Notes on using a single crystal diffractometer for powder diffraction experiments.

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A typical powder diffraction instrument operates in reflection mode. Sample preparation involves creating ~0.5 g powder. With the single crystal diffractometer the geometry involves transmission mode. Much less sample is required, and good resolution can still be achieved. However, absorption can be an issue because the experiment is in transmission mode. We have used two types of mounting systems for powder:

1. Capillary tubes. Requires tamping of sample into tube. This can be difficult, sometimes resulting in the breakage of tubes. Additionally the powder often adheres to the side of the capillaries, making it difficult to visualize if the powder completely fills the tube.

2. Polyethylene loops with superglue. These loops are the typical mounts used for protein crystallography available from Hampton Research. For minerals, the powder is ground and then mixed with superglue with the loop. For metals, a small shard (20-100 μm) is filed off the source metal and glued to the loop. The glue creates a relatively large background a low scattering angle, which is relatively smooth and can be easily removed in the data analysis programs.