

# **The Calvin Miller Foundation's Guide to Mineral Separation**



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## Changes in 5<sup>th</sup> edition

- 1) **All** crushed samples should be stored in zip-lock bags. This includes sample that you've just bashed with a sledgehammer and are about to send through the jaw crusher.
- 2) Promptly remove all cleaned glassware from the rock cutting room and return to the zircon separation lab.
- 3) What to do if you stain heavy liquids if you did not remove magnetite.
- 4) #4 Filter Paper is replaced by Whatman Reeve-Angel filter paper, grade 226, size = 12.5 cm. This new size works faster than the 11.0 cm size paper.
- 5) How to hand-pick grains (see under "Stanford Method" section).
- 6) What to do if the Frantz magnet is not working.
- 7) Chemistry store room (on campus) has face masks and lots of other supplies.
- 8) The gray Corning-brand magnetic stirrer hot plate belongs to Dr. John Ayers. If this hotplate is not available, then use the kitchen-style hot plates in the zircon separation lab.

# Mineral Separation

## Step 1

### Sample to mineral grains

1. Begin with at least ~4kg (~8 lbs.) of hand sample. This must be broken down to ~1 inch squares. Be careful of flying debris and wear eye and hearing protection. A sledgehammer, a solid surface, and some effort can achieve this. Also you can use the screw press in the "*specimen preparation lab*" (*Actually Russ broke this so it's not available*). Scrub screw press spikes down with metal brush and clean with alcohol. Make sure entire area is clean. Also, **any** crushed sample (this means at **any** stage of the steps below) should be stored in zip-lock bags. This will aid in keeping the lab clean and avoid contamination with other samples. **Cleaning steps should be done before and after use, EVERY TIME!!** If using the screw press: place the trash can with a clean garbage bag directly in front of and under the press to catch fragments, also lay computer paper on screw press table for a table cloth (found in drawer in screw press table).
2. Clean the Jaw crusher thoroughly with a nylon brush or rag saturated with alcohol. Make sure the Jaw crusher is set to crush pieces as fine as possible, this makes life easier on the disc mill in part 3. Next, place the <1-inch fragments in the Jaw Crusher, feed slowly! Use just enough to fill ~1/2 the collection bin (~6 pieces of 1 inch sample). Dump this in the trash (forget Freud the attachment can't be that strong). Clean the Jaw crusher thoroughly with a nylon brush or rag saturated with alcohol (the shop-vac works nicely also), Yes clean again-this is not a typo. You have just successfully precontaminated the apparatus, *learn it, know it, live it*; you will be doing this for the disc mill and the water table (**Very Important**). If you want to know why we precontaminate, ask Calvin and he

can explain the statistics of contamination. After running the sample through you should now have pieces smaller than typical breakfast cereal (<pebble size). Make sure you clean the crusher spotless when you are done with a sample! When you start another sample, start at the top and follow procedure again. If the crusher stops running, it is overheated and there is a red reset on the back of the motor; push it. If it stops again in a short amount of time wait thirty minutes to allow the motor to cool and then hit the red reset button. If no action follows pushing reset, check the breaker box in the graduate room.

3. Clean the Disc mill thoroughly with a nylon brush or rag saturated with alcohol (shop-vac also). Next, place the “aquarium gravel size” fragments in the Jaw Crusher, feed slowly! Use ~1/2 the collection bin from the Jaw crusher for precontamination. Dump this in the trash. Once again, Clean the Disc Mill thoroughly with a nylon brush or rag saturated with alcohol (the shop-vac works nicely also). You have precontaminated the apparatus, *learn it, know it, live it (Very Important)!* The Disc mill should give you sand size grains of individual minerals, look under the reflected light scope in the “clean preparation & study lab” to see if independent minerals are being freed or are still oppressed by other grains. Next, sieve the product of the Disc mill with a 500  $\mu\text{m}$  sieve everything that passes goes to the water table (the next step), everything that doesn't goes back through the disc-mill until it is small enough. Make sure you clean the disc mill when you are done with a sample! When you start another sample, start at the top and follow procedure again. If the mill stops running, it is overloaded and you will hear the sound of a circuit breaker being “tripped”. Check the breaker box in the graduate room and flip the breaker back to working position. If you are unsure of how to do this find

Alan, Calvin or a suitable replacement. If it stops again in a short amount of time flip the breaker back to working position again. The motor is strong and can not be burned up like the jaw crusher can, but if the breaker keeps “tripping” it indicates something is wrong with the setup and you can burn out a bearing (bad thing man). If the breaker trips a lot TELL SOMEONE!

## **Step 2**

### **water table**

1. Before moving to the water table make sure you have cleaned and prepared the “*specimen preparation lab*” for the next sample (this means thoroughly cleaning the machinery, cleaning the working space, and sweeping the floor)! The water table is in the basement (dungeon) of the math building; Alberstadt's office. Remove all rings and watches or anything that can scratch the table -- **scratches ruin the table!** Make sure ahead of time all hoses are attached and working properly, if they are loose first use super glue around the exterior of the hose, and glue it into hole in the table. Make sure to use as much glue as possible, applying both on top of and below the table surface. Make sure not to leave any irregularities on the table surface with the glue. When the glue totally dries, spread aquarium sealer around the bottom outside of the hose. Let it sit overnight again to ensure the hoses are strongly secured to the table. Never get anything (glue, epoxy, tape, etc.) on the table's surface, so only affix hoses to the bottom of the table!! Set up the two big white/clear trash cans at the elbows of the table to catch overflow. Place gray bucket with PVC pipe in the center over the drain in the floor. Place the two bigger hoses from the light-catchments (side hoses) into gray bucket. Secure the hoses into the yellow bucket with cinder block pieces, the PVC pipe should extend into floor

drain and the bucket then will act as a sediment trap. Run drainage hose from the pump in the black tub underneath the gray bucket and into the floor drain. Rub table down with horse shampoo\* (see below) paying extra attention to the central white pipe and hopper. The horse shampoo acts as a lubricant to help move the grains, and also to help keep the table clean. Start water running over the table, and try to remove any remaining grains that might still be on the table from the last run. Use the hose directly on the table if desired. Set 3 metal containers under the small hoses at foot of table, with the two center hoses flowing into the central metal container. Set battery charger to select rate charge and keep it on high rate (~10 amps?), outer room (make sure it is plugged in!). A cinder block can be placed on the floor so that the cables from the table to the charger do not stay on the floor where water collects. Turn water, hot & cold, halfway on. You should not adjust flow, valves, or height of the water table until you are experienced using it, and then only with Calvin's or the senior graduate student's permission. Run the table in **reverse**, with the movement turned all the way up. The movement should never be changed, and should therefore always be set all the way up. Add sediment slowly to the hopper. Make sure to wet spots that become dry on the table's surface using the hand water bottle. Periodically, turn on the pump to empty the black tub before it overflows, also if the large white/clear trash cans are filling up, dump them in the black bin and pump the water down the drain. Use the round small bucket to catch water/sediment when dumping the big white cans. When finished force remaining sediment down tracks using hand water bottle or hose with a small amount of water. Clean the grains off the table thoroughly!!!! Dump as much water out of the bins as possible, but don't lose any sample. Hose down table and spray with oxalic acid (mix 2 spoonfuls to 1 full hand

bottle of hot water, shake well to completely dissolve). Add some oxalic acid + water to the samples in the metal bins. Use enough oxalic acid so that the rust comes off the table, usually 2-3 bottles worth. Squeegee floor till dry – do not leave the floor wet. Mildew is already growing on the walls, so keep room as dry as possible. **Turn off battery charger!!** Leave room in good working shape, and every few runs dump the sediment trap outside in the trash bin at the loading dock.

\* The horse shampoo is Orvis brand, and can be purchased at Davidson Farmers Co-op, 3511 Dickerson Pike, (615) 860-4774 for around \$20. Take West End to I-40, go west to I-265, go north to I-65. Take I-65 north to Briley Parkway/Dickerson Pike west (exit 90A). Follow highway 31 west (Dickerson Pike). The store is about ~1 mile down the road on the right.

2. Take metal bins to the “clean preparation & study lab”. Take two sheets of large #1 filter paper, and label one as (sample name) middles and the other as outers. Use two 200-ml beakers and large funnels to strain water from grains - keep middles separate, combine two outer bins. Rinse all the grains out off the bins with alcohol and by running the bin in the ultrasonic bath for ~1 minute. Clean the bins with soap and warm water, rinse with deionized water, rinse with alcohol, and dry. Take funnel paper out, set on watch glass under lamp to dry.

### **Step 3**

#### **heavy liquids**

1. Spread dried middles (middle metal bin) on a table cloth (large piece of computer paper, in screw press drawer or in box in clean lab). Wrap hand magnets (kept stuck to side of the hood in clean lab) in a kimwipe and run over sample about 1/8 inch from grains to remove all magnetite, watch those little grains jump! Tap the magnetite-bearing magnet over sample so any caught minerals that are not magnetic

can fall free. Remove kimwipe over a trash can to get rid of magnetite. Do this till no more grains jump up to the magnet. Failure to do this will ruin the heavy liquids.

**NOTE:** If you forget this step, do not remove enough magnetic minerals, or end up staining the heavy liquids for some other reason, then you can make the liquids clear again by adding a few (2-5) drops of 30% Hydrogen Peroxide to the liquids. Stir the liquids (and/or add more hydrogen peroxide) until it is clear again. If you end up adding more than a few drops of hydrogen peroxide, then you might need to check the density of the liquids, as you could have sufficiently changed the density of the liquids

**2.** Place a large beaker and funnel with #4 small filter paper\*

\*(**NOTE:** #4 filter paper replaced with Whatman Reeve-Angel filter paper, grade 226, size 12.5 cm) under the separation funnel, under the hood with the separation funnel's valve off. Using glove and small funnel add heavy liquid to fill the funnel over half full (or half-empty depending on how your day is going). Leaving valve off, add sediments slowly (~1 teaspoon), stir with glass rod vigorously, then wait ~10 minutes until heavies settle out, should be mostly dark (red, gray, or black). Strain off heavies by opening valve and closing valve quickly. Spray some DI water on the sample in the filter paper to clean heavy liquid off of grains. The heavy liquid/DI water mix should all be saved, and stored in a bottle marked "heavy liquid + water". You must recover all heavy liquid diluted by water (no matter how much water you added). While heavy liquidizing, use one beaker to recover pure heavy liquid and one beaker to recover diluted heavy liquid. Continue until sample has all been processed. If surface becomes choked with lights you can add more

liquid or drain off liquids and lights into another funnel and then repeat step 2 up to this point. When finished, filter off lights and liquid into #4 filter paper (Whatman Reeve-Angel filter paper), and clean all glass thoroughly. **All heavy liquid must be recycled, as it is very expensive.** Rinse heavies into a small beaker, and bathe in ultrasonic bath with DI water for ~1 minute. Drain off cloudy water and repeat until the water comes out of the ultrasonic bath clean and not cloudy. Be careful not to pour off floating grains that might be on the surface!! If grains are floating on surface after running in ultrasonic bath, add a drop or two of alcohol to break the surface tension. Once water comes out clean, run one more time in ultrasonic bath, this time with alcohol instead of DI water. Place sample on filter paper, drain, place on a watch glass under lamp to dry.

**3. Heavy Liquid Recovery:** Heavy liquid should be checked periodically for density by placing small density standard rock chunks in liquid (in envelope on shelf). After about 750 mL of diluted heavy liquid is saved up, it is time to recover the heavy liquid! (This method will also eliminate the blue color of heavy liquid altered by iron). Begin by pouring about 400 mL of diluted heavy liquid in a beaker and place it on a hot plate (at about a setting of five – this is for the gray Corning-brand hot plate that belongs to Dr. John Ayers). In addition drop the small magnetic stirrer into the beaker and adjust the stirring speed on the hot plate. If Dr. Ayers' hot plate is not available, you can use the other kitchen-style hot plates in the separation lab. Place a density standard in the beaker (chunk of rock of known density) equal to the density you want the heavy liquid. The density standard will sink. Monitor the evaporating liquid and continue to pour additional diluted heavy liquid in the beaker as more evaporates and stir every so often. When the density standard is between floating and

sinking, the liquid is at the correct density (if its floating, you can add water until the standard reaches the point of buoyancy). Turn off the hot plate and allow to cool, adding some water while stirring during cooling to make up for evaporating water (but not too much water, or you'll have to heat it up again). When the heavy liquid is cool enough so that you can hold the beaker with your hand, funnel it through #4 filter paper (Whatman Reeve-Angel filter paper) and place it back in a heavy liquid container.

## **Step 4**

### **Frantz**

- 1.** Clean the Frantz with air hose and with alcohol and a kimwipe before use. Use settings from paper instructions in drawer below Franz for whatever mineral you are trying to get. Typically 3 separations are done, the first at 0.6 amps, the second at 0.9 amps, and the final at 1.2 amps. After each run, inspect the separates under the microscope and identify all minerals. If you cannot ID minerals by name, at least do so by description (i.e., dark opaque mineral, typically broken grains without nice prismatic faces). For zircon separations, re-run the non-magnetic separate at a higher setting until all magnetic minerals are separated and zircon and apatite are the only non-magnetic minerals remaining. Bottle and label each separation in small bottles located on shelf to the right above Frantz. Make sure the Frantz is cleaned after each use - air hose on hood followed by an alcohol bath.

2. **NOTE: IF FRANTZ MAGNET IS NOT WORKING**, but the motor is still working - check the black "reset" button on the back of the Frantz (where the power cords plug into).

## **Step 5**

### **Hand picking and mounting of grains**

#### *UCLA Method*

- 1a. Clean the glass plate with Windex and place a ~4 inch piece of double sided tape on the plate. Make sure there are no bubbles in the tape. Next place the sample on a piece of weighing paper adjacent to the tape, and put a small crease on one side of the weighing paper so that you can pour grains back into their bottle when done. Pick minerals with probe based on properties described by Calvin; the probe works best with a little nose, ear, or hair grease on it, Ask Calvin for a demonstration. Place chosen grains in a row on the tape ~25 to 50 grains per row, don't forget standards every few rows, ~4-6 standards is a nice row, spaced out evenly along entire row of unknowns. Place grains in patterns that you will be able to recognize while using the SEM or IMP. It is a good idea to NOT make the mount symmetrical, and to also include miscellaneous grains of apatite or other minerals (pyrite is also good) in the mount (standard 1" mount) to help find your way around. Sketch all grains in mount.

## *Stanford Method*

**NOTE:** For the last few years we have been pouring the mounts at Stanford, not at Vandy. Therefore the sections below on preparing the mount are rather obsolete. However, the sections on hand-picking the grains are still relevant. Calvin may have other tips, so you should consult with him the first time you hand-pick grains. This is what I have found most successful.

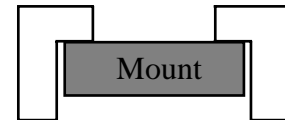
1. Thoroughly clean the stereomicroscope, table, glass plate, and any other tools you plan to use.
2. Select which ever fraction from the magnetic separation yields the most zircons.
3. Fold the 4 edges of a large sheet of weighing paper and place a clean, large (75x50 mm) microscope slide in the middle of the weighing paper.
4. Dump a small amount of your separate onto the clean glass microscope slide (I find the large, 75x50 mm size slides easiest to use).
5. Take a piece of smaller weighing paper and fold it in half. You will deposit the individual zircons on this paper. I find it useful to put this weighing paper on another large glass microscope slide. I then put the glass slide and weighing paper in a watch glass. Soak the crease of the weighing paper with alcohol. Periodically re-soak the weighing paper as the alcohol evaporates.
6. Look at the sample under the stereomicroscope and use the glass rod with a thin metal tip to sort through the sample. To extract zircons, gently wipe the metal tip of the glass rod on your forehead, nose, or through your hair (this is to get a little grease on the metal tip. Your grease makes the zircons stick to the metal). Transfer the zircons to the alcohol-soaked weighing paper. The zircons will come off the metal rod when it touches the alcohol. Try to deposit zircons in the same general area each time.
7. Wipe off the metal tip with a Chem-wip to remove any grains, grease, or alcohol.
8. Sort through the sample and when you are ready to move more zircons, re-apply some grease to the metal tip to pick up zircons again.
9. You may want to double check with Calvin, but in most cases you will want to collect between 60-100 zircons.

**(Everything below is somewhat obsolete as of Summer 2004)**

- 1a.** Clean the large glass plate with Windex and place a ~1.5 inch square piece of double sided tape on glass plate. Make sure there are no bubbles in the tape. Keep tape backing clean, you will use it to make a mask in the next step. Next move the large glass plate to the side and place the sample on a small glass plate (~2"x2"). Pick minerals with wire probe based on properties described by Calvin; place selected grains into a large pile (or

several small piles) that are made up of ~75-200 grains depending on size and abundance within the sample.

- 1b.** Place epoxy mount (metric 1" mount; 2.54 cm) in the center of the tape and trace the inside circumference with a fine point Sharpie™ marker. Remove mount from tape. Take tape backing and cut a ~0.75x3 mm strip from the center. This will serve as mask to place each sample of zircons and keep them separate from other samples. Prior to placement of samples, think about how many samples you want to put on this mount, you can put ~6 samples on each mount including on standard sample. Create a pattern of sample rows that will help you quickly recognize the orientation of the mount later. Place mask over tape and carefully scoot the pile of grains into the slot. Carefully fill the slot in with crystal trying to place them as flat as possible onto the tape. When finished, pick up mask and move it to the place where the next sample will be placed. Be sure to leave ~1.5 cm between the circle and the edge of the mount as the mount holder covers part of the top of the mount (Figure 1).



**Figure 1-** Shows relationship of mount holder and mount during analysis.

- 2.** Place plastic mold around rows of grains (choose correct mold, Cameca and SHRIMP molds are slightly different), then in small mixing cup mix 7.5 parts (cc's or cubic ml) of base (resin in large white bottle) with 1 part activator. This is plenty of epoxy for 2 mounts. Stir easily till well mixed (at least 1 minute) and try to avoid getting bubbles in the epoxy. Pour a little epoxy over grains, check under scope for bubbles, touch bubbles near grains with probe and they will float to the top. Fill the mold to around a third full, cover with weighing paper or plastic mixing cup and let set overnight.

3. Remove mold the next morning from the tape and from the mold. Place the mold in the small 110° oven in the geochem lab for a minimum of 2 hours. 5 hours is common. The harder the epoxy gets the less mounding you will have when polishing the mount.

## Step 6

### Polishing

- \* **Don't** polish the mold at Vanderbilt if you are using the SHRIMP, their facilities are much better than ours for this purpose and they make fun of us if we do a crappy job.
1. Start with 240 grit and polish the back of the mount (the side with no zircon) until it is flat, smooth out on the 320 or 400 paper.
  2. Start with 240 grit paper (next to sink in John's lab) on the zircon face of the mount for a maximum of 30 seconds, check with reflected light microscope at 15 seconds—to check switch between reflected and directed light on the microscope in John's lab where reflected light will show you the surface of the zircon, and directed light will show you the zircon at its maximum width in the epoxy, when the surface image matches the directed image, you are at the middle of the zircon (be careful, if you accidentally polish through the center you'll be fooled into thinking you haven't polished enough). While polishing, apply moderate pressure, but don't push down hard or with barely any pressure.
  3. Polish on 320 grit for a maximum of 30 seconds, check on scope
  4. Polish on 400 grit at 20 second intervals checking with the scope until you are pretty close to the center (you may need to polish it with 1000 grit before viewing)—beware of zircon plucking, keep an eye for lost and exploited zircons.

5. Ultrasonic bathe the sample for a few minutes in water before moving on to 1000 grit.
6. Polish on 1000 grit for approximately 5 minutes, check halfway through on scope—the 1000 grit will still remove some material but not much. Continue until you are at the center of a majority of the zircons and all large scratches are removed.
7. Ultrasonic bathe again before moving to the 1.0 pad.
8. Polish on 1.0 pad for a few minutes, put a small dab of 1.0 polish paste on the polishing pad for 1.0 (make sure it is the 1.0 pad!) and a few drops of the lubricant—the 1.0 will remove scratches you shouldn't be in danger of plucking zircons. Continue until you've removed most scratches (about two songs on the radio in polishing time).
9. Ultrasonic bathe again before moving on to the 0.3 pad.
10. Polish on 0.3 for a few minutes, put a small dab of 0.3 polish paste on the 0.3 polishing pad and a few drops of lubricant. Continue until you have a smooth unscratched surface (again listening to 2 songs on the radio is usually enough polishing time).
11. Wash off the 1000 grit, 1.0 polishing pad, and 0.3 polishing pad in the sink with deionized water, let dry, wrap in saran wrap, and place back on the shelf.

## Materials

1. Separation Funnels: Catalog Number: 10-436-1D (Fisher?), 1000 mL separation funnel with Teflon stop cock.
2. Double Stick Tape: Cam and Barkley 615-831-5213, 3M Tape, Product #9425, 3" core, 4" wide, clear double stick tape.
3. #1 Filter Paper: Available in chem stockroom, 185 mm diameter.
4. #4 Filter Paper: Re-order from Fisher catalog, 110mm diameter. (**NOTE:** replaced with Whatman Reeve-Angel filter paper; grade = 226; size = 12.5 cm (this works faster than the 11 cm); catalog # 5226-125; reorder from Fisher catalog, product # 5226-125)
5. Heavy Liquids: LST, density 2.85, reorder from Geoliquids 1-800-827-2411.
6. Epoxy: [http://www.struers.com/products/Mounting\\_products/consumables.htm](http://www.struers.com/products/Mounting_products/consumables.htm), reorder epoxfix cold epoxy mount system.
7. Polishing materials: re-order from Fisher catalog.
8. Oxalic Acid: reorder from Sigma-Aldrich Chemicals, product # 24,753-7 (oxalic acid dehydrate, 99%) (note: do not worry about high-purity grade, which is much more expensive)
9. Face masks (and many other supplies) are available in the Chemistry Store Room, located on the 3<sup>rd</sup> floor of Stevenson, in the main Chemistry building.