## Grinding & cleaning (for powders)

If the sample of interest was slabbed, the saw marks must be ground from the sample with coarse (60 to 100 grit) silicon carbide paper or grit on a steel wheel. Following careful rinsing, the samples are immersed in deionized water in clean glass beakers and place in an ultrasonic bath or probe for 15-30 minutes to efficiently remove adhering grit. The samples should then be thoroughly dried in a 100°C oven for several hours prior to rough splitting.

## Rough splitting (for powders)

Cleaned rock fragments or slabs are comminuted to <2cm by wrapping them in several polyethylene plastic bags and careful hammering. Care must be taken to minimize or avoid contact of the hammer or baseplate with the sample.

## Jaw-crushing (for powders)

The small ceramic jaw crusher is used to further comminute bulk samples to the sub-centimeter-size necessary for powdering in the Shatterbox. Feed material must be <2 cm in diameter following from careful sledging of the bulk sample. The jaw crusher is first cleaned by rubbing the plates with a coarse grit SiC adhesive paper on a flat wooden stick (e.g. a paint stirring stick); use a cotton-tipped stick to remove any residue from the joins between the ceramic plates. After blowing out with compressed air, start up the crusher and pass several rounds of coarse bull quartz through the plates. Again use a cotton tipped stick to remove residual quartz from joints while blowing with compressed air. Finally start up the crusher and pass several rounds of excess sample through the plates to pre-contaminate the plates—discard this sample and blow off excess. The jaw crusher is now ready to crush your sample. Clean through the quartz step when your finished with the device.

## The Shatterbox (for powders)

The Shatterbox comprises an alumina ceramic lined sample chamber clamped into a holder sitting on an eccentric spinning shaft which spins a ceramic puck inside the chamber. Collisions efficiently grind the sample material to a fine powder with 10-15 minutes for most rocks. The Shatterbox is first cleaned with a brush, deionized water and soap, rinsing well. Then several grams of acid-washed quartz chips are added to the shatterbox with a few milliliters of deionized water, the chamber is clamped into the holder and spun for approximately 10 minutes. The resulting slurry is washed from the chamber, it is rinsed with deionized water and then clean methanol, wiped dry with kimwipes and then thoroughly blown dry with compressed air. Sample can then be loaded into the chamber. It is preferable for large samples to precontaminate the chamber with a small amount of sample which is discarded. Cleaning follows.

More information on the processes listed on this page, including the use of the Shatterbox can be found in the ‘Sample Preparation for Geochemical Analysis Handbook’.

## Jaw-crushing (for mineral separations)

The large Bico-Braun “Chipmunk” steel jaw crusher (general accessory mineral separation for geochronology) or the small ceramic jaw crusher (bulk mineral separates for tracer isotope work) are used to comminute bulk samples to the centimeter-size necessary for disk-milling. Feed material must be <5 cm (< 2 cm for ceramic crusher) in diameter following from careful sledging or splitting of original samples. The use of the ceramic jaw crusher was previously described.

The Chipmunk crusher is used as follows:

1. Start up the single to three phase converter in the front corner of the garage, to the right of the garage door. Start up the dust collection bin and ensure the vacuum entrances are in proper position near the Chipmunk.
2. Drop the clean moving plate assembly into place, latching the top onto the steel bar.
3. Move the jaws fully apart by turning the large knob at the bottom right of the jaw crusher.
4. Start up the Chipmunk, making sure the jaws rotate smoothly (it will probably squeak like a chipmunk...). Make sure the collection pan is in place below the Chipmunk!
5. Feed sample chunks into the top of the Chipmunk, using a small piece of cardboard or masonite to cover the inlet as soon as you feed the sample in—this will stop the small chips that will spall off and fly back up the feed slot!
6. Feed all of the sample through, keeping an eye on the sample in the collection pan—make sure it doesn’t spill over the sides.
7. Move the jaws fully together by turning the large knob at the bottom right of the jaw crusher.
8. Feed all of the sample through again to produce a finer crush ready for the disk mill.
9. To begin cleaning the Chipmunk, remove the moving steel plate assembly from the Chipmunk by lifting straight up— a light tap on the top handle with a hammer might be necessary to free up the assembly after prolonged use.
10. Brush, vacuum and blow clean with compressed air the interior of the Chipmunk and the collecting pan. Pay close attention to the joints between the plates, cleaning each with the long steel brush.
11. Clean the moving and stationary steel plates with a rotary brush on an electric drill, and set aside for the next user.

## Disk-milling (for mineral separations)

Disk milling can be done either with steel plates (general accessory mineral separation for geochronology) or ceramic plates (bulk mineral separates for tracer isotope work). The latter are more brittle and are only appropriate to special applications.

1. Start up the single to three phase converter in the front corner of the garage, to the right of the garage door. Start up the dust collection bin and ensure the vacuum entrances are in proper position near the disk mill.
2. Fasten the clean rotating plate to the rotor with the three bolts. Loosely fasten the clean stationary plate to the side of the disk mill with the two large bolts, then raise the end of the mill up to vertical, swing the two clamps on either side of the end of the mill up and tight into place.
3. Using the plate alignment tool, align the plates carefully prior to final tightening of the two bolts.
4. Set the gap between the rotating and stationary plates so that you can just fit your thumbnail between the two plates. Rotate the plates by hand to ensure the gap is consistent.
5. Shut the upper door of the disk mill, and place the collecting pan beneath the grinding plates. It helps to slide a 16 oz. plastic tub lid under the collecting pan to snug it up tight under the lip of the lower mill exit. This will minimize the amount of sample loss out the sides.
6. Start up the disk mill, and then slowly feed jaw crushed sample through the entrance port. After about 16oz., stop feeding sample in, allow it all the pass between the grinding plates and then check the crushed sample in the collection pan.
7. Dump the milled sample into the large 500 micron sieve, assemble and start up the sieve shaker. Approximately 70-80% of the sample should pass through the sieve in a few minutes. If less than that passes through then tighten up the gap between the disk mill plates slightly. If more than that passes through then widen the gap between the disk mill plates slightly.
8. Replace the collection pan, and continue to pass the rest of the sample through the disk mill in increments, adding them to the shaker sieve. You will need to recycle the coarse fraction from the top of the sieve back through the disk mill a couple of times. You’re finished when 90% of your sample passes through the 500 micron sieve. The remaining coarse fraction can be saved or discarded.
9. Begin cleaning the disk mill thoroughly by blowing compressed air into the entrance port with the pan in place and the doors closed. The majority of dust should go into the collection hopper. Then open up the disk mill and use the ShopVac to vacuum up the majority of dust. Finally, use compressed air to blow out all the nooks and crannies of the device thoroughly, while using a steel brush on all the surfaces.
10. Clean the disk mill plates with a rotary brush on an electric drill, and set aside for the next user.

## Water Table (large lithic samples)

1. Clean the dry table by blowing with compressed air and brushing. Adjust the tilt of the table, start the flow of water, and then brush down the wet table again to ensure cleanliness. The water flow rate should be sufficient for water to flow regularly out of all of the ports on the upper perforated tube, except the last (upper) port, which should drip. During cleaning pay particular attention to the exit spigots at the end of the table; also make sure to clean the sample feed trough!
2. At this point go outside around the corner of the garage and make sure that the water exiting the table is draining out into the yard, and not pooling around the foundation.
3. Place the sample collection cups (tall 16 oz containers) beneath the two exit spigots (T1 and T2) at the left end of the table; if you’d like to save all of the crushed sample also place a container under the third exit spigot on the front of the table (T3) and start the shaker. Start the shaker with the speed all the way up (full counterclockwise) and then turn the speed down to the black mark on the housing; this will avoid putting the table into a violent jarring harmonic mode.
4. Place your sample in the (clean) vibrating feed trough and align so that the sample flow intersects the feed water spouts to form a slurry as its hits the table.
5. Adjust the sample feed rate to optimize removal of light minerals off of the front of table and concentration and transport of heavy minerals to the end of table and into the T1 tub. Intermediate density minerals will end up in the T2 tub, while light minerals will end up in the T3 drain (for collection or discarding). Because the separation of the heavies from the last lights at the end of table is inefficient, recycling of the T1 tub back onto the table can further concentrate the heavy minerals for very large samples. Be aware that all the riffles of the table should be filled for the most efficient separation and that very small or flat grains (like baddeleyite or molybdenite) will commonly float off the table early and be lost.
6. When all sample has been fed onto the table, allow the last of the sample to work its way down the table and into the tubs, prior to stopping the shaker, removing the sample tubs, draining excess water and transfering the samples to aluminum pie plates for drying under the heat lamps.
7. Clean the table carefully with water and a brush, and then turn off the water supply and dry the table completely with compressed air and paper towels.

## Washing (small lithic samples)

Place the disk milled sample into a large glass beaker with a small amount of water and a little dish soap, and swirl into a slurry. Fill the beaker up to the top with water and then let settle; general rule of thumb is that a 30 µm zircon will sink 30 cm in 3 minutes, so to be safe after 4-5 minutes, pour out the liquid containing the clay fraction. Repeat the filling and decanting process until no suspended clay-sized material remains (6-7 times?). Decant as much of the final water as possible (tapping the beaker helps to settle and dewater the sample) and set under a heat lamp to dry.

## Clay Separator (bentonites)

Efficient washing of clay-rich samples (i.e. bentonites) without loss of crystalline material (e.g. zircons) is complicated by the flocculating properties of clays, which tend to suspend even heavy minerals. A combination of ultrasonification, mixing and washing removes the clays without loss of crystalline material.

Lithified clays may be jaw-crushed and disk-milled as described above, and then mixed with water for form a slurry.

Unlithified clays should first undergo thorough wet disaggregation using a mortar and pestle or an electric blender in a large plastic bowl or beaker; this is an extremely important step that frees coarse crystalline grains from their clay matrix. It is best to soak the clay sample in a large (4L) beaker of water for a few days prior to disaggregation, to allow the clays to swell and self-disaggregate.

To use a mortar and pestle, dump a moderate amount of wet clay into the mortar, and gently mix to a paste with the pestle, adding water as necessary to achieve a smooth slurry. When using an electric blender, add clay and water in roughly 2:1 proportions to make a thick slurry. Remove any lithic fragments (e.g. carbonate nodules, fossils) by hand, rinsing the clay away and setting aside for later drying and storage in an appropriate sample container. Dump the slurry into an 8” brass 1 mm sieve that has been place in a large funnel on top of a large (4L) plastic pitcher. Using a squirt bottle and gloved fingers, gently wash/push the clay slurry through the sieve, keeping back any lithic fragments. Repeat the process until all sample has been put through the sieve. Place the sieve upside down on a piece of paper under a heat lamp to dry while gently brushing the lithic fragments onto the paper. Collect the lithic fragments into the appropriate sample container for storage.

After disaggregation, the slurry is ready to be placed into the first-stage pitcher of the clay separation device, consisting of two interconnected large pitchers on stir plates and a high-energy ultrasonic probe. To assemble the clay separator:

1. Find the two 4L pitchers with predrilled holes: one has a single upper hole and another two holes. Thread a black bulkhead connector through the upper hole of each pitcher, making sure there are rubber O-rings on each side of the pitcher wall, and screw the retaining nut on finger-tight.
2. Find the grey bulkhead connector and thread it onto the lower hole of the first stage pitcher, making sure the flat rubber gasket is in place, and screw on the retaining nut finger tight.
3. Assemble the floating stir-bars and place one in each pitcher.
4. Set the first stage pitcher on the upper stirplate (if disassembled, the upper stir plate rests on top of the large plastic pitcher, itself on top of the base of the ring stand; the hotplate is threaded through the stand rod). Slide the black plastic retaining block, then the ultrasonic horn stand onto the rod, then snug them down onto the top of the pitcher and tighten the set screw on the horn stand. The black bulkhead connect should point to the left and the whole assembly should be pretty solidly held in place.
5. Set the second stage pitcher on the lower stirplate to the left of the first stage pitcher assembly, again with the black bulkhead connector pointing to the left.
6. Connect the straight cut side of the longer piece of large bore tygon tubing to the first stage pitcher black bulkhead connector, and place the diagonal cut end into the second stage pitcher, toward the lefthand side. Connect the straight cut side of the shorter piece of large bore tygon tubing to the second stage pitcher black bulkhead connector, and place the diagonal cut end into the sink (feed through the ringclamp on the side of the faucet).
7. Connect the water feed line to the gray bulkhead connector on the first stage pitcher. Note that this feed line has a quick disconnect for cleaning and a one-way valve on the end.
8. Now that the clay separator has been assembled, turn on the upper stirplate to a setting of about 5.
9. The sample slurry can now be poured into the upper first-stage pitcher, rinsing out the last of the sample with a squirt bottle. About 1 L of slurry can be processed at once, depending upon the amount of crystalline material it contains. Make sure that the stir bar continues to rotate in the slurry.
10. Top up the first-stage pitcher with water up to the overflow bulkhead connector, then place the clean ultrasonic horn through its holder into the liquid. Turn on the ultrasonic generator, set the amplitude to 50%, and program the horn to run continuously for 1 hour. Press start to begin the first stage of sonic treatment, which will disaggregate the flocculated clay particles.
11. After an hour has passed, turn on the lower stirplate to a setting of about 4. Then switch the three-way valve on the faucet to the straight-up position to send water to the upper first stage pitcher (the flow rate has been set by a needle valve to approximately 250 ml/minute—do not adjust). Program the ultrasonic horn to pulse 5 seconds on / 5 seconds off, for 6 hours. Press start to begin the second stage of sonic treatment, which will float the clay particles out of the two pitchers and down the drain, leaving behind any crystalline material.
12. After several hours check and see if all of the clay has washed out of the first-stage pitcher:
    1. if not, continue with pulsed sonication.
    2. if so, and there is another aliquot of slurry to add: turn off the water feed; turn off and remove the ultrasonic horn from the first stage pitcher; loosen the set screw on the horn stand and remove the ultrasonic horn stand and black plastic retaining block; gently tip the upper first-stage pitcher to drain half of the water out of the first-stage pitcher, making room for the next batch of slurry which can now be poured into the upper first-stage pitcher, rinsing out the last of the sample with a squirt bottle; replace the black plastic retaining block and horn stand, and return to step #10.
    3. if so, and there is no other sample to add, then use a stirring rod to stir up the sediment on the bottom of both pitchers, to free up any clay lodged in the sediment; allow this clay to wash through the separator.
13. Once all of the clay has been washed out of the sample: turn off and remove the ultrasonic horn from the first stage pitcher; loosen the set screw on the horn stand and remove the ultrasonic horn stand and black plastic retaining block; gently tip the upper first-stage pitcher to drain enough water out of the first-stage pitcher to disconnect the large bore tygon tubing, placing it in the sink for washing. Do the same with the second-stage pitcher.
14. Carefully drain most of the water from the second stage pitcher into the sink, being careful not to pour out any grains. Remove the first-stage pitcher from the stand, disconnect the gray bulkhead connector (a small amount of water will come out, but the one-way valve on the water feed will stop most of the water), and again carefully drain most of the water from the first stage pitcher into the sink, being careful not to pour out any grains.
15. Transfer the contents of both pitchers to a new pitcher (with no holes), fill with water and allow the grains to settle for 3-4 minutes, after which you can carefully pour off any residual clay fraction remaining suspended in the water. The grains will make a pretty obvious front as they move down the side of the pitcher, in contrast to suspended clay; just make sure not to pour out the grains! Repeat this washing as necessary until all the clay is gone (e.g. the clear water above sediment after 3-4 minutes of settling).
16. Take the pitcher of wet sediment down to the acid lab (MG-138), and under the fume hood, add 500 ml of 10% formic acid to the sample. Allow any carbonate in the sample to react with the acid for a few hours, periodically swirling the sample and checking for reaction (bubbling). Once the sample stops reacting, or after no more than 6 hours, top up the pitcher with water, allow to settle 3-4 minutes, and pour off any suspended clay fraction freed up by the acid treatment. Repeat this washing at least 3 times, or as necessary until all the clay is gone (e.g. the clear water above sediment after 3-4 minutes of settling).
17. Return the pitcher of wet, acid-washed sediment to the dark room, rinse the sample into a large glass beaker, carefully pour off all excess water, and leave under a heat lamp to dry. Once dry, the sample is ready for magnet separation.

## Hand Magnet

After the table- or hand-washed sample has been dried under a heat lamp, the highly magnetic materials (e.g. steel from disc mill, magnetite or pyrrhotite) are separated with a plunger-type hand magnet. Spread a small amount of sample on a clean sheet of paper and pass the hand magnet with plunger depressed through the sample. Lift from the sample and tap the magnet to free loosely bound grains, remove the magnet to another clean sheet of paper and release the plunger to free the magnetic grains. Repeat this process with the sample aliquot until no further magnetic material is picked up, set aside the non-magnetic residuum, and repeat the process with successive aliquots until the whole sample has been processed. Store and label the magnetic fraction as “hand mag” or “filings”.

## Frantz magnetic separation (for accessory minerals)

The Frantz magnetic separator is used in accessory mineral separation to initially take out the most magnetic material (e.g. oxides, olivine, garnet, altered ferromagnesian minerals) and thereby reduce the volume of sample to go through heavy liquids. Target minerals like xenotime and monazite have moderate magnetic susceptibilities (paramagnetic at ~0.4-0.7A), titanite slightly lower (paramagnetic at ~0.7 to 1.0A) and rutile, apatite and zircon very low (paramagnetic at >1.0 to diamagnetic). Therefore different separation strategies can be developed to target each mineral. However the best generic method for complete sequestration of target heavy minerals follows.

Always clean the Frantz before and after each use. To clean the parts, blow out rigorously with compressed air. Never touch the tray or insides of receptacles with fingers, as finger oil will cause grains to stick. If any parts are soiled with finger grease, they can be wiped with acetone and then blown dry. Be sure that the magnet is turned off for a minute before you clean it, to allow the magnetic field to dissipate. Use a large kimwipe folded several times to clean between the magnet pole pieces. Also, wipe off other surfaces on and around the machine. Place clean paper under the chute area and under the cups to catch grains that might accidently fall.

For the initial split, operate the Frantz in a paramagnetic position, i.e. with the entrance (right side) of the chute within the pole pieces of the electromagnet, and the split (left side) of the chute below the pole pieces. Set the side (right to left) tilt to a ~7° angle, and the forward (back to front) tilt to a +20° angle (toward the front). Turn on magnet and with illuminated knob set current to about 0.30A.

Position the feed tube to dispense onto the far side of the chute (relative to the operator), as close to the chute as possible to avoid grains jumping over the barrier before entering the magnetic field. Before turning on chute or feed, make sure that they are turned to a low setting. Turn on chute first, then feed. Adjust the levered door and vibration of the feed tube and chute so that grains flow in a moderately fast stream, with the nonmagnetic grains falling out efficiently near the top (right side) of the chute. Check periodically to refill feed and to make sure that magnetic grains aren't clogging passage, that stream is still satisfactory and that cups aren't overflowing.

Put products into labeled tubs, one marked mag @ 0.30A, 20° (or m<0.3A/20°) and the other nmag @ 0.30A, 20° (or m>0.3A/20°). Take the nonmagnetic portion to heavy liquids.

## Frantz magnetic separation (for other minerals)

The Frantz magnetic separator can be used to finely split other mineral fractions, using the same general procedures. Consult the magnetic susceptibility charts for relative susceptibilities. Generally minerals exhibit the following decreasing susceptibilities: olivine and orthopyroxene, garnet, biotite, clinopyroxene, muscovite, feldspars, quartz.

## Heavy Liquids: LST (Lithium-based Tungstate)

**Notes:** The purpose of heavy liquids is to separate dense grains, such as zircon and sphene, from the bulk of lighter grains such as quartz and feldspar (LST); separation of accessory minerals from micas and pyroxenes can be done using halogenated hydrocarbon heavy liquids (e.g. methylene iodide). LST and the other tungstate-based liquids are non-toxic, therefore it is not necessary to wear rubber gloves during their use; however, since you shouldn’t ingest LST, and it dries out your skin, it is recommended to use vinyl gloves during the process. Use only deionized (DI) water when rinsing glassware of LST. If there is a possibility that grains could have gotten into your pure heavy liquid flask, refilter the liquid before putting it back into its storage bottle.

**Equipment to be used**: separatory funnel, teflon stopcock with o-ring and retaining nut, two filtering flasks, two filter paper cones made from Whatman 114 (25 micron), two conical funnels with #8 rubber stoppers, a plain conical funnel, a stirring rod and a small beaker to put it in.

1. Rinse washed glassware with acetone before use and blow dry with compressed air (or a blow dryer). Fit the teflon stopcock into the separatory funnel and fasten using an o-ring and plastic retaining nut. Turn stopcock in separatory funnel to the closed position, and place on a ring stand.
2. Prepare a cone from Whatman 114 filter paper by folding in half twice. Place the filter paper in a conical funnel with stopper in a 1000 ml “Pure LST” filtering flask. Place the funnel and flask assembly under the separatory funnel. Fill the separatory funnel with an amount of LST necessary for the size of the sample, usually up to the waist of the funnel (e.g. for a 500 ml funnel, one small bottle is usually sufficient). There are plenty of sizes of funnels to choose from to optimize separation and minimize LST use. Once the LST is funneled into the separatory funnel, rinse the used upper funnel with DI water into a 1000 to 2000 ml beaker for LST reclamation, or suspend it over small beaker for later use.
3. Pour your sample into the separatory funnel with a tube fashioned out of weighing paper. Stir the mixture thoroughly and allow heavy minerals to settle; continue to stir occasionally, always placing the stirring rod in a beaker when not in use. Wait for grains to settle between stirrings. Repeat process as many times as necessary (at least thrice). During this process, using a blow dryer to gently warm the LST in the separatory funnel can aid in settling by lowering the LST viscosity.
4. Assemble the inlet and outlet tubing on the vacuum pump, and attach pump inlet hose to the pure LST filter flask. When all the heavy grains have settled, release them by opening the stopcock carefully then closing; quickly repeat the opening and closing turning the stopcock the opposite direction. Turn on pump to suck pure LST down into the filtering flask. When all the heavy grains are in the filter paper, let the pump run for a little longer to remove as much pure LST from grains and filter paper as possible; it helps to lightly press the filter paper against the walls of the funnel.
5. Remove the filter paper with the heavies from the pure LST flask, place it in the second conical funnel and filter flask for water washing (LST Wash flask), and place a clean Whatman 114 (25 micron) filter paper cone in the Pure LST flask. Now release the remaining LST into the Pure LST flask. Again, let the pump remove as much pure LST as possible. Pour pure LST into “used, to be filtered” bottle.
6. Everything that goes through LST must be rinsed thoroughly with water, and then acetone. The LST is soluble in both water and acetone so this step should remove any remaining LST from the grains and the filter paper, and maximize LST recovery. Before putting water or acetone on anything, be sure that you are doing so into the proper flask (the LST Wash, NOT the Pure LST flask). Rinse the heavies sample and sides of filter paper 3 times with DI water, using the pump to pull all the water through the paper.
7. Then rinse the heavies 3 times with acetone into the LST Wash flask, using the pump to pull all the acetone through the paper. Once grains are completely rinsed, place the acetone-rinsed heavies filter paper in the small oven (propped in a white teflon beaker) to dry thoroughly (5-10 minutes). Once dry, transfer sample onto weighing paper and into a 1.5 ml polystyrene sample vial. Label the vial such that it is clear the sample has been through heavy liquids by writing "> 2.85".
8. Remove the filter paper with the lights from the Pure LST flask and place on the LST Wash flask under the separatory funnel. Using DI water, rinse the light minerals out of the separatory funnel into the lights filter paper on the LST Wash flask, and also rinse the small beaker and stirring rod into the lights. Rinse the lights sample and sides of filter paper 3 times with DI water, using the pump to pull all the water through the paper.
9. Then rinse the lights 3 times with acetone into the LST Wash flask, using the pump to pull all the acetone through the paper. Once grains are completely rinsed, place the acetone-rinsed lights filter paper in the small oven to dry thoroughly (5-10 minutes). Once dry, transfer lights into the original sample tub. Label the tub such that it is clear the sample has been through heavy liquids by writing "< 2.85".
10. Remove the tubing from the pump inlet and outlets and let it run “open” for 5 minutes prior to shutting down.
11. All glassware must be rinsed thoroughly with DI water into a 1000 to 2000 ml beaker for LST reclamation, before being put in the sink for soap and water washing. Wash used glassware thoroughly in soap and tap water, rinse and hang to dry. Leave the work area clean! Place the contents of the LST Wash flask into the 1000 to 2000 ml glass beaker, and cover with a watch glass until ready to reclaim.

## Heavy Liquids Reclamation: LST

LST can be reclaimed from water and acetone washings by simple evaporation of the solvent. To reclaim:

1. Place the 1000-2000 ml beaker of LST Wash on a stirring hot plate under the fume hood, and set the spin speed at 3.5 and the temperature at 160°C. The liquid will reach a temperature of approximately 60°C under these conditions (you can check this with a thermometer), which is safe for thermal stability of the LST (<80°C). Place a heat lamp over the beaker of LST Wash to speed the evaporation process.
2. Evaporate the liquid down to about 500 ml. If at this point the liquid looks very cloudy you may need to filter the liquid through a pair of Whatman 5 (1-2 micron) filter papers using the vacuum pump. If so, set the beaker off the hot plate to cool to room temperature, and prepare two Whatman 5 filter cones and place on a conical funnel – filtering flask assembly. Pass the cooled liquid through the filters using the vacuum pump to speed the process. This will take a while (e.g. half a day)! Then rinse the filter paper with acetone and allow it to pass through the filter. Discard the filter paper. Transfer the now clarified liquid back into the 1000-2000 ml beaker and rinse out the glassware into the beaker.
3. If the liquid is strongly colored a dark blue – indigo, a small proportion of tungstate has been reduced by metal cations. To restore translucency, add 5 ml of 30% H2O2 per liter of dilute tungstate solution; the reaction may be rapid or might take place overnight--heating will speed the reaction as well.
4. Place the beaker back onto the hot plate at 160°C and continue to evaporate the liquid until it starts to look very viscous and/or small crystals of LST start to form on the sides of the liquid container.
5. Set the beaker off the hot plate to cool to room temperature; the liquid may start to crystallize if it is very concentrated.
6. Fill a 100 ml glass graduated cylinder up to 85 ml with the LST, and then immerse the hydrometer (2 to 3 g/cm3) in the liquid and read off the specific gravity. The target is 2.85 g/cm3; if the liquid is too dense, add small aliquots of DI water to dilute to the target density. If the liquid is too light, return the liquid to the beaker and continue evaporating on the hot plate.
7. Rinse all glassware with DI water back into the 1000-2000 ml LST Wash beaker, cover with a watch glass, and set aside for the next reclamation.

## Heavy Liquids: Bromoform (CHBr3) and Methylene Iodide (CH2I2)

The purpose of heavy liquids is to separate dense grains, such as zircon and sphene, from the bulk of lighter grains such as quartz and feldspar (bromoform) and micas and amphiboles (methylene iodide).

Always wear rubber gloves when using these heavy liquids; they provide better barrier and aren’t soluble in acetone. Don't mix original and reclaimed heavy liquids. If there is a possibility that grains could have gotten into your pure heavy liquid flask, refilter the liquid before putting it back in its bottle.

Equipment to be used: separatory funnel, stopcock, metal stopcock clamp, two flasks, two funnels with stoppers, a funnel for the heavy liquid, a stirring rod and a small beaker to put it in.

1. Rinse washed glassware with acetone before use and blow dry with compressed air or heat gun.
2. Fix the stopcock into the separatory funnel: put a very small amount of stopcock grease on the stopcock, twist around a few times in funnel, then remove and clean off any excess grease, especially from the hole (with a pipe-cleaner). Otherwise grains will become clumped together in the grease. Replace stopcock in separatory funnel in closed position and fasten securely with metal clamp.
3. Prepare two filter paper funnels from “fast” (Whatman 2) filter paper and one from “slow” (Whatman 5) filter paper by folding the circles in half twice.
4. With the separatory funnel suspended above one of the flasks with the slow filter paper in it, funnel heavy liquid (HL) into separatory funnel through a fast (Whatman 2) filter paper. Fill the separatory funnel with an amount of HL necessary for the size of the sample, usually up to the waist of the funnel. Generally, large separatory funnels are used for the bromoform stage, small funnels for the methylene iodide stage; there are plenty of sizes of funnels to choose from to optimize separation and minimize HL use. Once the HL is funneled into the separatory funnel, suspend used funnel over small beaker for later use.
5. Pour sample into separatory funnel with a tube fashioned out of weighing paper. **Make sure that you examine the starting cup/vial under a microscope to make sure all grains have made it into the separatory funnel; small grains can adhere to the side of plastic sample cups via static electricity.** Stir the mixture thoroughly and allow heavy minerals to settle; continue to stir occasionally, always placing the stirring rod in a beaker when not in use. Wait for grains to settle between stirrings. Repeat process as many times as necessary.
6. Attach aspirator hose to the filter flask. When the heavy grains have settled, release them onto a filter flask with slow (Whatman 5) filter paper by opening stopcock carefully. Turn on aspirator to suck pure HL down into funnel. When all the heavy grains are in the filter paper, let the aspirator run for a little longer to remove as much pure HL from grains and filter paper as possible; it helps to lightly press the filter paper against the walls of the funnel.
7. Remove the slow (Whatman 5) filter paper with the heavies from the pure HL flask, place it in the other flask for acetone washing, and place a clean fast (Whatman 2) filter paper cone in the pure HL flask. Now release the light grains and the remaining HL into the pure HL flask. To ensure that the grains don't stick to the separatory funnel, stir as you open the stopcock. Again, let the aspirator remove as much pure HL as possible. Pour pure HL back into original bottle.
8. Everything that goes through HL must be rinsed thoroughly with acetone. The liquids are soluble in acetone so this step should remove any remaining HL from the grains and the filter paper. Before putting acetone on anything, be sure that you are doing so into the proper flask (the HL wash flask, NOT the pure HL flask). Rinse sample and sides of filter paper about 10 times with acetone, using aspirator to pull solution through.
9. When rinsing the lights with acetone, rinse empty glassware used for pure heavy liquid as well as the beaker and stirring rod into the lights. All glassware must be rinsed thoroughly with acetone before being put in the sink.
10. Once grains are completely rinsed, open up the filter paper and place it on a sheet of paper in the hood to dry. Transfer sample to a vial or tub as soon as possible, but not before it is completely dry. Label the tub such that it is clear the sample has been through heavy liquids by writing "density greater or less than 2.85 (bromoform) or 3.32 (MeI)."
11. Wash used glassware thoroughly in soap and water, rinse and hang to dry. Leave hood area clean. Wear gloves while washing glassware. Discard used filter paper and kimwipes in large beaker below hood.

## Heavy Liquids Reclamation: Bromoform and Methylene Iodide I

## Using Water Washing Only

Heavy liquids can be reclaimed from acetone washings by differential solubility in water; acetone is infinitely soluble while both bromoform and MeI are sparingly soluble (about a half a gram per liter). The capital cost of this method is minimal, however it does produce a significant waste stream, since about 500 mL of water is used to reclaim about 100 mL of acetone washings; this water is contaminated with a small amount of HL and thus should be disposed of properly. Also some HL is obviously lost with each round of reclaim.

To reclaim, use a large (1 L) separatory funnel with a well greased, closed stopcock. Add approximately 200 mL of acetone washings to the funnel and top up with about 700 ml of water. Stopper the separatory funnel, carefully remove the funnel from the ringstand, and shake the funnel vigorously for a minute. Replace the funnel in the ringstand and allow the HL to settle for several minutes. Remove the stopper from the funnel, and draw the HL out of the funnel into a clean beaker. Empty the water wash into a storage bottle- you will see drops of HL still in this water and forming along the sides of the funnel; this residual HL can be recovered from the bottom of the storage bottle later.

Repeat the process until all of the acetone washings have been treated, accumulating the HL into the same beaker. The cumulative HL should then be treated a final time, adding it to the separatory funnel and topping off with about 500 mL of water. Again, shake, settle, un-stopper and extract the HL into a beaker. This liquid should be a clear yellow color, not cloudy. Test the specific gravity of the liquid with an appropriate hydrometer—reclaimed bromoform should reach a density of approximately 2.85 g/cm3, and reclaimed MeI should reach a density of approximately 3.25 g/cm3. Once satisfied with its density, transfer the reclaimed HL to a storage bottle for reclaimed HL.

Rinse the separatory funnel and beaker with acetone and transfer this acetone to the HL washings beaker for future treatment. Leave the water washings storage bottle uncapped under a fume hood for the acetone to vent (washings will turn from cloudy to clear). The small amount of HL in the bottom of the bottle can be recovered by transferring the bulk of the water to a second bottle (labeled with a hazardous waste tag for pickup), and the last of the water and HL to a separatory funnel for separation.

## Heavy Liquids Reclamation: Bromoform and Methylene Iodide II

## Using Rotary Evaporator followed by Water Washing

*Stage 1*

1. Switch on the Immersion Cooler Unit; allow the Cold Trap to cool to <-10°C (measured on thermometer immersed in ethylene glycol— takes about 10 minutes).
2. Slowly raise the Evaporator Assembly with the main handle.
3. Carefully connect the Receiving Flask to the lower ball joint with metal clip.
4. Carefully slide the Evaporating Flask onto the glass stem, rotate the Combi-Clip down the threads of the glass stem, flip the metal loop to the right over the lip of the Evaporating Flask, and slowly rotate the Combi-Clip back up the threads of the glass stem drawing the Evaporating Flask snug against the stem— DO NOT use excessive force.
5. Check that Heating Bath is filled with water to within 2 inches of top; if adding water, use MQ-H2O + 1 gram borax to protect bath against corrosion).
6. Switch on the Heating Bath; allow to reach 30°C (about 5 minutes).
7. Slowly lower the Evaporator Assembly with the main handle.
8. Check that there is water in the Water Aspirator, up to the overflow tube.
9. Close the Continuous Feed Valve (handle straight up).
10. Check that the Vacuum Line Valve is closed (handle pointed out).
11. Switch on the Water Aspirator.
12. Open the Vacuum Line Valve (handle straight down) and evacuate the evaporator— takes about a minute, system is evacuated when the aspirator stops frothing vigorously.
13. Close the Vacuum Line Valve (handle pointed out); the system should hold an adequate vacuum for upwards of half an hour— if you notice otherwise, someone may need to grease the valves with silicone.
14. Place the Continuous Feed Line in a reservoir of HL washings.
15. Open the Continuous Feed Valve (handle straight down) to draw HL washings into the evaporating flask; fill the flask to within an inch of the neck of the evaporating flask.
16. Switch on the Rotation Control Switch; the appropriate rotation speed is marked on the Rotation Control Dial.
17. Evaporation and condensation of acetone into the Receiving Flask should begin immediately, with a drop of acetone falling off the cold finger at a rate of at least 1/sec.
18. If necessary, touch up the system vacuum by switching on the Water Aspirator, and then opening the Vacuum Line Valve until the acetone in the flasks begins to boil, then close Vacuum Line Valve and switch off the Water Aspirator.
19. Periodically add washings to the Evaporating Flask by opening the Continuous Feed Valve and drawing HL washings into the flask.
20. Periodically touch up the system vacuum as described in step 14.
21. When Receiving Flask is full, vent system by slowly opening the Vacuum Line Valve to air (handle straight up); remove clip and carefully rotate flask off of ball joint. Empty the reclaimed acetone into an available bottle.
22. When all washings have been fed into system, continue evaporation of acetone until the rate of condensation slows dramatically, and the remaining HL in the evaporating flask “looks” sufficiently reclaimed (takes experience).
23. Switch off the Rotation Control Switch and raise the Evaporation Assembly.
24. Remove the Receiving Flask and empty acetone into an available bottle; reconnect the Receiving Flask.
25. Remove the Evaporation Flask by reversing the process described in step 4.

*Stage 2*

1. If the HL is not strongly discolored, add approx. 100ml of water to the HL in a 250ml separatory funnel, shake vigorously and allow to settle for a few minutes, two or three times.
2. Decant a small volume of HL into a graduated cylinder and check the density of the HL; if the appropriate density is reached, decant off the rest of the HL and filter into the appropriate bottle for reuse.

*Stage 3*

1. If the HL is strongly discolored (e.g. deep red bromoform), and/or Stage 2 did not result in coming up to density, then add approx. 10 g of fullers earth to the HF in an Erlenmeyer flask, and place on the shaker table for a couple of hours under the hood (i.e. in the dark cabinet).
2. Filter the mixture of fullers earth and HL through a filter flask to recover as much HL as possible.
3. Decant a small volume of HL into a graduated cylinder and check the density of the HL; if the appropriate density is reached filter into the appropriate bottle for reuse.
4. Dispose of the used fullers earth into the appropriate waste container.

## Final Frantzing (accessory minerals)

The heavy fraction from the HL separation now needs to be Frantzed for final separation; this will separate residual ferromagnesian minerals (e.g. pyroxene, amphibole, biotite) which are magnetic, from accessory minerals like apatite, most rutile, and zircon (non-magnetic). Accessory minerals like titanite, monazite, xenotime, and some rutile will also be relatively magnetic- look for them between 0.3A/20° and 1.0A/20°.

With the Frantz set to the paramagnetic position (feed, or right side of chute within magnetic field, splitter or left side outside of magnetic field, feed onto far side of chute), set forward tilt angle to +20° and the magnet to 1.0A and run the grains through. **Make sure that you examine the starting cup/vial under a microscope to make sure all grains have made it into the Frantz hopper; small grains can adhere to the side of plastic sample cups via static electricity.** Once the grains are split, store the magnetic fraction in a labeled vial (e.g. 0.3A/20°<m<1.0A/20°).

Increase the magnet to 1.4A and run the nonmagnetic fraction through again. Again store the magnetic fraction (1.0A/20°<m<1.4A/20°), and then keeping the magnet at 1.4A, shallow the forward tilt +10° and run grains through.

Repeat this splitting, always setting the magnetic grains aside in vials and rerunning the nonmagnetic grains though angles 5, 3, and 1°, or until no nonmagnetic sample remains.

If necessary, for the final split set magnet to diamagnetic position (feed or right side of chute out of magnetic field, splitter or left side within magnetic field, feed onto near side of chute). Set angle to -1. Turn magnet on and set current to about 1.4A. If there are many diamagnetic grains, set angle to -2 and run diamagnetic grains through again, putting the non-diamagnetic at -1 grains back in the vial for now. Place those grains that are not diamagnetic at -2 but are diamagnetic at -1 in a labeled vial. Change angle to -3 repeat process until all of the separate falls into the magnet.

Label all vials with a sticker detailing sample name and magnetic fraction. Wrap the label on the vial in scotch tape to ensure that stickers don't dry up and fall off. Place all vials in a labeled box.

## Hand-picking (accessory minerals)

### A note on the choice of solvent:

In general, ethanol is the most appropriate medium for hand-picking under liquid, due to its lower toxicity and higher vapor pressure relative to methanol or acetone. Anhydrous (100%) ethanol (e.g. from Pharmco) rather than denatured ethanol is preferred.

On the other hand, ethanol (unless it is HPLC grade) is a very poor solvent for cleaning equipment, as it is a poor solvent for oils and as well contains a certain amount of residual 'fusel oils' if it has been made by fermentation and distillation. Chemical dictionaries say this residual materials’ composition depends on the material fermented and on the yeast used, but is mostly isoamyl, laevorotatory amyl, isobutyl, isopropyl and normal propyl alcohols, with fatty acids and their esters, boiling over the range 105-107°C. Practically, if you allow a volume of ethyl alcohol to evaporate it will leave an oily residue, exactly what you don't want. High quality methanol or acetone (e.g. “Omnisolv”) are much better for cleaning vacuum or optical parts, being better and cleaner solvents. For the same reasons, drying down mineral separates in ethanol will “stick” them to the bottom of the petri dish to some degree.

Small amounts of minerals are best stored in either small polystyrene petri dishes, or 1.5ml polystyrene vials with polypropylene caps, as these are relatively static-free containers that fit in the small white boxes for storage, and from which grains are easily dispensed in the latter case. CARE SHOULD BE TAKEN NEVER TO INTRODUCE ACETONE INTO SUCH CONTAINERS AS IT WILL DISSOLVE THE POLYSTYRENE AND SEMI-PERMANENTLY FUSE YOUR GRAINS INTO THE VIAL. The polypropylene caps are however stable in acetone.

### Procedure:

To begin the picking process, select a new small polystyrene petri dish and write the sample name and magnetic fraction on dish cover with a Sharpie. Generally, one starts picking the diamagnetic, or least paramagnetic fractions first, however procedure varies from sample to sample. Pour contents of vial into the dish and fill dish half way with ethanol. Swirling of the dish in a circular manner efficiently sweeps the grains into the center of the dish, and also generally results in the heavy minerals being concentrated along the edges of the resulting pile.

Grains are maneuvered either with fine jeweler’s tweezers or a dental pick. The use of transmitted light is most useful for observing internal cracks or inclusions in grains. Putting a piece of white paper under the petri dish generally allows optimal visibility of the grains in reflected light. It is often a good strategy to separate a few grains from the main pile, pick out the good ones and start establishing a second pile of "already picked through” grains, keeping the good grains separate from these two piles.

Ask yourself how many and what kind of picks need to be made. Careful record keeping of each pick is recommended so that you remember what the color and state of the grains were and at what stage the pick is at. If possible, pick a few more grains than are necessary, in case some get lost. Photos may be taken for archival purposes and also as a means to estimate the weight of a pick and the degree of abrasion.

To remove grains from a petri dish, first take a disposable fine-tipped plastic transfer pipet and cut the end off at an angle. The pipet should only be used for a single sample. Fold a piece of weighing paper twice corner to corner, and fix two points into slits cut into the sides of a paper box to make a boat. Pipet the sample into the boat, then pipet out the excess ethanol back into the Petri dish. Add a few drops of distilled acetone to the boat to coat the grains, remove the excess, and place the boat under a heat lamp to dry. The dried sample can be easily and quantitatively transferred back to a vial.

## High-temperature annealing of zircons

Following Mattinson (2005), the vast majority of zircons should be treated by the high temperature annealing and chemical abrasion method, to mitigate the effects of Pb loss through selective removal of high-U, radiation-damaged, open system zircon domains.

The chemical abrasion procedure is described in detail in a later section. The high-temperature annealing step is recommended for all grains prior to further procedures including air abrasion (for example to remove young overgrowths) and grain mounting and polishing for imaging.

To anneal zircons:

1. Transfer your grain selection from a Petri dish to a 10 ml quartz glass crucible using a transfer pipet and ethanol. Pipet any excess ethanol from the beaker, then add a small amount of distilled acetone, swirl, and again pipet out any excess actetone leaving behind only a small amount of acetone with the grains which will rapidly evaporate.
2. Bring the loaded quartz crucibles into the oven lab, and place in the muffle furnace. Close the door and switch on the muffle furnace. The set temperature should be 900°C; to check the set temperature press and hold “Set” until the set temperature flashes. After several seconds of inactivity the display will return to the actual temperature.
3. The recommended annealing period is at least 60 hours. Upon completion of the annealing period, switch off the muffle furnace, open the door of the furnace slightly and allow the interior to cool.
4. When the crucibles are cool to the touch, return them to your picking station and transfer the grains back into the appropriate container (e.g. Petri dish, vial).

## Air-abrasion (accessory minerals)

If grains are to be abraded ( for example to remove young overgrowths from zircons, or remove outermost Pb-loss domains from monazite and sphene), prepare an abrader by cleaning all parts with ethanol and a kimwipe. Cut two pieces of 100 mesh sieve cloth using the compass cutter and spray adhesive on a piece of plexiglass, and assemble abrader.

While looking through the microscope, use a transfer pipet to pick the grains up and move them out into the awaiting abrader. Slosh zircons to one side and pipet up excess ethanol. To be sure that you aren't losing any grains in this process, squeeze the excess ethanol back into a clear area of the petri dish as you watch through the scope. Rinse the grains in distilled acetone. If grains are left to dry in ethanol, a waxy residue will form around them. They must be rinsed in acetone. Let acetone evaporate, then add about 3 times as much pyrite as there is zircon. Match pyrite size to the grain size of the zircon.

Close the abrader, bring down to the darkroom/mineral separations lab and attach the stem to an air hose. Always put a piece of paper under the abrader with the name and magnetic fraction of the sample, the start time and the psi. Writing your name on the paper is also a good idea in case you have to ask someone else to turn it off for you or if someone has a question about the sample. Generally, samples are abraded for 3-30 hours at 2.5 to 4.5 psi; it depends on the sample. Check the abrader occasionally to be sure that the sieve cloth hasn't ripped and to check on the grains. Usually, the goal is to get polished grains with no crystal faces.

To remove the abraded grains, dump the contents of the abrader dry, or add ethanol to abrader and pipette into the *top* of a clean petri dish. Add more ethanol, swirl, and sort through the pyrite to recover the abraded zircons (transmitted light is useful for this task). Pipet the zircons (without the pyrite!) to the bottom of the petri dish and then discard the pyrite in the top by rinsing with water and drying. The abraded grains are now ready for acid washing.

## Acid-washing following air abrasion

Zircons are washed with dilute nitric acid to dissolve adhering pyrite and surface-correlated common Pb. The first acid washing is done in one of a set of dedicated 15 ml Savillex beakers the clean lab.

1. Transfer the abraded grains from the Petri dish of ethanol in which you extracted the grains to a 15 ml Savillex beaker using a transfer pipet. Pipet any excess ethanol from the beaker, then add a small amount of distilled acetone, swirl, and again pipet out any excess actetone leaving behind a small amount of acetone with the grains.
2. In the clean lab add a few ml of 4M HNO3 to the Savillex beaker (titanite and monazite are more susceptible to partial dissolution and should be washed with 1M HNO3), cap and leave on the hotplate for 30 minutes. Then transfer dish to the ultrasonic bath and leave there for 30 minutes.
3. To remove nitric acid, swirl the grains into the center of the beaker, and then carefully pipette off nitric acid from the side of the beaker and discard. Rinse twice with distilled acetone before leaving hood.
4. Bring the Savillex beaker back to your picking station and transfer the grains back into the appropriate container (e.g. Petri dish, vial).

The grains should be photographed prior to for further treatment, in order to estimate the degree of abrasion.

## Grain mounting and polishing—hand placement method

If grains are to be mounted in epoxy for imaging, it is recommended that they be already annealed.

### Materials:

Struers EpoFix epoxy and hardener

1 cm inner diameter glass ring (approx. 1.5 cm tall), ground flat

Buehler release agent

1” round glass or plastic ring mold

Kapton double-stick tape

Small (e.g. 2” x 2” square) glass plate

PSI-1415S-8 (8" disks, plain backed polishing film, SiC, 15 micron, 50/pk)

PSI-1409S-8 (8" disks, plain backed polishing film, SiC, 9 micron, 50/pk)

PSI-1403-8 (8" disks, plain backed polishing film, Al2O3, 3 micron, 50/pk)

PSI-1401-8 (8" disks, plain backed polishing film, Al2O3, 1 micron, 50/pk)

Buehler Micropolish II Alumina, 0.3 micron (part number 40-6363-006)

Buehler Texmet 1500 cloth (part number 40-8618)

Buehler MasterMet colloidal silica Polishing Suspension (part number 40-6370-006)

Buehler Microcloth (part number 40-7218)

### Procedure:

To make the mount, trace a 1 cm diameter circle inside a 1” diameter circle on the glass plate, then center a piece of Kapton double-sided tape over those circles on the glass plate. Transfer grains from your petri dish by cutting a small 1 cm square of Kapton tape and pressing this lightly down on your dry grains in the dish. Move this small square of Kapton tape with grains to the edge of the 1” circle on your glass plate.

Use a tweezers to pick up and arrange the grains and place them down carefully onto the tape surface. The tape surface is relatively elastic and will rebound nicely after pressing them down lightly onto the tape. Arrange the grains in a matrix of double rows within the 1 cm inner circle so that you can keep track of them during imaging, and record a map of the mount for the log book.

Once you’ve finished laying out the grains, dunk the 1 cm glass ring into Buehler release agent and allow to dry, and then center the 1 cm glass ring around the matrix of grains and press firmly against the Kapton tape. Coat the interior a 1” diameter ring mold very lightly with silicone grease, then press it tightly against the Kapton tape with the 1 cm PFA ring + grain matrix centered within it.

Weigh out 25 parts Struers resin to 3 parts hardener in an aluminum pan on the OHAUS balance, stir a bit with a wooden stick, and place under the heat lamp for a minute to thin it out a bit, then stir thoroughly. Pour the resin slowly along the side of the mold, covering the grains and filling the mold up to about ¾”. Under the microscope, use a thin bent wire to free any air bubbles that may have formed on the surface of the mount around the grains (they will float up to the surface).

Allow the mount to harden for 18 hours.

Mounts are then gently ground to approximately the centers of the grains using 15, 9, 3, 1 micron SiC and Al2O3 lapping film. Place the film on a flat hard surface (stick it down with a bit of water on the back), and then start doing figure-eights with moderate pressure. When grinding down to near (but not to) the centers of grains, periodically examine the mount under a stereomicroscope in reflected light. That initial grinding with 15 or 9 micron film (depending upon grain size) should be done in short, careful increments, being very careful to not over-grind the mount. The rest of the lapping film steps are just a minute on each; clean the mount in an ultrasonic bath for 3 minutes in between steps.

Finish the mount by fairly aggressive polishing for several minutes with Buehler Micropolish II Alumina, 0.3 micron on Texmet 1500 cloth. You should be able to remove all pits and scratches, and get a lovely flat polished surface on the majority of grains. Ultrasonicate and wipe the surface of the mount thoroughly with methanol prior to carbon coating for CL imaging.

## Grain mounting and polishing—detrital zircon ‘terrazzo’ method

If grains are to be mounted in epoxy for imaging, it is recommended that they be already annealed.

### Materials:

150 and 70 micron nylon sieve cloth

Custom micro-sieves (1.5 ml vials with joined lids)

1” round glass or plastic ring mold

Kapton double-stick tape

Small (e.g. 2” x 2” square) glass plate

Struers EpoFix epoxy and hardener

PSI-1415S-8 (8" disks, plain backed polishing film, SiC, 15 micron, 50/pk)

PSI-1409S-8 (8" disks, plain backed polishing film, SiC, 9 micron, 50/pk)

PSI-1403-8 (8" disks, plain backed polishing film, Al2O3, 3 micron, 50/pk)

PSI-1401-8 (8" disks, plain backed polishing film, Al2O3, 1 micron, 50/pk)

Buehler Micropolish II Alumina, 0.3 micron (part number 40-6363-006)

Buehler Texmet 1500 cloth (part number 40-8618)

Buehler MasterMet colloidal silica Polishing Suspension (part number 40-6370-006)

Buehler Microcloth (part number 40-7218)

### Procedure:

Use clean 2 cm squares of nylon sieve cloth and a microsieve to sort the zircon separate into >150 micron, 70 to 150 micron, and <70 micron size fractions. Label each new 1.5 ml vial. Sprinkle the required amount of each grain size into a polystyrene picking dish.

To prepare the mount, trace a 1” diameter circle on the glass plate, then center a piece of Kapton double-sided tape over the circle on the glass plate.

Create your Kapton tape apertures by cutting a 1 cm wide strip of Kapton tape, and a set of 1 x 1.5 cm square masks of weighing paper. Place one of the weighing paper masks onto the end of the Kapton tape strip, overlapping just a couple of millimeters. Place a second weighing paper mask with an approximately 0.5 millimeter gap between it and the first piece of weighing paper. Then carefully cut off the end of the Kapton tape strip a couple of millimeters from the aperture. Remove the offcut of weighing paper mask from the larger Kapton tape strip; if any weighing paper adheres to the Kapton tape, cut it down to a fresh end. Repeat this process until you have a sufficient number of 0.5 mm x 1 cm apertures for mounting.

Pick up one of your Kapton tape apertures with a tweezers by the weighing paper ‘handle’, invert and lightly press the sticky side of the aperture down onto the dry grains in your picking dish. Repeat picking up and pressing the aperture down until the entire sticky area is coated with zircon grains in a monolayer. Tap off any excess grains adhering by static to the weighing paper masks. Now with a tweezers carefully remove the plastic backing from the Kapton tape aperture, and invert this to place the aperture strip, grains up, onto the larger square of Kapton tape within your 1” circle on your glass plate. Gently press this aperture strip down onto the larger square of Kapton tape making sure there are no grains stuck underneath between the two tape pieces.

Repeat this process for as many strips as necessary for each grain size for each sample. Multiple samples of the same grain size can be placed on the same mount by this method; make sure you carefully map the distribution of these strips. Placing them in an asymmetric array helps distinguish each sample.

Coat the interior a 1” diameter ring mold very lightly with silicone grease, then press it tightly against the Kapton tape with the 1 cm PFA ring + grain matrix centered within it.

Weigh out 25 parts Struers resin to 3 parts hardener in an aluminum pan on the OHAUS balance, stir a bit with a wooden stick, and place under the heat lamp for a minute to thin it out a bit, then stir thoroughly. Pour the resin slowly along the side of the mold, covering the grains and filling the mold up to about ¾”. Under the microscope, use a thin bent wire to free any air bubbles that may have formed on the surface of the mount around the grains (they will float up to the surface). Allow the mount to harden for 18 hours.

The next day remove the mount from the mold, and then peel off the small Kapton tape aperture strips leaving a set of troughs with the grains at their bottoms. Again weigh out 25 parts Struers resin to 3 parts hardener in an aluminum pan on the OHAUS balance, stir a bit with a wooden stick, and place under the heat lamp for a minute to thin it out a bit, then stir thoroughly. Use a transfer pipet to fill in the troughs with resin, viewing the process under the microscope. Allow the mount to harden for 18 hours. If the troughs are subsequently still softer than the larger mount surface place the mounts in the vacuum oven for a few hours to harden further.

Use the finer rectangle of SiC lapping paper to slowly and carefully grind the surface of the mount down until the very tips of the zircon grain surfaces are exposed. Monitor the grinding process using the Leica stereomicroscope with both transmitted and incident reflected light illumination. If you begin to see grinding happening differentially, e.g. you’re closer to the grains on one side of the mount versus another, then apply additional pressure on the opposite side of the mount to even out.

Mounts are then more gently ground to approximately the centers of the grains using 15, 9, 3, 1 micron SiC and Al2O3 lapping film. Place the film on a flat hard surface (stick it down with a bit of water on the back), and then start doing figure-eights with moderate pressure. When grinding down to near (but not to) the centers of grains, periodically examine the mount under a stereomicroscope in reflected light. That initial grinding with 15 or 9 micron film (depending upon grain size) should be done in short, careful increments, being very careful to not over-grind the mount. The rest of the lapping film steps are just a minute on each; clean the mount in an ultrasonic bath for 3 minutes in between steps.

Finish the mount by fairly aggressive polishing for several minutes with Buehler Micropolish II Alumina, 0.3 micron on Texmet 1500 cloth. You should be able to remove all pits and scratches, and get a lovely flat polished surface on the majority of grains. Ultrasonicate and wipe the surface of the mount thoroughly with methanol prior to carbon coating for CL imaging.