. When the sieve shaker stops, lift the arm, remove the lid, and carefully remove the sieve stack from the shaker.
18. Lift off the 850 micron sieve and pour the coarse sediment into the plastic catch tray that you previously used to hold the crushed rock fragments.

19. Label a plastic sample bag with the sample ID and “250 – 850 µm” and pour the sediment in the 250 micron sieve into this bag.
20. Label another plastic sample bag with the sample ID and “<250 µm”. Pour any fine material in the bottom sieve catch tray into this bag.
21. Assess how much material you have in the 250 – 850 micron fraction. You will have to make a judgement call about whether or not you have sufficient material in this size fraction, or if you should do another round of disc milling (with a narrower gap) and sieving to obtain more material.
22. This decision can be tricky, feel free to ask a lab expert for advice. Ultimately you will need 20-40 grams of clean quartz for the beryllium extraction procedure. Depending on how quartz-rich your sample is, this could mean that you need anywhere from 40 to several hundred grams of sediment to obtain this mass. As a general rule, the less quartz-rich your sample is, the more sediment in the 250-850 micron size fraction you should try to obtain before starting mineral separation.
23. If you decide to do another round of disc mill and sieving, take the sediment that is >850 micrometers and bring it back to the disc mill.
24. Turn the spacing knob on the disc mill clockwise to narrow the gap between the plates, close the disc plate cover, and start the machine.
25. Repeat steps 9-12, pouring your sample into the disc mill and then sieving the resulting crushed sediment.
26. Again, assess how much sample you now have in the 250-850 micron size fraction. If necessary, repeat steps 9-21 as many times as needed to obtain sufficient sample material. By going through several stages of narrowing the disc gap, we reduce the amount of sample that gets pulverized too much (becomes too fine) and is thus unusable.
27. When you are satisfied with your 250-850 micron sample mass, label another plastic bag with your sample ID and “>850 µm” and pour any remaining sediment that is larger than 850 micrometers into this bag.
28. Clean the jaw crusher and disc mill before starting on a new sample. First, double-check that both units are off **AND** that their respective power supplies are off. Open the disc mill to expose the disc plates. Use brushes and the dustpan to sweep away any rock fragments, sediment, or rock dust. Dispose of these in the rock waste bin under the yellow magnetic separator. Next, use the vacuum to thoroughly clean the entire area and all surfaces of both machines. Finally, with the air vent still running, use the compressed air hose to spray any rock dust off the disc plates.
29. Clean the sieves using the metal brushes in the rock room. Over the sink, brush the sieves to clear out any grains that became lodged in the mesh. Do this carefully so as not

to tug excessively on the wires and potentially rip any holes. Try to get as many grains as possible out of the wire.

30. When the jaw crusher, disc mill, and sieves are clean, you are ready to move on to the next sample. You will repeat steps 3 – 29 for all samples.
31. After your final sample, turn off the jaw crusher, disc mill, compressed air hose, and ventilation fan. Vacuum the entire area, wipe down the countertops, and take out the trash. Make sure all lab spaces are clean for the next user.

IF GRINDING PRODUCES MATERIAL THAT IS TOO COARSE OR TOO FINE

The size of this gap determines how coarse or fine your sample will be ground by the disc mill (bigger gap = coarser grind).

It is better to start with a larger gap (~1-2 mm) and go through several rounds of narrowing to obtain as much sample material in the desired 250 – 850 micron size range as possible. Starting too narrow can result in excessive production of sediment under 250 microns that cannot be used for nuclide extraction.

If the gap is too narrow (plates are too close together), turn the knob on the right-hand side of the disc mill counter-clockwise part of a turn and try a test sample. If the gap is too wide, the plates are too far apart. You then need to turn the turn the knob on the right-hand side of the disc mill clockwise part of a turn. Remember, only do this when no sample material is in the disc mill.

Magnetic Separation - Rock Room, Delehanty 108

(Takes approximately 20-30 minutes per sample)

Personal Protective Equipment: Dust mask, shatterproof safety glasses.

Instructions:

1. Turn on the magnetic separator, a big yellow box that we lovingly call Big Bird, by pulling on the big red button. You will hear the instrument starting up.
2. On the front of Big Bird are switches to turn on the magnet ("Magnet Control"), the spinning wheel ("Roll Speed Control"), and the vibration for the sample funnel ("Vibrator Control"). Begin by turning on the magnet and wheel.
3. The settings for the magnet and wheel should be set as follows. If a setting does not match, use the buttons next to the screen in question to adjust the setting:
    a. Magnet: 12
        i. Note that the magnet control will probably display an error message in red. This is ok, pay attention instead to the small green number underneath.
    b. Roll Speed: 35 rpm
4. Position the two metal trays under the funnels at the bottom of Big Bird.
5. Position the metal separating plate to the right of the wheel.
    a. The wheel is magnetized, so magnetic grains stick to it as it spins and eventually get forced off by the stiff brush on the other side of the wheel. Non-magnetic

grains (such as quartz) will instead fall off the wheel and land in the other metal tray. However, this process is not perfect. Some magnetic grains inevitably fall off the wheel early, and some quartz grains stick to the wheel for too long. The position of the metal separator plate determines how much of the sediment flow ends up in each collecting tray. Positioning the plate very close to the wheel will result in more magnetic grains ending up in your non-magnetic fraction, but positioning it far away runs the risk of some quartz grains ending up in the magnetic fraction. Generally, if you have a large sample, you can position the plate farther from the wheel and risk losing some quartz to the magnetic fraction while having a more complete removal of magnetic grains. On the other hand, with a small sample you might want to position the plate closer to the wheel to preserve most of the quartz in your sample while knowing that the separation of magnetic grains may not be as effective.

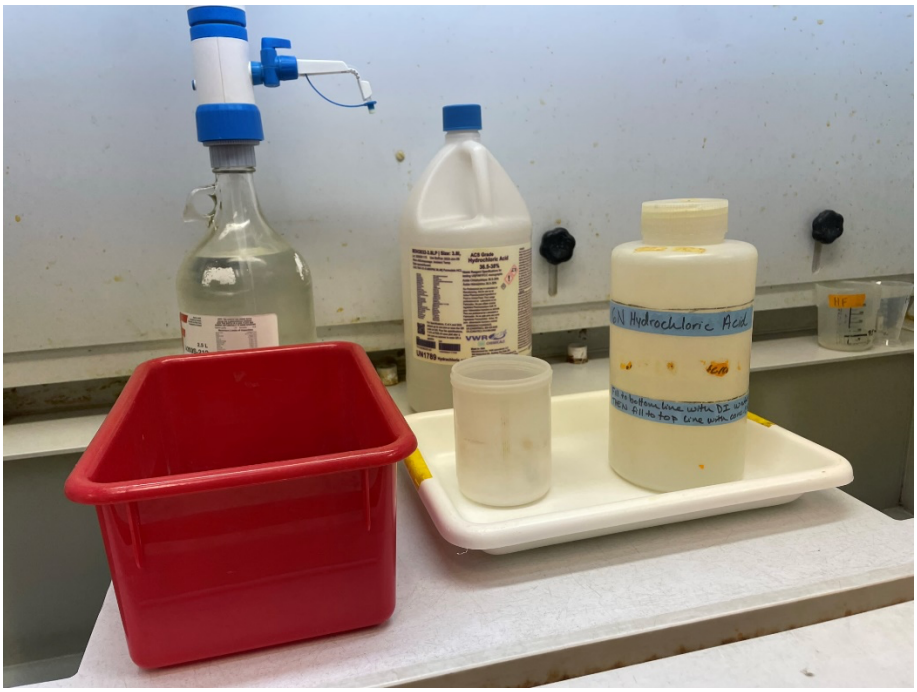
   b. You can always adjust the wheel as your sample starts flowing across the wheel, fine tuning it until you are satisfied with the separation.

6. Flip the “Roll Speed Control” switch to “On” to start the wheel.
7. Make sure that the “Vibrator” knob is set to zero.
8. Take your sample and pour it into the hopper at the top.
9. Slowly start turning the vibrator knob up; this determines how fast your sample gets poured onto the magnetic track. You want to aim for a moderate sample feed speed, not so fast that the magnetic pull of the wheel is ineffective, but not so slow that this process becomes inefficient.
10. Observe the separation of grains as they start flowing down across the wheel and into the two collection trays. If you see lots of black specks ending up in the non-magnetic fraction, consider moving the separation plate a little farther from the wheel. On the other hand, if the magnetic fraction is accumulating much faster than the non-magnetic fraction, move the plate a bit closer to the wheel to catch more grains in the non-magnetic fraction.
11. When all of your sample has gone through Big Bird, flip the “Roll Speed Control” switch to “Off” to stop the wheel. Turn the vibrator knob to zero and turn off the magnet.
12. Label a plastic sample bag with your sample ID, “250-850 µm”, and “Non-Magnetic”. Pour the non-magnetic fraction into this bag.
13. Pour the magnetic fraction into the same sample bag you previously used for this sample but add “Magnetic” to the label.
14. Clean Big Bird for the next sample. Use the broom, vacuum, and compressed air to get into the tight spaces on the machine. Remove the black brush that sits below the wheel for cleaning as well, then replace it before starting the next sample.
15. Repeat the entire process for all samples.

Hydrochloric Acid (HCl) Etches – Mineral Separation Lab, Delehanty 326

(Typically takes part of three days to do two successive etches)

Personal Protective Equipment: Thin gloves while setting up and doing initial wash. Double gloves (thin and thick), rubber smock, goggles, and face shield for the rest of the procedure.



For this procedure you will need:

- Up to 22 samples
- Plastic, rectangular bins (1 for each sample)
- HCl etching beaker (1 for each sample)
    - IMPORTANT: Make sure you are using the HCl etching beakers, which have lids with one or two vents in the top.
- HCl beaker holder (a rigid, plastic sheet with large holes cut into it, typically stored to the left of the ultrasounds against the back wall; each has 11 slots and there are 2).
- Concentrated HCl and mixing jug (located under the mineral separation chemical hood. The mixing jug is a large, Nalgene bottle with a wide cap and labeling on the side indicating mixing volumes).
- Spoon
- Large spill tray
- Lab Tape
- Sharpie
- Ziplock Bags

Starting process – washing sample

1. Place the HCl beaker holder on an ultrasound bath. This holder allows the HCl beakers to sit high enough for their bodies to remain submerged while their lids are out of the water.
2. For each sample, use lab tape to label one rectangular bin and one HCl etching beaker lid with the sample ID.
3. Pour the sample into the HCl etching beaker until the beaker is about two-thirds of the way full (this is just for the sake of measuring the proper amount). Then, transfer the sample material to its labeled rectangular bin.
4. Rinse the sample material many times (at least 5-6, as many as 10-12 if needed) in tap water, swishing sample back and forth, until the runoff is clear.
5. Transfer the sample from the bin to the labeled HCl etching beaker. Using a spoon can help with sample transfer, but make sure to wash the spoon thoroughly between samples to avoid cross contamination. Using a squirt bottle with water can help to remove sample material from the bin. Carefully pour off any excess water in the beaker.
6. Rinse off any remaining grains from the rectangular bin and set on drying rack. Do not remove sample label, you will need the bin again over the next couple of days.
7. Repeat steps 2-6 for all samples.

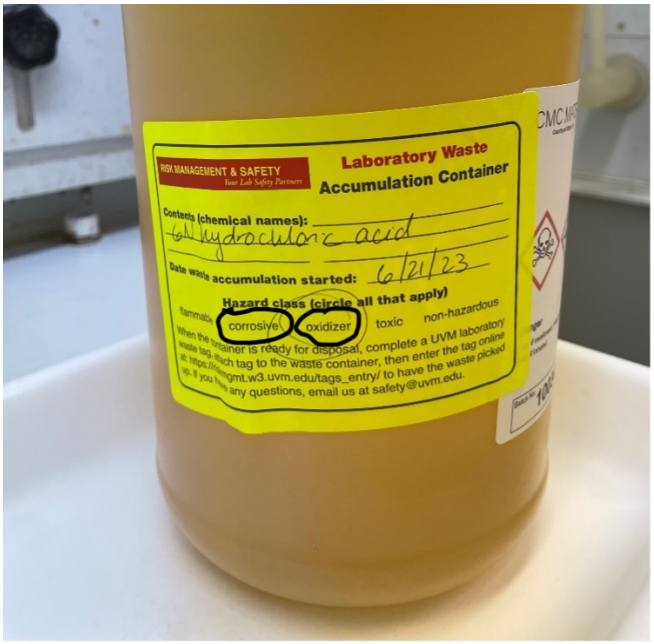
Adding acid to remove grain coatings and adhered meteoric 10Be

1. **PUT PPE on at this point!** Bring the HCl container, HCl mixing jug, and a large spill tray into the chemical hood.
2. If any liquid is left in the HCl mixing jug from previous users, proceed directly to adding acid step. If not, mix dilute acid.
3. Fill the mixing jug with DI water up to the first line.
4. With the mixing jug in the large spill tray, *carefully* pour HCl directly from its container into the mixing jug, filling to the second line. Put the cap securely on the mixing jug and slowly invert a couple of times to mix the acid while holding the bottle over the sink.
5. Cap the HCl container and move it out of the way (against the back of the hood).
6. One at a time, take a labeled HCl etching beaker with washed sample and set it in a spill tray. Pour a small about of diluted HCl from the mixing jug into the beaker with sample. **Wait and watch to see if the sample reacts** – if it foams and bubbles, the sample contains carbonate minerals. Do not add more acid until the reaction subsides. If there is a large amount of carbonate and the reaction continues, find Paul or Lee. The sample may need to be transferred to a larger bottle (1 liter or 4 liter) where we add acid slowly until all the carbonate is gone. Failing to remove carbonate can cause the sample to react violently in the ultrasound spilling acid and damaging the metal tank. Do NOT move the sample to the ultrasound if it is still reacting.
7. Fill the beaker with acid to about an inch above the level of the sediment.
8. Put a lid on the beaker and carry it to the ultrasound using two hands, holding the beaker by the bottom and not by its lid. Place it in one of the slots in the beaker holder.

9. Repeat the process above for all samples, re-mixing the diluted HCl when you run out. Leave one hole open in the beaker holder for adding water to the ultrasound. There are two HCl beaker holders, so you can use two ultrasounds to increase the number of samples if needed.
10. When finished, return the HCl and the HCl mixing jug to its designated storage bin below the hood.
11. When all beakers are in the ultrasound holder, fill the ultrasound with water until the water line reaches the top row of holes (on the inside surface of the ultrasound). If you are trying to fully maximize sample numbers, fill to just below this line and add another beaker to the last hole in the beaker holder.
12. Set the timer on the ultrasound by turning the dial so that the pointer on the outside of the dial is just to the left of the fixed starting position (at the "6-o'clock" position). Turn the ultrasound on by flipping the timer switch to 'on' (this switch is inside the timer box, below the dial). When on, the ultrasound will make a high-pitched buzzing sound. Touch each ultrasound and make sure you feel a subtle vibration.
13. **CLEAN UP** Rinse the spill tray and spoon three times with DI water. Wipe down the deck of the hood with a squeegee, then close the sash fully. Make sure all water is off.
14. Sonicate the samples at least overnight and preferably 20+ hours

Draining acid and rinsing samples

1. Check to see if there is an HCl waste collection container currently in use. This would be located with the HCl containers under the hood. Waste collection containers are typically re-purposed Hydrofluoric acid (HF) containers, and they must have a large, yellow label on the side with a description of their contents. If a partially-filled waste collection container is available, proceed directly to step 4. If not, create a new waste collection container (steps 2-3).
2. To make a new waste collection container, first obtain an old, triple-rinsed acid container. These are stored in the back-right corner of the mineral separation lab and have a sticker on the side of them with the triangular recycling symbol and a label saying 'triple-rinsed'.
3. In the same drawer as tape and pens (the top left drawer), find the yellow waste-labelling stickers. Fill one of these out by noting the current date, the chemical being poured into the container ("6-normal hydrochloric acid") and circling both 'corrosive' and 'oxidizer'. Affix this label to the side of the triple-rinsed HF container. You are now ready to use this as a waste collection container.



4. **PPE must be on at this point!** Carefully lift each acid beaker, one at a time. Check the color of the liquid in any beaker. If the liquid is very light yellow, then one HCl etch is sufficient and the sample can be washed of acid with repeated rinses and dried. If the sample is dark yellow, orange, or red, an additional HCl etch is necessary. **Make a note of the color of each beaker's liquid before rinsing.**
5. Uncap the waste collection container and place a plastic funnel in the mouth. Set the container in the left-hand sink (which you will not be using) to provide additional secondary containment.
6. One at a time, take an etching beaker out of the ultrasound and carefully bring over to the chemical hood, carrying it with two hands. Uncap and pour the HCl into the waste container, making sure to always angle the beaker so that you are pouring away from yourself.
7. Three times, fill the beaker with water and use a spoon or a stir stick to mix the sample. Decant the wastewater into the sink.
8. Get the rectangular bin associated with the current sample and, inside the hood, pour the sample from the etching beaker into the bin. You may need to rinse the beaker a few times with DI water to get all the sample material out of the beaker.
9. Rinse the sample at least 5 times with copious DI water in the rectangular bin. If the water is still murky after 5 rinses, continue rinsing until the water runs completely clear.

If the sample's liquid was light yellow, etching is finished and it's time to rinse and dry

1. Rinse the HCl beaker thoroughly in the hood so that you are not bringing acid fumes into the lab space. Then, move the beaker to the outer sink and triple rinse with hot water and, if necessary, a scrubbing brush. Place the clean beaker on the drying rack.
2. Turn the oven on and place the bin containing the sample in the oven for drying.
3. Repeat the step above for all samples.
4. Allow any samples in the oven to dry overnight. Larger samples will need more time.
5. Once the samples in the oven are **COMPLETELY DRY**, take them out and let them cool for 30 minutes (otherwise the heat will melt the plastic). Label plastic Ziploc bags with each sample name, "250-850 μm "and "HCl-etched". Carefully pour each sample into its matching bag.
6. **CLEAN UP -** Rinse the spill tray, funnel, and spoon three times with DI water, then set on the rack for drying. Cap the HCl waste bottle and place it in the HCl bin below the hood. Wipe down the deck of the hood with a squeegee and make sure all water is off. Close the sash completely.

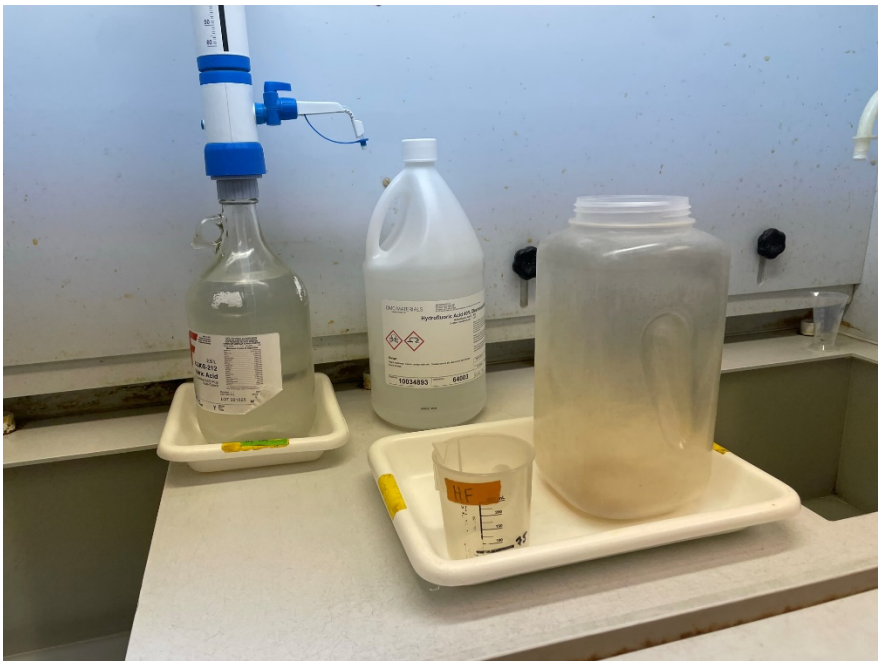
If the sample's liquid was red or orange:

1. Repeat the instruction in the section titled, **Adding acid to remove grain coatings and adhered meteoric 10Be** until acid is yellow. Then rinse and dry as described above. While most samples will be fine after 2 HCl etches, some (especially those from deeply-weathered environments) may need 3 etches.

1% Hydrofluoric and Nitric Acid (HF/HNO_3) Etches – Mineral Separation Lab, Delehanty 326

(Typically takes four days to do three successive etches)

Personal Protective Equipment: Thin gloves while setting up and massing samples. Double gloves (thin and thick), rubber smock, goggles, and face shield while handling acids.



For this procedure you will need:

- 4-liter Nalgene jugs (at least 1 for each sample, potentially 2 or more if sample masses and ultrasound availability allows)
- Balance
- Concentrated hydrofluoric acid (stored under the fume food)
- Small plastic beaker for measuring hydrofluoric acid volumes (typically located on the deck of the chemical hood)
- Nitric acid with top-mounted volume dispenser
- Two large spill trays
- Sharpie
- Lab tape
- 50 mL centrifuge tubes (one per sample)
- Two centrifuge tube holders (one large and one small)
- Clean paper sheets

Notes before starting

- No more than 40 grams of sample material can be added to each 4-liter jug. If your samples are relatively quartz-poor, you may have to use two or even three jugs to ensure that you will have enough quartz after these etches for the Be/Al extraction procedure (20-40 grams of clean quartz is the goal).

- Each ultrasound bath can fit three 4-liter jugs. Before you begin the etches, assess the number of jugs you will be able to fit into the available ultrasounds. Use this number and your evaluation of the quartz content in your samples to plan how many jugs you will use per sample, and if you will need to do multiple rounds of these etches to process all of your samples.

ACID NEUTRALIZING SYSTEM. We are allowed to dispose of diluteacid waste down the sink drain because there is an acid neutralizing system in Delehanty. However, that system can become overloaded. These is an orange indicator light on the wall near the drying oven. The light will turn on if the pH in the acid neutralization unit is out of range (usually from pouring to much acid too quickly down the drain). If this happens, pause the rinses for a few minutes and let tap water flow down the drain. When the light turns off, you can start rinsing again.

Day 1: Mass samples

1. Use lab tape to label one or more 4-liter Nalgene jug(s) with a sample ID. Place the label on the upper shoulder of the bottle so that it is not submerged below the water level in the ultrasound.
2. Put a labelled jug on the balance (without a cap) and tare.
3. Carefully pour the sample into the jug, limiting the sample mass to no more than 40 grams. Record the sample mass in the mineral separation tracking log. Note, samples should be dry before starting the HF/HNO_3 etches.
4. If using multiple jugs for the sample, repeat steps 2 and 3 for each jug.
5. Repeat steps 1-4 for all samples.
6. Fill jugs with DI water using the tap in the hood, add their lids, and stage them on the rolling cart near the chemical hood.

Add acid to 4 liter JUGS for etching

7. **PPE must be on at this point!** One at a time, bring one sample jug into the hood and place it on the spill tray.
8. Make sure that the volume dispenser on the nitric acid bottle is set to 50 ml. Position the nozzle over the jug in the spill tray. Pull up on the dispenser to load the dispenser, and then gently and slowly plunge it down to dispense the nitric acid into the jug.
9. Move the nitric acid bottle to the back of the chemical hood and bring the hydrofluoric acid bottle close to the sample jug. Place the small plastic measuring beaker on its own spill tray.
10. Very carefully, uncap the hydrofluoric acid bottle and pour 75 ml into the measuring beaker. The beaker has a sharpie line indicating the volume for easy visibility. Put the cap back on the hydrofluoric jug when done. The volume of acid here does not have to be

exact. Safety is the priority in this step, so aim for between 70-80 ml and pour carefully. PUT THE CAP ON THE HF JUG AS SOON AS YOU ARE DONE POURING.

11. Set the sample jug either in a spill tray or in the sink. Carefully pour the hydrofluoric acid from the beaker into the sample jug.
12. Put a lid on the sample jug.
13. Place the sample jug in an ultrasound, carrying it carefully with two hands, and holding it by the bottom instead of by the lid.
14. Repeat steps 7-13 for all sample jugs.
15. When all sample jugs are in the ultrasounds, fill the ultrasounds with water until the water line reaches the top row of holes (on the inside surface of the ultrasound basket).
16. Set the timer on the ultrasound by turning the dial so that the pointer on the outside of the dial is just to the left of the fixed starting position (at the "6-o'clock" position). Turn the ultrasound on by flipping the timer switch to 'on' (this switch is inside the timer box, below the dial). When on, the ultrasound will make a high-pitched buzzing sound. Put your gloved hand on the outside edge of the metal basket of each ultrasound. You should feel a subtle vibration.
17. Fill out the ultrasound use diagram on the front of the drying oven. Put your name and bottle contents (1% HF/HNO_3) on each ultrasound that you are using. If you are using several ultrasounds, you can put your name on one and draw an arrow across the others you are using.
18. **CLEAN UP** When finished, return the HF to its designated storage bin. Rinse the spill trays three times inside the hood before moving them to the drying rack. Rinse the HF beaker three times and store it inside the hood. Wipe down the deck of the hood using the squeegee and then close the sash fully. Make sure all water is turned off.

Days 2 & 3: Rinse samples and add fresh acid

1. When you arrive in the mineral separation lab, turn on one of the cold water faucets (NOT DI) in the hood. Leave it flowing as long as you are dumping acid.
2. **PPE must be on at this point!** Set up in the hood: the hydrofluoric acid container, measuring beaker, and large spill tray
3. Take a jug out of an ultrasound and bring it into the chemical hood, carrying it carefully and with two hands.
4. Unscrew the cap, give the cap a few rinses in DI water, and set aside in the spill tray.
5. Carefully, decant as much of the liquid as you can into the sink, ensuring no sediment pours out. You may have to pour slowly. Make sure to always pour away from yourself.
6. Fill the jug about a quarter of the way with DI water. Swirl the jug around to mobilize the sediment. This helps ensure that the water washes all grain surfaces. Let the sediment settle.
7. Carefully decant the liquid into the sink, again making sure not to let any sediment pour out. This is rinse number one.
8. Repeat steps 6 and 7 two more times, resulting in a total of three rinses.

9. Fill the jug up to the top with DI water.
   Note: When rinsing out the jugs on days 2-4, pay attention to the acid neutralization unit warning light. This is an orange indicator light on the wall near the drying oven. The light will turn on if the pH in the acid neutralization unit starts lowering too much (from pouring dilute acids down the drain). If this happens, pause the rinses for a few minutes and let water flow down the drain. When the light turns off, you can start rinsing again.
10. At this point, you can proceed to steps 11-15 for the current jug and add new acid before moving on to the next jug. Or you can repeat steps 3-9 for all samples, getting all jugs rinsed and filled back up with DI water, before proceeding to the next steps.
11. Add acid as described in the section above entitled, **Add acid for etching**
12. Fill out the acid disposal log. This is located on a green clipboard mounted to the cabinets to the left of the chemical hood. Record the date, how much acid was dumped, and the type of acid.
13. You will repeat this process on day 3.

Day 4: Rinse samples and put in drying oven

1. When you arrive in the mineral separation lab, turn on one of the cold water faucets in the hood (NOT DI). Leave it flowing as long as you are dumping acid.
2. **PPE must be on at this point**! Take a jug out of an ultrasound and bring it into the chemical hood.
3. Unscrew the cap, give it a few rinses in DI water, and set aside in the spill tray.
4. Carefully, decant as much of the liquid as you can into the sink, trying not to let any sediment pour out.
5. Fill the jug about a quarter of the way with DI water. Swirl the jug around to mobilize the sediment. This helps ensure that the water washes all grain surfaces.
6. Carefully decant the liquid into the sink, again making sure not to let any sediment pour out. This is rinse number one.
7. Repeat steps 6 and 7 two more times, resulting in a total of three rinses. Make sure to rinse the sides of the bottle thoroughly, both inner and outer, to remove all traces of acid since the bottle will now be removed from the hood.
8. On the final rinse, carefully pour out as much liquid as you can without losing any sediment and place the jug in the drying oven.
9. Rinse the lids well in the hood to remove all acid, then place in the outside sink, then rinse 3x with hot water, then put on the rack to dry. Repeat steps 3 – 9 for all jugs.
10. **CLEAN UP** When finished, wipe down the deck of the hood using the squeegee and close the sash fully. Make sure all water is turned off.
11. Can remove all PPE but thin gloves. Clear the ultrasound use diagram on the front of the drying oven, indicating to others that you are no longer using the ultrasounds.
12. Using the sharpie, label the 50 mL centrifuge tubes and their lids with the sample ID's and place in a large tube rack.

Massing and tubing the dry, etched samples

Leave samples overnight or until they are fully dry. With the thin gloves, check on the sediment in the bottles. If they are dry, you can turn the oven off and remove all bottles from the oven. If they are not dry, wait until they are dry

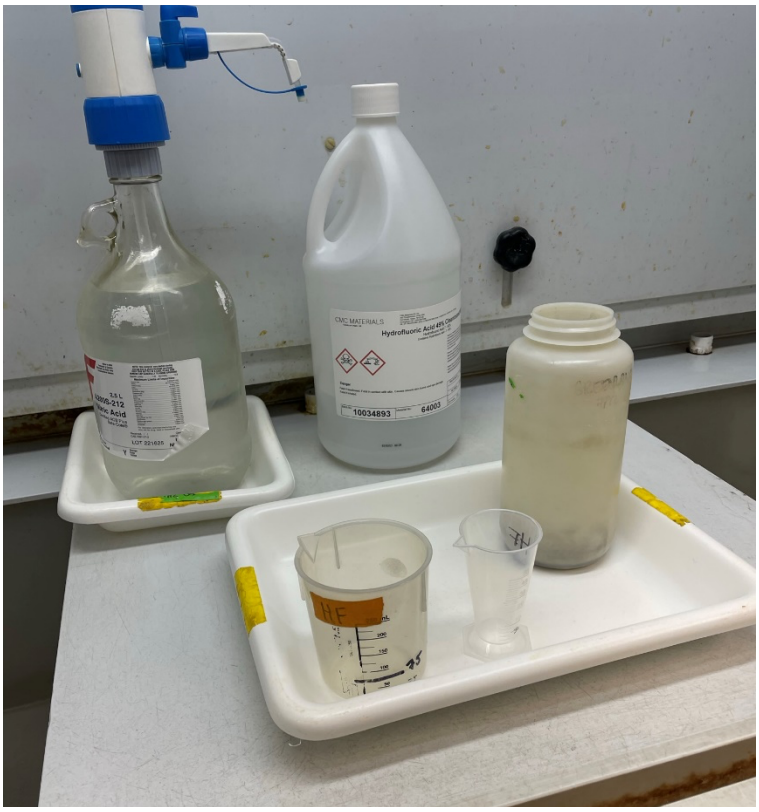
1. Place the 3 by 3 tube rack on the balance and turn it on.
2. Place a 4 liter jug with dry sample next to the balance and place its matching 50 mL centrifuge tube in the middle of the tube rack on the balance. Tare the balance.
3. Cut a 1-inch piece of tape and place it on the counter. Form a funnel with the clean sheet of paper and secure it with the tape. Place the funnel in the centrifuge tube on the balance.
4. Double check the sample ID on the bottle and centrifuge tube match exactly and carefully pour the dry sediment into the funnel. Tap the 4 liter jug to dislodge as much material as possible
5. Remove the paper funnel from the tube and tap gently to make sure no sediment sticks to the paper funnel. Recycle funnel and do not reuse for different samples.
6. Record the weight that the balance reads in your lab sheet.
7. Cap the centrifuge tube and place it back in the large (24 slot) tube rack. Place the empty bottle in the sink.
8. Repeat the above steps for all dry sample jugs.
9. Triple rinse all empty bottles in the sink.

10. **CLEAN UP** Turn off balance and return items where you found them. Wipe down the counter thoroughly with a wet sponge to remove all sediment grains.
11. Examine and compare all 50 ml tubes closely. They should contain mostly pure quartz at this point, with minimal feldspar and mafic material. Work with Paul or Lee to develop a strategy for which samples need more mass, which samples need density separation, and which samples are ready to proceed to the next step.

Weak Hydrofluoric and Nitric Acid (1/4% HF/HNO_3) Etches

(Minimum 3 days to multiple weeks)

Personal Protective Equipment: Thin gloves while setting up and massing samples. Double gloves (thin and thick), rubber smock, goggles, and face shield while handling acids.



For this procedure you will need:

- 1-liter Nalgene bottle (1 for each sample)
- Concentrated hydrofluoric acid (stored under the fume food)
- Small plastic beaker for measuring hydrofluoric acid volumes (typically located on the deck of the chemical hood)
- Plastic graduated cylinder (typically located on the deck of the chemical hood)
- Nitric acid with top-mounted volume dispenser
- Two large spill trays
- Lab tape
- Sharpie

Notes before starting

- These etches are designed to slowly dissolve any lingering non-quartz minerals remaining from the 1% etches. However, they are not very effective at dissolving biotite or muscovite grains. Carefully inspect your samples prior to starting these weak etches. If you see any dark grains (biotite) or shiny, flat grains (muscovite), skip this step for now

and do one of the physical mineral separation procedures (density separation, paper shaking) on the next few pages.

Day 1 (Transferring samples to small bottles, acid addition)

1. Use lab tape to label one 1-liter Nalgene bottle for each sample, placing the label toward the top of the bottle so it will not be submerged. Check each bottle for cracks and make sure it sits flat on the counter. If the bottom of the bottom is distended or you see any cracks, dispose of the bottle in the trash.
2. Carefully pour each sample from the centrifuge tube into its matching 1-liter bottle.
3. Fill all bottles with DI water, cap them, and stage them on the rolling cart.
4. **PPE must be on at this point!** Bring a bottle into the chemical hood and place it in the spill tray.
5. Set the nitric acid dispenser to 2.5 ml and uncap the dispenser to dispense nitric acid into the bottle.
6. Bring the small plastic beaker and graduated cylinder onto the spill tray.
7. Carefully, uncap the hydrofluoric acid bottle and pour a small amount into the plastic beaker. Put the cap back on the hydrofluoric acid bottle and move it out of the way.
8. Pour approximately 5 ml of hydrofluoric acid from the plastic beaker into the graduated cylinder. Pour this volume of acid into the bottle. Immediately recap the HF bottle.
9. Cap the 1 liter bottle tightly.
10. Place the 1 liter bottle in an ultrasound, carrying it with two hands and holding it by the bottle instead of by the lid. You will be able to fit 10-11 bottles into each ultrasound.
11. Repeat steps 4-10 for all samples.
12. Store the hydrofluoric acid back under the chemical hood and recap the nitric acid dispenser.
13. Fill out the ultrasound use diagram on the front of the drying oven. Put your name and bottle contents (1/4% HF/HNO_3) on each ultrasound that you are using.
14. When all bottles are in the ultrasounds, fill the ultrasounds with water until the water line reaches the top row of holes (on the inside surface of the ultrasound).
15. Set the timer on the ultrasound by turning the dial so that the pointer on the outside of the dial is just to the left of the fixed starting position (at the “6-o’clock” position). Turn the ultrasound on by flipping the timer switch to ‘on’ (this switch is inside the timer box, below the dial).
16. The bottles will stay in the ultrasounds for at least 72 hours (and potentially up to a week). To keep the etch going, return once a day as possible to fill the ultrasounds back up with water to the top row of holes and to reset the timer.
17. After the first dilute acid etch, drain off the liquid, rinse and decant the sample, and refill with ¼% acid. Set the 1 liter bottle with the sample and weak acid into an ultrasound and sonicate for at least a week refilling and turning on the ultrasounds daily as possible. The longer samples sit on weak acid, the better the quartz purity.

18. **CLEAN UP** Rinse the plastic beaker, conical graduated cylinder, and spill tray three times with DI water. Wipe down the deck of the hood with a squeegee, then close the sash fully. Make sure all water is off.

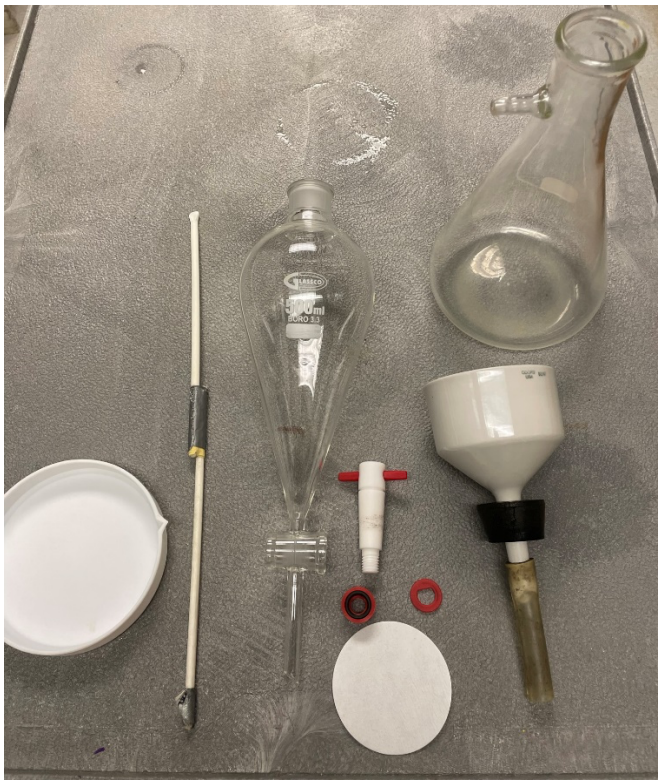
After at least 72 hours:

1. When you arrive in the mineral separation lab, turn on one of the cold water faucets in the hood (NOT DI). Leave it flowing as long as you are dumping acid.
2. **PPE must be on at this point!** Take a bottle out of an ultrasound, bring it into the chemical hood, and set it on a spill tray.
3. Unscrew the cap, give it a few rinses in DI water, and set aside in the spill tray.
4. Carefully, decant as much of the liquid as you can into the sink, trying not to let any sediment pour out. Make sure to pour away from yourself.
5. Fill the jug about a quarter of the way with DI water. Swirl the jug around to mobilize the sediment. This helps ensure that the water washes all grain surfaces.
6. Carefully decant the liquid into the sink, again making sure not to let any sediment pour out. This is rinse number one.
7. Repeat steps 6 and 7 two more times, resulting in a total of three rinses.
8. On the final rinse, carefully pour out as much liquid as you can without losing any sediment.
9. Inspect the sample visually, looking into the bottle. Check to see if there are visible **non-quartz** grains such as dark mafic minerals, light feldspar grains with distinct cleavage plains, or platy, shiny, silvery muscovite mica grains. If the answer is yes, you need to make a choice. For feldspar, another HF/HNO3 acid etch is the best solution. For large amounts of mafic grains, density separation is more efficient. Paper shaking and floatation works well for large amounts of mica.
10. If you want to put this sample through another weak acid etch, fill the bottle with DI water and set the bottle aside for now (on the rolling cart).
11. If you want to dry your sample, place the bottle in the drying oven. Place the washed bottle lid on another shelf in the drying oven next to the bottle.
12. Repeat steps 2-11 for all samples.
13. If you are putting samples back on to weak etches, repeat steps from Day 1 for each bottle.
14. This process can be repeated as many times as necessary to obtain clean quartz.
15. When done with an ultrasound, clear the ultrasound use diagram on the front of the drying oven, indicating to others that you are no longer using the ultrasound.
16. **CLEAN UP** Wipe down the deck of the hood with a squeegee, then close the sash fully. Make sure all water is off.

Density Separation

(30 – 45 minutes per sample)

Personal Protective Equipment: Thin gloves and goggles. The heavy density liquid is non-toxic but is a skin and eye irritant.



For this procedure you will need:

- Glass separatory funnel
- Glass vacuum flask (has a small side tube coming out of the neck)
- Teflon stopcock that fits funnel, with plastic washer and nut that fit on the stopcock threads
- Ceramic Buchner filter funnel with rubber stopper
- Filter paper
- Petri dish
- Plastic stir rod
- High density liquid (LST)
- 1L bottle for each sample
- Lab tape
- Sharpie
- Scrap paper *or* plastic funnel
- DI water squeeze bottle

Instructions

Orient yourself with the density separation chemical hood (in the back right corner of the mineral separation lab). In this hood you will see a hotplate on the right side of the deck, and ring stands on the left side. On the left side of the hood, you will see a knob with a yellow label. This knob turns the vacuum on and off, and you will use it later.

1. Use lab tape to label one 1-liter Nalgene bottle for each sample that must undergo density separation.
2. Set a vacuum flask onto one of the ring stands, and attach the vacuum hose to the side tube (be careful, the side tube is delicate).
3. Place the white ceramic Buchner funnel with the rubber stopper into the top of the vacuum flask making sure it's secure. Place filter paper into the Buchner funnel and moisten it with DI water so it sticks to the funnel and covers all the holes.
4. Find the separation funnel and insert the plastic stopcock into the ground glass hole near the bottom of the funnel. Use the plastic washer and nut to secure the stopper in place. Rotate the stopper so that holes that go through it are 90 degrees to the neck of the funnel, so that no liquid will flow out of the funnel).
5. Place the separation funnel into the ring stand. Adjust the height of the ring as needed so that the bottom of the separation funnel is slightly above the top of the white Buchner funnel.
6. Place the sample and its matching 1L bottle on the cart outside the hood for later use.
7. Carefully pour LST into the separation funnel so that there is about 2-3 inches of LST in the bottom of the funnel (for greater sample masses, use more LST). Ensure that the LST is not draining through the stopcock. Adjust if needed.
8. Pour the sample into the top of the glass separation funnel. Use a clean, dry plastic funnel as needed to move the sample from the 50 ml tube to the funnel so as to minimize the risk of spilling.
9. Using a clean and dry plastic stir rod, carefully and gently stir the sediment until it is well mixed with the LST. This will be easier if you hold the separation funnel with one hand while stirring with the other hand.
10. You may notice some darker grains sinking to the bottom of the vial, but allow your sample to sit for at least 5 minutes to allow for a more complete separation. If the dark grains remain floating, add a few drops of DI water, stir again to mix and wait and watch for the grains to fall. Continue adding very small increments of water (a small squirt), re-stirring, and waiting until most dark grains have settled to the bottom on the funnel.
11. When most denser minerals have sunk to the bottom of the funnel, turn the vacuum hose on (you will hear a hiss as suction is applied through the funnel) and again moisten the filter in the Buchner funnel with DI water.
12. Slowly turn the stopcock in the separation funnel allowing the dark, heavy mineral grains to flow out of the funnel and onto the filter paper. Keep your hand on the stopcock so that you can quickly close it after the dark grains have flowed out. You want to minimize

how much LST flows out during this step, and you want to avoid letting any of the floating light-colored quartz flow out.

13. On the filter you should now see a collection of dark mineral grains. Rinse the grains and the filter with DI water several times under vacuum to remove the LST. Remove the paper filter from the funnel and, depending on your project goals, either keep these grains for further analysis or dispose of the grains and filter. If keeping the dense grains, use a small ziplock bag and label it with the sample name and “dense minerals”.
14. When ready to remove the quartz, **add a new filter paper to the funnel** and wet it with a squirt of DI.
15. Turn the stopcock and drain the remaining LST and quartz through the stopcock. You may need to add some more DI water to ensure all the quartz leaves the separation funnel. Rinse the quartz with DI water.
16. Turn off the vacuum and carefully remove the white Büchner funnel from the vacuum flask and use a spatula to move the quartz into the labelled 1L bottle for that sample without removing the filter paper. Rinse any remaining quartz grains off the paper and into the 1 liter bottle using DI water in the squirt bottle. Dispose of the filter paper after all quartz grains have been removed from it.
17. After density separation, you will need to put your sample back onto a weak acid etch (1/4% HF/HNO_3) to ensure that the LST is completely removed from the grains.
18. Thoroughly rinse (using DI water) the ceramic Buchner funnel and separation funnel between samples to remove any lingering grains and avoid cross-contamination. Make sure to remove the stopcock and clean it paying careful attention to the holes.
19. Repeat the above steps for each sample needing density separation.
20. When finished with all samples, get a large Teflon beaker, and pour the used LST from the vacuum flask into the beaker. We recover and re-use LST, but must evaporate off water that has been added during this process to get the LST back to the correct density.
21. Use lab tape to label this beaker with the following message: “LST evaporating, non-toxic”.
22. Place the beaker onto the hotplate. If there is a need for the LST soon, turn the hotplate on to a low setting to speed up the evaporation process. However, you must be careful with this step, as LST can crystallize if evaporated too much, so you will need to check on the LST beaker frequently; never leave it unattended for more than an hour. If there is not a rush, keep the hotplate turned off and let the water evaporate over the course of a day or two.
23. To test if the LST has returned to its proper density, we have some fragments of feldspar in one of the drawers next to the density separation hood. These have a similar density to quartz, so you can drop one of these into the LST beaker and if it floats up to the surface quickly, the LST is at its proper density. If it floats up slowly or if it sinks, then more time is needed to fully evaporate the water.

24. When the LST is back to its proper density, run it through a clean, dry filter paper in the density separation set-up to remove any residual grains, then carefully pour it back into its original bottle.

Paper Shaking (to Remove Micas)

(30 – 45 minutes per sample)

Personal Protective Equipment: None

For this procedure you will need:

Clean, white 8.5 x 11 sheets of paper
Clean, dry white Teflon Petri dish
Clean, dry plastic funnel

1. Make sure you have a new, clean piece of white paper for each sample.
2. Lay the sheet of paper on a flat surface and sprinkle the surface with a small amount (several grams) of sample, ensuring that all of the grains are in contact with the paper.
3. Hold the paper over a clean Teflon petri dish, slightly bending it into a gentle U-shape and holding it at a low angle.
4. Gently shake the paper so that the sediment grains slowly slide downward. You'll see that the quartz (as well as any remaining feldspar and mafic grains) will roll down the paper into the dish, while muscovite grains will remain stuck to the paper.
5. Continue shaking until all of the non-muscovite grains have moved down into the dish.
6. Hold the paper over the trash and tap it until all the muscovite grains are removed. If you have trouble removing them, use a new piece of paper.
7. Repeat the steps above, working incrementally until all of the sample has been separated.
8. When the sample is done, carefully use a clean, dry plastic funnel to transfer the sample material back into its labeled centrifuge tube.
9. If muscovite grains still remain, repeat the steps above to separate the sample a second time.