Draft 5: July 10, 2018

UVM Cosmogenic Laboratory – Final Quartz Etching

Purpose: This method details how we do a final clean etch of quartz mineral separates and clean the tubes in which they are contained. This etch ensures that any surface contamination or dust from the mineral separation lab is removed from the samples before dissolution. This method facilitates the rapid and safe cleaning of 24 samples at a time using 1% HF and HNO_3 and is performed in the *in situ* laboratory. After this procedure is completed, the samples are labeled as final etched and are ready for dissolution.

Hazards: The primary hazard associated with this method is contact or inhalation exposure to concentrated HF and HNO_3.

Important Notes
The ultrasound cannot run more than 14 hours without being topped up with water. Always set the time to limit run time to no more than 14 hours. FAILURE TO DO THIS RESULTS IN REPAIR BILLS > $400.

Getting Ready
Personal Protective Equipment: Thin gloves, goggles, and lab coat.

1. Select a batch of 24 samples that have been deemed clean after quartz purity testing. Place them together in a centrifuge tube rack.
2. Remove the bin of 180 mL Savillex final etching beakers from under the hood, drain off the 1% HNO_3 to the sink in the wash hood, and rinse the beakers and lids several times with **Milli-Q** water. The beakers are now clean and ready to use.
3. Line the 24 sample beakers up on the countertop in a 6x4 matrix and label with blue or yellow tape (on the side of the beaker, not the lid) with the sample names.
4. Add quartz from each tube to its pre-labeled beaker, swiping the sample tube through the anti-static device before pouring. Do this one sample at a time to prevent errors. Pour over the sink in the wash hood to make clean-up easier.
5. Use the 1% HNO_3 squirt bottle to rinse any remaining quartz into the Savillex beaker, then rinse the sample tube and lid with **Milli-Q** water thoroughly.
6. Place the tubes in the rack, place the lids on a tray, and dry everything in the oven.

Mixing and Adding Acid
Personal Protective Equipment: Double gloves (thick over thin), goggles, rubber smock, and face shield.

1. Fill the dedicated final etch 5 L stock solution jug to the fill line with **Milli-Q** water.
2. In the hood, add 50 mL HNO_3 using the dispenser (dispense into a clean acid beaker first, then pour into the jug).
3. In the hood and in a spill tray, add 75 mL HF. Do this addition by pouring from the

HF bottle into a clean acid beaker, then pouring from the beaker into the solution.
4. Cap the jug of stock solution tightly and invert several times to mix.
5. Wash the clean acid beaker using Milli-Q and cover it with parafilm.
6. Place the clear plastic rack for holding the beakers on the ultrasound.
7. One at a time, place a sample beaker on the deck of the hood in a spill tray. Carefully dispense from the stock solution to fill the beaker to within several cm from the top. Cap the beaker tightly and place it into the rack sitting on the ultrasound.
8. Repeat for all samples until the rack is full.
9. Fill the ultrasound to the top row of holes with DI water.
10. Turn on the ultrasound using the wall timer and let it run for at least several hours.
11. Rinse out the empty stock solution jug with Milli-Q and store it under the hood.

Finishing the Etch

Personal Protective Equipment: Double gloves (thick over thin), goggles, rubber smock, and face shield.

1. Remove the beakers from the ultrasound and place them on the countertop.
2. Remove the clear plastic rack from the ultrasound and place it on the bottom shelf of the oven next to the wash hood.
3. Place the colander in the sink to hold the beaker lids.
4. One at a time, remove a beaker from the matrix and bring it into the hood. Remove the lid and place it in the colander in the sink.
5. Decant the acid into the sink while running the DI water tap.
6. Rinse the beaker and quartz at least four times with **Milli-Q** water, bringing the Milli-Q dispenser into the hood to avoid acid fume inhalation. Also, rinse off the outside of the beaker so that no acid gets into the oven. Pour off as much water as possible to speed drying time.
7. Put beaker with quartz in the oven to dry, sitting it in the plastic rack.
8. Repeat for all samples. Fully dry samples in the oven, which may take 24-48 hours.
9. Wash the beaker lids well to get rid of all acid, then leave them in the back of the hood until they are reunited with the beakers.

Final Steps

Personal Protective Equipment: Thin gloves, goggles, and lab coat.

1. When the quartz is completely dry, transfer each sample back to its centrifuge tube by pouring carefully. Do one sample at a time over the wash hood sink, swiping the beaker through the anti-static device first.
2. Place a RED sticky dot on the top of each tube to indicate that it has been final etched. Set tubes in racks labeled with your name/project on the shelf above the computer.
3. Remove the label tape. Thoroughly rinse beakers several times with the DI spray to remove all remaining quartz; visually check to ensure the beaker is free of grains.
4. Nearly fill beakers with **Milli-Q** water and add 2 mL HNO_3 from the dispenser. Cap tightly and shake. Store the beakers full of 1% Nitric in the labeled box in the storage cabinet under hood.
5. Rinse the hood and sink well after you are done.