# *PHYSICAL PREPARATION OF QUARTZ SAMPLES FOR Al/Be EXTRACTION*


## *Summary*

This method describes the initial stages of quartz separation from rocks or sediments intended for $^{26}Al/^{10}Be$ analysis. Rock samples are crushed, and then crushed rock or natural sediment is sieved to an appropriate grain size. The objective of the crushing and sieving process is to maximize yield of the desired grain size, usually between 250 and 850 microns.


## *Version*

Revised July 2002 by Greg Balco.

## *References*

None.

## *Sample size*

Decide on the appropriate amount of rock to crush. The goal is to wind up with ~30 g of quartz at the very end of the quartz purification process. For a typical granite with 25% quartz, this requires ~150 g of crushed rock in the proper grain-size range. Again for the typical granite, this usually requires 0.75-1 kg whole rock at the beginning of the process. If you have more rock (unless the sample is really huge, 2 kg or better), crush the entire sample. The crushing yield is very dependent on rock type and operator skill -- getting it right requires some experimentation as described below. A good rule of thumb is that a sample about the size of a softball will yield more than enough material. Very quartz-rich rocks require much smaller samples; very quartz-poor rocks may have to be significantly larger.

First, either photograph or sketch the rock. Also, this is your last chance to determine how thick your sample is (information you will need later), so make sure the sample has been measured properly before you start crushing,

Break the rock into small pieces (< 5-6 cm) using the large hammer located in the crushing room. Just outside the JHN courtyard door there is a large metal plate which makes a convenient place to do this. Unless the sample is particularly small, save a representative chip of the rock so you will remember what it looked like -- about a golf-ball-sized chip is sufficient. It's easiest to just write the sample number on the chip with a permanent marker. Put the rest of the chunks into a large labeled plastic bag. Banging on rocks with a hammer invariably produces lots of finer material as well as the large chunks -- try not to lose too much of the smaller stuff if the sample size is small, but remember that the most important thing is not to cross-contaminate one sample with another. Be

sure that all the chunks you put in the bag actually came from the rock you think they did. Sweep away the dust from the previous sample before crushing the next one.

***Jaw crushing***

The basement of Johnson Hall (Room 26) contains two rock crushers: on the left, a jaw crusher with infeed size of ~6 cm and outfeed size of ~1 cm; and on the right, a plate grinder with infeed size ~1 cm that produces sand-size material.

Vacuum all parts of the jaw crusher, including the collection tray. It's not necessary to worry about contamination by fine dust (fine-grained material will be removed later in the process) but the machine must be clean of all fine-sand-sized and larger debris before crushing each sample. Remember to replace the collection tray before you start crushing.

After putting on ear and eye protection as well as a dust mask or respirator, turn on the jaw crusher.

**NOTE**: This machine has an extremely powerful motor, many uncovered moving parts, and a heavy flywheel that imparts enormous inertia to all of the moving parts. In addtion, it has neither a timer or a deadman switch. **DO NOT OPERATE THE JAW CRUSHER WHILE WEARING ANY LOOSE CLOTHING THAT COULD BECOME CAUGHT IN THE BELTS OR FLYWHEEL. BE ESPECIALLY CAREFUL OF LONG HAIR.**

Begin feeding chunks of rock into the jaws. Since the machine will periodically try to eject rock fron the jaws, hold one of the wood 2x4's kept near the machine over the jaws except when you are actually feeding rock in. When you are done, all the material in the tray should be in 0.5 - 1 cm chunks. If any larger pieces slipped through the jaws, pick them out of the tray and toss them back in.

Turn off the machine. Pour the crushed sample back into the labeled sample bag. Vacuum the crusher thoroughly before starting the next sample.

***Plate grinding***

Grinding plates are kept in the tall cabinet in the inner room of the thin section lab. Bolt the plates to the appropriate places on the grinder. The rotating plate is rigidly fixed to the motor spindle; the boltholes for the fixed plate are slightly oversized so that it can be moved around somewhat. When you bolt on the fixed plate make sure that it is exactly aligned with the rotating plate.

Close and toggle shut the front door of the grinder. Set the gap between the plates using the screw and lockring on the other end of the motor housing. Setting the gap correctly is very important to getting the right grain size -- too small a gap and much of the sample will be crushed too finely to be useful. For large samples, it is most efficient

to start with a gap of approximately 1 mm. For small samples where you are worried about losing too much as powder, crushing in two stages will improve yield of the proper grain size -- crush the sample once with a large gap (~1-2 mm), sieve the sample, re-crush the fraction larger than the desired grain size, then sieve the results a second time.

Clean the crusher thoroughly. Brush all interior surfaces with the plastic brush. Brush the grinding plates themselves thoroughly with the stiff wire brush. Make sure there are no sand-sized particles adhered to the plates.Vacuum all surfaces. Vacuum the sample tray. Again, it's not necessary to worry about contamination by fine dust (fine-grained material will be removed later in the process) but the machine must be completely clean of fine-sand-sized and larger debris before crushing each sample. After brushing and vacuuming, blow the remaining dust off interior surfaces with compressed air.

Close both doors, don eye, ear, and respiratory protection, and start the grinder. MAKE SURE THE SAMPLE TRAY IS IN PLACE. Insert the vacuum hose into the port below the sample and turn on the vacuum. Feed the sample slowly into the intake. Listen to the machine -- if it starts to slow down, stop adding sample until it speeds up again. Relatively brittle rocks like granite can be fed more rapidly than more resilient ones such as quartzite. If the machine jams entirely (possible with very refractory quartzites), TURN OFF THE POWER FIRST, then open the door, clear the jam, close the door and resume. After you have crushed a small amount of sample, pull out the sample tray and look at the results. There should be a fair amount of coarse sand-size material in the tray -- if it seems too coarse or fine you may need to readjust the plate gap.

When finished crushing the sample, turn the machine off. Pour the sample back into the labeled bag and clean the machine thoroughly before loading the next sample.

***Dry sieving of crushed rock***

The later steps of the quartz purification process (heavy liquid separation and HF etching) work best on grains between 500 and 850 microns. Grain sizes down to 250 microns are acceptable. Grains smaller than this are hard to work with. However, there are two other factors to consider when selecting the grain size to be processed. First, the grain size you are using must be smaller than the grain size of mineral grains in the rock. This minimizes multimineralic grains and makes later separation processes more efficient. Second, enough material, usually 150 g, must go on to the next step. In general we try to use the 850-500 micron grain size fraction if enough material is present. If not, we add the 250-500 micron fraction. In exceptional cases we will work with smaller grain sizes. Generally, however, we onlty extract these two fractions from the crushed rock.

Crushed rock samples should be dry-sieved. The sieve room next door to the rock crushers contains sieves and a sieve shaker. Assemble a stack of sieves as follows: pan, 250 micron, 500 micron, 850 micron, and lid. Clean the sieves by inverting them and banging them (hard) on the bench, then by brushing the underside with a brass brush. 500 and 850 micron sieves can be brushed aggressively; be more careful with the 250. It's

impossible to remove all of the old grains that are stuck in the mesh, but do the best you can. Pour the sample into the uppermost sieve. Note that simply dumping in 1 kg of sample has a good chance of breaking the sieves when they are placed in the shaker -- the process has to be repeated several times, with a small amount of sample each time, for large samples. Put the sample in the shaker and shake for ~5 min. Label four bags with the sample name and the respective grain-size fractions. Dump the contents of the sieves into the appropriate bags. Repeat until all of the crushed sample has been sieved. If the 850-500 micron fraction contains 150g or greater at this point, you're done. If not, you may have to re-crush the > 850 fraction and sieve it again to get more sample. If you have done this and

Clean the sieves thoroughly before sieving the next sample.

***Wet sieving of sediments***

Sediment samples are typically collected wet and are typically somewhat indurated by clay, carbonate cement, or iron oxide cement. Thus, it's easier to disaggregate them in water or other aqueous solution and then wet-sieve them to the desired grain-size fraction. Again, the goal is to obtain ~150g of sediment in either the 250-500 or 500-850 grain-size fraction. For mature quartz-rich sands, less sample is needed -- typically only 50-100 g -- to produce the correct amount of pure quartz at the end.

Clay-rich sediments can be disaggregated by trying the following techniques, in order of aggressiveness. First, rinse the sample in water and soak for a day or two. Second, sonicate the sample in a beaker full of water for several hours. Third, submerge the sample in a beaker containing a mixture of water and Calgon water softener, then sonicate. In all of these cases a high ratio of water to sediment causes the sample to disaggregate faster, i.e., don't fill a beaker entirely with sediment, add a small amount of water, and expect anything to happen. Large samples may require multiple beakers. If none of these techniques work for indurated clay-rich sediments, the last resort is to dry the sample thoroughly into a brick, then crush it as you would a rock sample.

Carbonate-cemented sediments will need to be treated with nitric acid. Again using a high ratio of liquid to sediment, place the sediment in a beaker and fill with 2% $HNO_3$. Repeat as necessary. Particularly well cemented samples may require stronger $HNO_3$ solutions, upt to 10%.

Iron oxide cements are most efficiently removed with HCl. Again, soaking in dilute (2%) HCl usually does the job. Boiling the sample in stronger HCl (careful) will speed up the process for particularly well-indurated samples.

When the sediment is fully disaggregated, wet-sieve it. The wet-sieving apparatus is in the rock storage room attached to the mineral prep lab.

Assemble a stack of sieves (without a pan -- the fine fraction is lost in this process). Make sure the sieves are clean. If not, clean them by washing wth a high-pressure stream of water from the tap and brushing with one of the brass brushes. The larger-size sieves can be brushed aggressively -- be more careful with the finer meshes.

Place the sieve stack over the sediment trap. The sediment trap is the gray plastic bin in the sink: it will gradually fill up with water until the water begins to drain through the hole in the side. The idea is that the sediment will settle into the bottom of the tub before the water goes down the drain. Gradually add the sample to the uppermost sieve while washing it with tap water. Continue to rinse the uppermost sieve until you are sure that all the grains that should go through the mesh have done so, then put the uppermost sieve aside and repeat the process for the next sieve down. When you have rinsed all the sieves, label an appropriate number of Pyrex beakers with the sample name and grain size. Rinse the contents of each sieve into the approriate beaker. Rinse the material in each beaker several times with tap water, then either put in the oven to dry (for samples that will be archived) or move on to the nitric acid pre-clean (for samples that will be processed immediately).

Clean the sieves thoroughly before loading the next sample.