XRD Sample Preparation for a small amount of material

There are two techniques for XRD analysis to mount small amount of material depending on the volume of material available. Separating a vein or other spot can be done by using a hammer, or other spatula or tool, to chisel out only the parts you want. Small flecks of the other parts of the sample are okay to have mixed in with the material of interest, but do your best to pick these out with tweezers or a brush.

Preparing Mount for Small Amount of Material

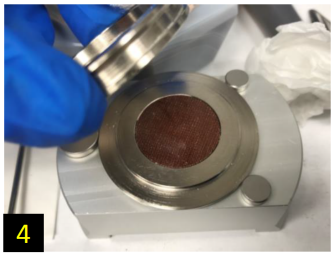
Note: Sample mounting on Figure 1 is for backloading sample holder of the Aeris XRD. Similar technique should be used for frontloading sample holder of the Bruker XRD. In case of Bruker XRD, spacer is put first.

1. Freeze dry the sample prior to analyses (for about 12 h). It helps to collect sample material of interest (e.g., mineral vein in hard rock samples) (Figure 1, Step 1).
2. If enough material, grind the sample to a talc-like powder (<0.062 mm) (Figure 1, Step 2).
3. Place a small amount of sample powder in the center of a sample holder (Figure 1, Step 3). Spread evenly and make the surface flat (using small cylinder). Place a cardboard spacer on top of the sample (Figure 1, Step 4).
4. The sample is now ready for analysis (Figure 1, Step 5 right).

1. Separating material of interest (vein on the picture) from bulk sample

2. Grinding material to a talc-like powder

3. Mou holder Aeris XI



4. Placing a cardboard spacer

5. Mounting small amount of material: Comparison Slurry (left) vs. Powder (right)

Figure 1. Preparing mount for small amount of material (here, preparation for Aeris XRD)

Sample Slurry/Smear Slide Mounting for Small Sample Amounts

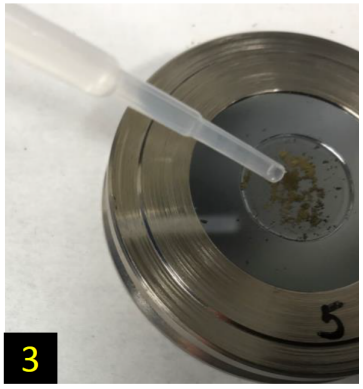
The following technique is modified from the *U.S. Geological Survey Open-File Report 01-041, A Laboratory Manual for X-Ray Powder Diffraction*. For a very small amount of sample material (i.e., end of a tooth pick), samples may be ground to a fine talc-like powder and smeared onto one of the quartz disk or zero background inserts. Although not useful for semi-quantitative analysis, this method is useful for rapidly determining bulk mineralogy.

Note: Sample mounting on Figure 2 is for frontloading sample holder of the Aeris XRD. Similar technique should be used for frontloading sample holder of the Bruker XRD. Two quartz disks will adequately fill the Bruker sample holder. The upper quartz disk will be used for the slurry.

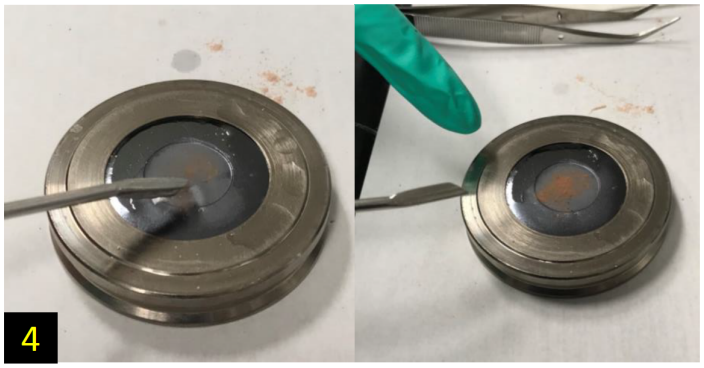
1. Freeze dry the sample prior to analyses (for about 12 h). It helps to collect sample material of interest (e.g., mineral vein in hard rock samples).
2. Use tools to scratch the surface of interest (Figure 2, Step 1) and use mortar and pestle to grind uniformly.
3. Place a small amount of sample powder in the center of the zero background silicon disk (Figure 2, Step 2). See XRD Sample Preparation Clay Separations for how to mount the sample holder.
4. With a disposable glass or plastic Pasteur pipette or eye-dropper, add 2-3 drops of 70 % isopropyl alcohol or distilled water to the sample (Figure 2, Step 3). *Note: Isopropyl dries faster than water.*
5. Spread the sample to a thin layer using the tip of the pipette or eye-dropper. Alternatively, use a glass rod or Teflon spatula. The slurry should be spread evenly across the disk. If there is not enough material to cover the entire disk, concentrate the sample in the middle of the disk where the X-rays will contact the sample (Figure 2, Step 4). You can also use a piece of tape, stick it to the sample holder and sprinkle the ground sample onto it.
6. Place the sample in the desiccator to dry before running in the XRD (Figure 2, Step 5). See Figure 1, Step 5 left for the final product.

1. Scratching the surface of interest (vein on the picture)

2. Putting sample powder in the middle of a zero background Si disk



3. Adding isopropyl alcohol wi an eye-dropper



4. Spreading the sample to a thin layer

5. Drying in a dessicator

Figure 2. Sample Slurry/Smear Slide mounting