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Separation & Preparation of Biotite and Muscovite samples for 40Ar/39Ar analysis

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When dealing with detrital samples, sample contamination is a big problem. To avoid this *at all costs* be clean, this means handling samples one at a time and cleaning all equipment entirely after each sample handling. Only handle samples in a quite (i.e. not windy) environment, where there is little chance of spilling or blowing samples away. Handle samples over clean copier paper, and change the paper after each sample. Use compressed air (either canned or from a compressor) to clean all equipment in separation stages and methanol to keep equipment dirt and dust free in the preparation stages. This can be often be tedious work, I recommend finding a good book on tape or podcast to listen to. Estimate times to completion are stated.

Detrital Samples

Separation

I. Sample drying (1-3 days)

For detrital samples it is likely that samples will be wet. Simple let the samples dry in the open air, or under a *mild* lamp on paper plates. It may be necessary to occasionally stir up samples to get them to dry faster, if you do this, clean the stirrer after each sample. Split samples using a riffle splitter to obtain a quantity for the rest of the process, do this in the rock room.

II. Sample Sieving (3-7 days)

First locate a set of sieves that can be intensively cleaned. There are a set of appropriate sieves in 317 for this use, be careful using other's sieves as you will likely bend the meshes during cleaning. A stack of >2 mm, 1-2 mm, 0.5-1 mm, 0.25-0.5 mm, and < 0.25 mm sieves is usually appropriate. Meshes finer than 0.25 mm are absurdly difficult to clean. Load sample into sieves and run of a Ro-tap or similar sieve shaker for ~10-15 minutes. Meanwhile use compressed air to carefully clean workspace, and lay down fresh copier paper. Mark and bag each GS separate. Carefully clean the sieves so that there are no grains left in the meshes or crevices around the meshes. There are wire brushes and picks in 317 and the rock room to do this. Go back again and make sure each sieve is clean, this should take the majority of your time. Re-clean workspace and change paper.

Repeat for new sample.

III. Papershaking (2-4 days)

This is more of an art than a science. Some like to use blotter paper to do this, it is available in 317 in a variety of sizes; John Stone mentioned to me that the Australians use watchglasses, these are also available in 317. You are best off experimenting a little at first what works best for you, I will briefly describe my preferred method. the object here is to isolate flat particles, this gets rid of most of the quartz and feldspars, but does not leave you with 100% micas.

I prefer to start with the largest grain size and work down. The 1-2 mm size fraction is easy, the 0.5-1 and 0.25-0.5 are what you will probably use for analysis (so make sure you have a large sample of the these fractions), and the <0.25 are difficult and annoying, but may be necessary. This is a game of diminishing returns, once you have enough sample separate (several hundred grains, preferably) you can stop; remember the more sample you isolate here, the less likely you will have to return to this step.

For the shaking I like the employ the "Cascade Method." First I take two circles of blotter paper (large or small, personal preference) and two fresh sheets of copier paper. One of the sheet will hold the "waste" and one will hold the flat particles, do not actually throw away the waste, simply keep it is a separate container. Put an eraser size (or less) amount of sample on the small circle, tilt the circle slightly and watch the "round" particles roll preferentially off the paper. To prolong this rolling effect, spin the paper slowly while still keeping it at an angle. Do this over the other circle and one of the sheets of copier paper, allowing the "waste" particles to stay of the copier paper and switch back and forth between the two blotter circles, periodically knocking the flat particles that stick to them onto the other sheet of copier paper. If you prefer, you can exchange the two blotter circles for watchglasses. Also, the static electricity from the plastic container bags is an excellent separator, I have been trying to obtain a van de graff-type device to experiment with this, if you are interested please let me know. Experiment.

Again, switch out fresh papers between grain size fractions and clean everything thoroughly between samples.

IV. Frantzing (1-3 days)

This step separates magnetic (biotite, etc) from non-magnetic (muscovite, etc) flat particles. Use the Frantz Isodynamic Magnetic Separator up in the mineral separation lab across from the cosmogenics lab on the 4th floor. There is a Carpco as well, I have not tried this, if you do, good luck. If you have not used a Frantz separator before, please see me for details. After determining the angle and voltage for most efficient separation (this is sample dependent, I usually use ~0.5 V (?) and angles of ~12 and 12 degrees) run the sample. Notice that the two containers at the end of the chute contain either magnetic or non-magnetic separates; leave the magnetic bin and re-run the non magnetic bin 3 times, then do the opposite. This should provide pretty distinct biotite and muscovite separates. Clean the chute with compressed air after each GS run, and double check there are no grains mixing between samples.

V. Picking/Brushing (4-10 days+)

This is the most tedious step. Hopefully by now your samples separates are nearly all biotite or muscovite of their respective grain size. This object of this step is to ensure they are at least 98% pure. Use the "Pic-Mic" table or similar device to assist you. To use the Pic-Mic table lift up the marble top and place a clean sheet of copier paper underneath. Adjust your chair height so that the top of the table is near eye level, you want to be able to the the grains up close. Take a sample separate and place the grains between the holes in the marble. Use a fine brush or tweezers (I prefer brushes) to knock everything that is not a mica into the holes. This also includes chloritized micas, which hopefully are sparese in your sample. This takes patience, and you will get better at it with time. when finished, carefully lift the marble up, pull out the non-mica "waste" and save in the "waste" container. Place a fresh sheet of copier paper under the marble. Sweep in all of the micas on top of the marble, and voila! A biotite or muscovite sample ready

for cleaning.

Use the air compressor to clean everything, including the table and marble top, between samples and when you are finished.

Preparation

VI. Cleaning (1-2 days)

Make sure your samples are in little glass vials with the name of the sample permanently written on the cap, which should stay out of the cavitating water.. Cleaning occurs in an an ultrasonic bath, so anything written on the outside of the vial is usually cleaned off. Make sure the ultrasonic bath is filled to the working level. Fill each vial with DI water in an amount proportional to the amount of sample, ~2/3 full if the sample is ~1/4 of the vial; enough to clean the sample. Place the samples in the cleaner and run for ~5-10 minutes, securing the vials with a rubber band is often helpful. Small grains may dissolve in DI water is the sample is run longer than 5 minutes, so be careful. Decant the dirty water, or use an eyedropper making sure to no spill out any grains. Repeat until the water is clear, then repeat with methanol. Make sure to collect the methanol waste and store in the "Methanol Waste" container in the lab. Allow the cleaned samples to dry in their vials over night.

VII. Making packets (1 day)

The object of this step is to make the small aluminum "packets" we will load samples into for irradiation. You should first gather all of the necessary materials for making the packets and set up an assembly line for putting them together. Practice making a few before actually keeping them for packets. Try to make several hundred, they are easy to make all at once, and will last. Use the thin variety of aluminum foil, the normal kitchen stuff. Thicker foil is a little easier to work with, so if you have the option you might consider this, however ASU requests thin foil. Cut off a sizable portion, perhaps half the size of a half sheet of paper, making sure not to crinkle it too much. You can use a razor blade, or the roller-cutter to cut the foil, cut slowly as not to crinkle. Put on latex gloves. Wipe everything down with methanol, this includes the 3 mm diameter rod (the one with the red tab), the aluminum brick, aluminum packet holder, any scissors, tweezers, razors you use. Wipe down both sides of the foil piece. Cut small squares out of the foil piece using the roller-cutter, these should be ~0.5 - 0.75 in. square. wrap them around the end of the rod tightly (but not too tight!) so that they form a hollow cylinder. This can be difficult to do in gloves, so practice. Nudge the cylinder off the end of the rod about ~2-3mm. Use the fines tweezers available (your picking tweezers) to fold in the sides, making two tabs, then fold these in making a base for the cylinder, gently tamp this end closed in the sample holder against the aluminum brick (use the scratched up hole 2nd from the end, it's pretty obvious). check the end to make sure the foil hasn't ripped. This makes a nice little tube. Use tweezers to put the tube in a clean beaker. Repeat to make several hundred tubes. Sonicate the tubes in methanol for 5 minutes or so, watch that they don't begin to indent due to the cavitation. Let them dry completely.

Keep everything clean, hydrocarbons from your greasy fingers can screw up mass spectrometers something awful.

VIII. Packeting (1-2 days)

In this step it may be helpful to have a picking microscope available, make sure this scope provides plenty of room for hand movement under the oculars. Wipe all instruments down again with methanol, and put on a new pair of latex gloves. If you use a scope, wipe the stage down as well, just in case. Use tweezers to put a clean packet in the packet holder on top of the aluminum brick. Tap a few grains into the scoopula/sample holder, you can really only load 20-80 grains per packet (depending on grain size), so you may need to make several packets per sample separate. Inspect the grains one last time under the microscope, pick out anything that doesn't look like a mica and make sure to avoid chloritized micas. Carefully load the packet with the grains. Use the tweezers to seal the packet just above the height of the sample holder, snip off the top of the packet with scissors. You will need to open this packet again and at that point it will be radioactive, so carefully fold a little tab over and tamp the sides down with the 3 mm diameter rod. The irradiation tray for ASU has holes that are 4 mm in diameter and 3 mm deep, 3 mm is ~half as deep as the thickness of the sample holder. Measure the height of the packet to make sure it is 3 mm. If it checks out, put the packet into a plastic vial and label the vial. Repeat for all samples, making sure to clean everything up between samples (particularly the scoopula) and wipe down with methanol periodically. Weigh each packet precisely (to 0.001 at least) and record these masses in case you need to identify an unknown packet later on.

These packets are ready to be irradiated. Make sure to fill out a sample sheet to send out with your samples! Most facilities have their own, let me know if you want a copy of ASU's.

Bedrock Samples

Bedrock samples must first be reduced to sediment. This can be done in a river, or to save time, use a jaw crusher and disc mill following standard rock crushing techniques. A wonderful, illustrated guide to using these machines is available at the Union College Geology website, under Department. Generally, try to preserve the largest mineral grain size possible. The output of this process is a (hopefully) sand-sized sediment that follows the aforementioned procedures with a few important differences:

1. Depending on the mineral/grain size of the bedrock the grains produced in the sediment may be very small. Stick with ~100 microns or greater, as such a dimension is plenty large enough to avoid possible effects of 39Ar recoil on age spectra.

2. Bedrock grains are often analyzed via step heating, so you won't have to open the foil packets once you package them. For this reason it is not as important that you are able to open the packets, so you don't have to be as careful folding them shut. Still, it pays to be careful when making these packets so they don't rupture. Also for this reason, bedrock samples must be 100% pure mica (of uniform grainsize) before loaded into the packets.

3. Measure the mass of the bedrock sample, not just the complete package. You can do this by balancing the empty (snipped) packet first, and then the total packet afterwards. This requires a very precise scale, up to 0.0001 precision.

Author's Note: These notes are compiled from instructions given to be by Kate Huntington, Jo-Ann Wartho and from readings.