Mounting Samples

March 30, 2017

Chemical Crystallography Laboratory

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Distribution

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Revision Record

Date	Version	Responsible Person	Description of Change
1/4/2013	3.001	Douglas R. Powell	Initial Release
1/30/2013	3.002	Douglas R. Powell	Minor revisions
3/30/2017	3.003	Douglas R. Powell	Minor revisions

The following laboratory users have read this manual.

Name	Signature	Date

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A. Scope and Availability

This manual describes mounting samples for single crystal data collection at the Chemical Crystallography Laboratory at the University of Oklahoma. These procedures are publicly available from the Laboratory's web site.

B. Summary of Method

These tasks include mounting samples for room temperature and low temperature data collection as well as mounting air sensitive samples.

C. Responsibility

Any problems with performing these steps should be immediately reported to the Laboratory manager.

D. Safety and Training

Personal Protective Equipment for sample mounting must be determined by the properties of the sample and solvents in use. Appropriate gloves are often needed.

E. Equipment and Supplies

- Goniometer head.
- Sample to be analyzed.
- Crystal mounting tools and microscope.
- Adhesives such as super glue, 5-minute epoxy, Paratone N oil, or stopcock grease.

F. Procedure

Before performing these tasks, be sure that only the sample to be mounted is on the table with the microscope. No other samples should be on the table or microscope. Also, be sure to read information about the sample and note any solvents that may be present or other hazards that may be present.

1. Clean the probes, cutting blades, and glass slide that you plan to use to mount the sample. Items are typically cleaned by simply wiping or scraping the tools, but solvents such as Windex may be useful. If the sample is air sensitive, be sure to dry all of the tools and glass slide.

2. Move some pieces of the material onto a glass slide. Reseal the vial with the remaining material. Place the slide on the stage of a microscope and examine the pieces using the microscope.

   Good crystals typically have flat faces, sharp, straight edges, and no cracks or inclusions. Most synthetic and natural product (biological) crystals are transparent. Opaque pieces of these organic materials may have a coating or may have decomposed. A variety of minerals, metals, metal chalcogenides, and inorganic metal complexes are naturally opaque materials.

3. Touch one of the pieces with a probe while observing that piece through the microscope. If the probe enters or changes the shape of the piece then the material is a gel and will probably not diffract X rays.

4. Pieces that have reentrant faces are most likely twinned. Try to cut an obvious twin to produce a single crystal (see step 6 below).

5. Test for possible twinning by using the polarizing lenses of the microscope. Place the upper polarizing lens on the microscope, increase the intensity of light coming through the lower light source and direct the upper light source away from the sample, rotate the upper polar until the background is nearly black, rotate the sample stage until the sample extinguishes (goes dark). This transition between seeing light through the sample and seeing no light should occur within a very narrow angular rotation of the sample. If part of the piece appears to extinguish, but another part of the piece transmits light then the piece is a twin, and the twin boundary should be visible. Try to cut the sample along this twin boundary. If possible, rotate the sample onto another surface of the piece and repeat this test. Typically this effect will occur about every 90° of rotation of the sample.

   Note that gels and glasses also do not exhibit this effect. Also, samples that have cubic symmetry do not exhibit this effect. Tetragonal, hexagonal, and trigonal samples viewed along their *c* axis will not exhibit this behavior because the axes in the two view directions have the same coefficient of extinction. Thus the absence of this extinguishing effect suggests higher symmetry in the crystalline samples.

6. Select a piece to examine using the diffractometer. The piece should have no dimensions greater than 0.8 mm. However, samples that give the best results typically have all dimensions near 0.5-0.6 mm. Samples with very heavy atoms such as iodine, mercury, or actinides should have no dimensions greater than 0.4 mm.

   Cutting a sample piece is performed with a razor blade, knife, or scalpel. Before the cut, decide the location for the cut. Successful cuts are usually either parallel

or perpendicular to an existing face. Place one point of the blade on the slide near the chosen piece and align the blade with the path of the cut. Gently lower the rest of the blade until the piece is cracked; then remove the blade. Coax the crack to continue through the piece with a probe.

If the blade appears to mash or flatten the material, then first reexamine the bulk sample for a piece that does not need to be cut. If none can be found, then find another large piece of the material and use a new blade to make your cut. Choose the cut path to make the piece about 10% larger than needed. Mashed parts of the material can often be removed by gentle pokes with a probe.

7. Attach your piece to a mount. Mounts can be made of a glass fiber, a glass capillary, a plastic loop, or a plastic support (E.g., Mitigen). The ideal crystal mount rigidly holds the sample in the X-ray beam with as little other material as possible. The mount is then placed on a goniometer head.

   If the sample is being mounted on a glass fiber, position the smallest surface of the sample on the end of the fiber. If the sample is being mounted on a plastic loop or a plastic support, place the sample on the outer end of the loop or support.

   Adhesives such as super glue, 5-minute epoxy, or Elmer's glue should be used if the sample will be examined at room temperature on the diffractometer, and greases or oils such as Paratone N, mineral oil, or stopcock grease if the sample will be cooled to a low temperature on the diffractometer. Use the smallest amount of adhesive that is needed to hold the sample in place.

   If the sample contains perchlorate ions, do not use a plastic loop. Perchlorate ions destroys plastic loops.

8. Clean all tools after the sample has been mounted.

9. If the sample is air or moisture sensitive, then mount the sample in a dish filled with argon gas, or a glove bag filled with nitrogen or argon gas. If mounting is taking place in an argon dish, then be sure to wear gloves to keep the moisture from your skin away from the sample. Mounting must be performed quickly, so using a glass fiber mount with stopcock grease adhesive will usually give the best results. Oils should not be used to mount air sensitive samples, because both water and oxygen can be adsorbed by the oils. Place the dish/microscope or glove bag/microscope as close to the diffractometer as is reasonable to minimize the amount of time the sample is in air before being cooled on the instrument.

10. If the sample is radioactive then be sure to wear gloves to perform the mount. When the mounting is complete clean all tools used with a wipe and place the wipe in the box for disposal as a radioactive material. When the diffraction experiment is finished, use a wipe to remove all remaining sample to the disposal box.

G. Records Management

Users are responsible for recording the type of mount used for later publication of the crystal structure of the sample. Equipment malfunctions are to be reported to the lab manager and recorded in a Maintenance Log book kept by the lab manager.

H. Quality Control / Quality Assurance

Selecting a large, single crystal will produce the best quality intensity data.

I. References

None.