Separation and Purification of Quartz from Whole Rock

Introduction

The following sections describe methods for obtaining clean quartz from a sample of whole rock using a combination of both physical and chemical preparation steps. The physical preparation involves sawing, crushing, grinding and magnetic separations. The chemical methods involve leaching the crushed rock sample in acids.

Physical Preparation of Quartz from Rock

***Safety information**: The crushing, grinding and sieving of rocks produces high amounts of dust, and inhalation of dust particles should absolutely be avoided. Review the procedures for operating the ventilation systems for these pieces of equipment and procedures. ALWAYS WEAR A DUST MASK (NIOSH approved N95), safety goggles, work gloves, long pants and closed shoes.*

1) Rock samples may need to be cut using the small or large saw in the basement of the Comer building.

2) The samples are crushed into small pieces (less than 4 cm^3) using a hydraulic. Place the protective plastic ring around the rock sample during crushing to contain rock pieces.

3) Samples are then crushed using a disk mill. Place the nozzle of the high-powered vacuum next to the disk mill. This will remove the majority of the dust particles from the source area. Crush rock pieces into sand-sized grains (generally < 0.7 mm). It is necessary to put the sample through the disk mill numerous times and progressively move the disks closer together to achieve the desired grain size without producing excess fine-grained sediment.

4) The crushed rock can then be put though a column of sieves to sort the sample by grain size.

Cleaning: Saws and rock crushing machines should be thoroughly cleaned after each sample. Rinse saws with water and dry them completely afterward. Use oil-spray to protect the metal pieces from oxidation. Empty water collection buckets below saws. Rinse and dry the plates of the hydraulic press. Clean the disk mill using a vacuum, air compressor and small broom or brush. After cleaning the disk mill, turn it on and let it run for a few seconds without putting a sample in and observe to see if grains are in the pan. Clean sieves with a brush and put in the small ultrasonic bath in Geochemistry Lab

3. Dry sieves in an oven and inspect them for cleanliness. If grains are still present in sieves, clean further with a brush or air compressor.

Magnetic Mineral Separation

1) An initial rough non-magnetic from magnetic separation can be achieved by putting the sample through a chute magnet. The grain size used is generally between 0.125-0.7 mm.

2) The non-magnetic fraction attained using the chute magnet is then put through a Frantz Isodynamic separator located in the Geochemistry building. Put sample through the Frantz Isodynamic separator (generally at 0.5 Amps and a 5 degree tilt) until about 150 g of non-magnetic grains are obtained. Collect magnetic and non-magnetic fractions and store in well-labeled Ziploc bags. Store all fractions of samples in a small well-labeled plastic container.

2) Cleaning: Clean the chute magnet and Frantz isodynamic separator thoroughly after each sample. Rinse and dry the chute magnet. All parts of the Frantz isodynamic separator should be taken apart and cleaned using paper towels and the vacuum. Rinse and dry glass beakers used to collect samples.

NOTE: Magnetic separations are not always needed, but when they are, we often do this step after the phosphoric acid boiling and the first two hydrofluoric acid leaches so as to reduce the total sample size before this step.

Quartz Purification – Chemical Preparation Steps

VERY IMPORTANT Safety Information

The following steps require the use of strong acids that present skin and inhalation exposure risks, and for HF, systemic toxicity. Review the MSDS sheets and any other documentation provided. Understand the risks associated with handling the chemicals you are working with, the procedures for reducing any risks and emergency procedures in the event of an accident.

- Always work in a fume hood with the sash as low as practical.

- Wear safety/splash goggles, and use a full coverage face shield if there is any risk of splashing.

- Wear appropriate gloves. For work with hot acids, use heavyweight (22 mil) neoprene gloves. For work with HF, you must wear HF resistant gloves – not all materials are HF resistant (for example, latex). Use heavyweight neoprene or nitrile gloves with a thin pair of the blue nitrile gloves underneath. Check your gloves regularly for holes and excessive wear and replace as needed. The thin gloves get holes in them very easily.

- You must wear long pants and closed shoes. Shorts, skirts, and open toed or fabric shoes are not permitted when working with chemicals.

- Know where the eye wash stations, safety showers, spill kits, and tubes of calcium gluconate gel are located.

- Small spills contained in a hood, we can clean up. In the event of a large spill or accident, call x555.

- All HF exposures much be treated as a medical emergency. Flush the exposed area with water until medical help arrives.

- All chemical waste is collected in labeled containers and picked up as hazardous waste. Understand the procedures for collecting, labeling and disposing of your waste.

- Empty bottles must be thoroughly rinsed out. Fill the bottle with water in the hood to avoid breathing vapors, and then rinse out at least 3 times in the sink. Deface the label, and write very clearly on the bottle, “RINSED”.

Phosphoric Acid Boiling

Samples are boiled in O-phosphoric acid to dissolve a whole host of minerals in many rock types.

- Always plug the hotplates into a variable transformer (the Variac) to control the temperature (it controls the power to the hotplate). The hotplate should be set to 10 when plugged into the Variac.
- Set the hotplates on a large fiberglass spill tray.
- Check the beakers thoroughly for cracks.
- Clearly label your beakers.
- Be very careful of cross contamination if you are boiling more than 1 sample.

1. Weigh up to 80 g. of non-magnetic sample into 1000 ml beakers. Weigh the sample directly into the beaker in the fume hood both to avoid both inhaling dust and contaminating the lab with dust.

2. While in the fume hood, add some DI-H_2O to each beaker (to keep the dust down). Then, at the sink rinse them thoroughly with DI-H_2O to wash off the fines. (You don't need to use MQ-H_2O for this.)

3. In the fume hood, add 400 ml of concentrated (85%) O-phosphoric acid to each beaker and cover the beakers with a watch glass. Set the hotplate to about 325°C and monitor the hotplate temperature using a surface thermometer. Bring the samples to a boil. The boiling can be very vigorous at first, so you must stay in the lab until it has reached a steady rolling boil. Make sure the vigorous boiling isn't causing the beakers to "walk" off the hotplate. After about 1 hour the boiling will become gentle. Boil for 1 – 2 hours longer (or until the volume reaches about 300 ml). After a while, usually a total of about 2-3 hours, the rolling boil subsides and the surface can become quite flat. This is a good time to take off the samples.

   CAUTION: Sometimes when the sample has boiled too long the acid will become very thick and jelly-like. (It seems it happens more w/ samples that have a lot of fines and organics, such as lichen from the surface of the rocks – another reason to rinse well.) To reduce the amount of sample lost in this thick gel, pour it off before stirring the sample up and suspending it in this dense liquid. If the samples boil for too long, a dense gel can form which can be difficult to remove without losing a lot of sample. If this should happen, read about "What to do if your sample gel's" below.

4. Remove the beakers from the hotplates and place them on heat resistant tiles. You can remove the watch glasses so they cool faster, but then rinse them with DI-H_2O into a container in the hood. Do not squirt water into the hot acid! Let the samples cool for about an hour. The acid may form a gel around the sample and on the side

of the beaker (this film of supersaturated silica solution), which will dissolve during the sodium hydroxide cleaning.

5. Once the beakers are cool (lukewarm is ok), pour off the acid into the Phosphoric Acid waste container. In the hood squirt down the sides of the beakers with ~200 ml MQ-H_2O and stir the samples with a clean metallic micro-spoon spatula. Allow the samples to settle and decant the water off into the waste container. Then add another 500 ml of DI-H_2O. You can now take the samples over to the sink without risk of inhaling acid fumes. Rinse them 3 or more times with DI-H_2O in the sink.

6. Add 300 ml DI-H_2O to each beaker. In the fume hood, add 100 ml 50% NaOH (sodium hydroxide) to each beaker. The NaOH will dissolve the silicate coating around the quartz grains left by the phosphoric acid leach. Cover the beakers with the watch glasses and boil for ten minutes. (Use the same watch glass for the same sample as before, otherwise thoroughly rinse off any sample grains so as to avoid cross contamination of your samples.)

   DO NOT LEAVE THE SAMPLES! At this step the boiling is usually very vigorous and beakers can "walk" off the hotplate! If the boiling is too vigorous, reduce the heat. After 10 minutes, place the beakers on the heat resistant tiles and allow them to cool (about 30 minutes). You can remove the watch glasses immediately, rinsing the lids directly into the beaker. Once cool, pour off the solution into the NaOH waste container. Rinse w/ ~100 ml DI-H_2O and pour off into the waste container and then rinse three times with DI-H_2O water and in the sink.

7. Either proceed directly to the HF/HNO_3 leaching step or dry the sample in the oven overnight. If you are drying the samples, transfer them to small beakers, combining the same sample into one beaker. Once the sample is dry let it cool, weigh it and record the weight in the notebook. Cover the sample with parafilm. If you are going directly to the HF step, combine 2 beakers of the sample into each bottle for the leaching step on the shaker table.

BEAKER CLEANING:

Scrub the beakers in the sink using a brush or sponge if necessary and rinse thoroughly so that no samples grains remain in the beakers. If they are really filthy, you can soak them in a soapy solution. Use MQ-H_2O for the final rinse. Dry beakers on the drying rack.

Hydrofluoric/Nitric Acid Leach

Samples are leached in a dilute hydrofluoric/nitric acid solution in order to dissolve minerals other than quartz and to remove meteoritic ^{10}Be.

Samples are generally leached twice in 1000 ml of a 5% HF/HNO_3 solution and placed on the shaker table, each time for 1 day, and once in a 1% HF/HNO_3 solution in a heated ultrasonic bath for 24 hours. Some samples require additional leaching steps before they are sufficiently clean.

SHAKER TABLE LEACH

You can put ~150 g. of sample in a bottle, though this will vary from sample to sample. Most samples dissolve a lot after the first leaching step, but you might want to adjust the amount of sample for sample types that don't dissolve as much at this step.

1. If you use the narrow mouth bottles, use a funnel. Always use a clean funnel to put the acid into the bottles, but when moving the funnel between bottles containing different samples, make sure you are not transferring sample grains between bottles.

2. For a **5% HF + 5% HNO3 solution** –

   Add 850 ml MQ-H2O.

   Then, working in the fume hood, add 100 ml concentrated (49%) HF and 50 ml concentrated (79%) HNO3. (Use certified A.C.S. grade.)

   NOTE: **ALWAYS ADD WATER FIRST! NEVER ADD WATER TO ACID!**

3. Place the bottles on the shaker table overnight. Make sure there are no drips of acid on the sides of the bottles. The samples do not need to be on for a full 24 hours. If you put them on in the afternoon, it is ok to change them the next morning.

For a **5% HF + 2% HNO3 solution**, use 875 ml MQ-H2O, 100 ml HF and 25 ml HNO3.

RINSING

4. In the hood, pour the acid solution into a properly labeled waste container being careful not to pour out your sample. Use a funnel to reduce dripping.

5. While working in the fume hood, add ~1000 ml of water to each bottle. Shake them vigorously, and then decant the water into the sink, again being careful not to spill any sample. (It is dilute enough that you can work outside of the hood.) Rinse the

samples two more times filling the bottles about a third of the way and shaking them vigorously each time. (The vigorous shaking breaks up weaker feldspar grains.)

6. Repeat this shaker table leach step for a total of 2 leaches.

ULTRASONIC LEACH in 1% HF 1% HNO3

1. Transfer the samples directly from the 2000 ml bottles into labeled 4000 ml bottles. We generally put in about 50g per bottle. (You can approximate the amount. It's not worth drying them first.)

2. Fill the bottle with 3800 ml MQ-H2O

3. In the hood, add 80 ml HF and 60 ml HNO3.

4. Put the lids on tight when putting into the ultrasonic bath.

5. Fill the bath to the brim with DI-H2O.

6. Turn on both the sonicator and the heat, but leave the heat off overnight. The water evaporates too much overnight when the heat is on. You will need to check the level of the water from time to time. Even without the heat on the water will evaporate. Keep it full to the brim.

If you are keeping the samples in the 2000-ml bottles, adjust the amounts accordingly.

1,900 ml MQ-H2O, 40 ml HF and 30 ml HNO3.

RINSING

7. Remove the bottles from the bath and allow them to cool for about 30 minutes.

8. Decant the acid into a waste container. Four bottles will completely fill 1 blue waste container. These are a little trickier to pour, so be very careful, and pay attention to where your sample is. It is not as easy to pour your sample out as it may appear, but still it carefully.

9. Under the hood, fill the bottles ½-way with MQ-H2O to rinse. You do not need to pour this rinse into a waste container. This is already sufficiently dilute for the sink (do the math, a few ml 1% HF and HNO3 diluted to 2000 ml).

10. As with the small bottles, shake these up vigorously, decant into the sink and repeat for a total of 3-4 rinses.

11. Transfer sample into a very clean and labeled beaker for storage and cover with Parafilm™. Do not put the Parafilm™ on a hot beaker or it will melt!

BOTTLE WASHING

Make sure you remove all sample grains from the bottles before adding a new sample!

Rinse the bottles thoroughly using the water straight from the DI line. You can turn the bottle upside down and forcefully clean off any grains that may be stuck to the bottom and sides. Give the final rinse with MQ-H2O. Once your bottles are cleaned, remove all labels and put them away. Only bottles without labels are assumed to be clean!