# 1 Bruker D8 Advance ECO: Slit optimization for low angle measurements

## 1.1 Sample and preparation

The sample used in these measurements is a powdered metal-organic framework from the Phillip Milner group, identified as Mg-MOF-274. It is a yellowish powder, and is very static sensitive. I had a difficult time removing powdered material from a scintillation vial and packing into a quartz sample well due to static repulsion. The grain size was also fairly large (observable by unaided eye). I therefore mixed the sample will a lesser but comparable volume of corundum powder and crushed them together with a mortar and pestle. The mixture had a fine grain size and exhibited no static repulsion. I loaded it into one of the small single crystal quartz wells with 0.2 mm depth and 5 mm diameter. After loading the sample into the diffractometer, the knife-edge was lowered near the sample surface (less than 1 mm away).

## 1.2 Detector slit

Using a small divergent beam slit (0.1 mm) and 1.0 mm PSD opening, I measured powder diffraction over the $2\theta$ range of $2 - 15°$ using a range of detector slit settings. The guidelines I had been working with stated that for high angles, the detector slits should be set to 9 mm, and for low angles a value of 3 mm used. The detector slits appear to have no affect over 2.5 mm. Below that value they decrease the signal and in some cases improve the background level. Figure 1 shows that below $2\theta = 4.0°$, detector slits of 1 mm give significantly lower background compared with 2 mm and larger slits, at least when using narrow source slits.

## 1.3 Divergent beam slit

I measured a range powder patterns using divergent beam slits of {0.1, 0.2, 0.6} mm and detector slits of {3, 9} mm. Figure 2 shows these scans, normalized by their divergent beam heights. They all show nearly the same peak positions and background profile. The primary difference among them is the height of the peak near $2\theta = 3.67°$ relative to the peak near $2\theta = 6.30\circ$ for the two measurements with a divergent beam size of 0.6 mm. It is more likely that the smaller beam sizes give an accurate ratio of the intensities (*i.e.* what would be measured with a small beam of low divergence using a detector far away).

## 1.4 Summary

- Any detector slit setting larger than 2.5 mm is equivalent to 2.5 mm.
- Below $2\theta = 6°$, the divergent beam slit should be reduced from 0.6 mm to 0.2 mm to measure the relative intensities of peaks accurately.

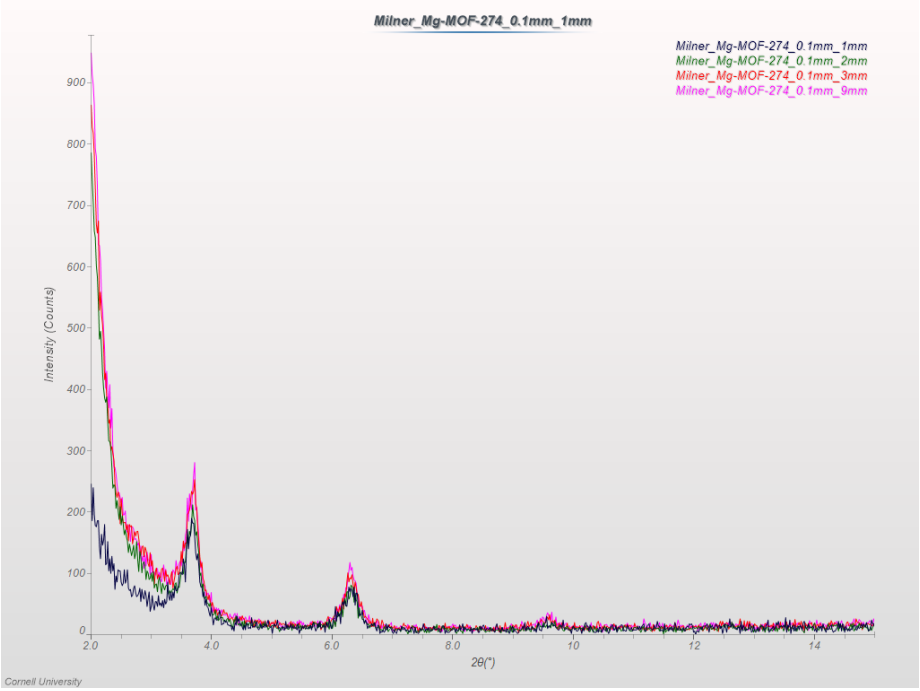


Figure 1: Milner-Mg-MOF-274 powder pattern measured using narrow incident slits (0.1 mm) and a range of detector slits. The 1 mm detector slit data is scaled 2x.

- Below $2\theta = 3.5 - 4.0°$, the divergent beam should be reduced to 0.1 mm and the detector slit to 1.0 mm to reduce background scattering.

Note that these are not strict requirements. For example, if expect your first peak to appear at 5 degrees and want to start a scan at 4 degree, using a divergent beam slit of 0.6 mm and detector slit of 2.5 mm should be safe - possibly resulting in a higher baseline than if narrower slits had been used, but not affecting the position of the peak near 6.

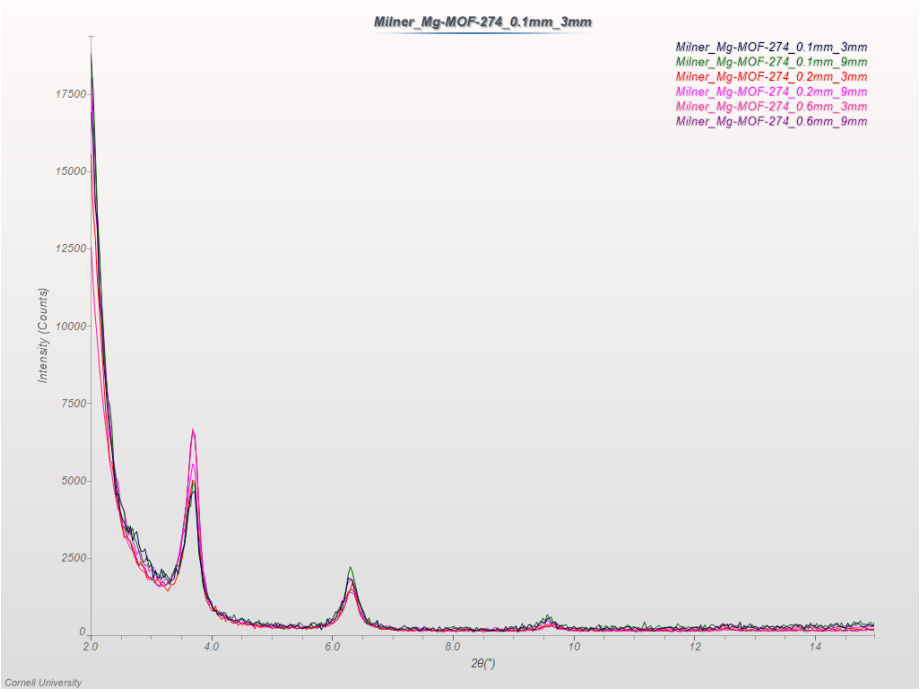


Figure 2: Milner-Mg-MOF-274 powder pattern measured using a range of source and detector slits. All patterns are normalized relative to the source slit height.