# Reducing the background of ultra-low temperature X-ray diffraction data through new methods and advanced materials

C.J. McMonagle[1]*, M.R. Probert[1]

[1]*Newcastle University, Chemistry, Kings Road, Newcastle upon Tyne, UK*

Keywords: ultra-low temperature, technique development, etc.

*e-mail: Charlie.McMonagle@ncl.ac.uk

The XIPHOS diffraction facility at Newcastle University has been designed to broaden the range of extreme sample environments available in the home laboratory. [1] One of these extreme conditions is the ultra-low-temperature regime, with temperatures as low as 3 K routinely accessible. In order to reach these ultra-low temperatures, the sample is cooled using a three-stage Displex closed-cycle refrigerator and must be kept under vacuum and maintained inside a thermal radiation shield.

The vacuum chamber is provided by a cylindrical beryllium shroud which, while mostly transparent to X-rays, result in contamination of the diffraction pattern by characteristic beryllium powder rings. Additional powder rings are contributed by the thermal shield of beryllium or aluminium foil, which results in an intense and complex background of overlapping powder patterns from at least four surfaces.

These powder rings are not smooth or consistent from one orientation to the next and as the beryllium contains randomly distributed larger crystallites making simple background subtractions inadequate. In the case of weakly diffracting crystals, the irregularities in the background can be of the same order of magnitude as the sample reflections, making the extraction of accurate observed structure factors difficult.

Here we present new methods and advanced materials that significantly reduce the background when collecting single-crystal X-ray diffraction data at ultra-low temperatures using a closed-cycle helium refrigerator. These include a magnetically controlled internal beamstop and a separate internal collimator that together completely remove the scattering contribution to the background from the beryllium vacuum chamber. Additionally, a new radiation shield made from flexible graphite significantly reduces the background and maintains excellent thermal properties. In combination these improvements have led to a 6-fold reduction in the average intensity and a 15-fold reduction in peak intensity of the background observed for diffraction experiments conducted with a closed-cycle helium refrigerator. Moreover, access to ultra-low base temperatures, 2.05 K, has been maintained.

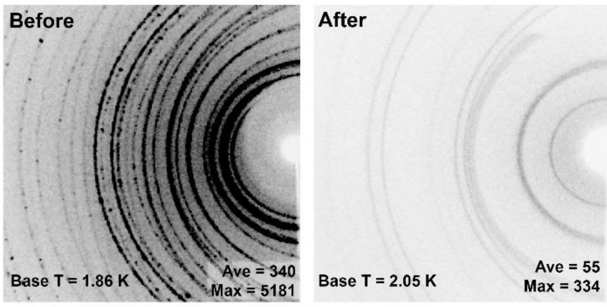


*Figure 1*. Two frames (20 s exposure time, 0.5° rotation) with the same colour scaling before and after improvements. Insets show the base temperature achievable with the respective setups and the average and maximum intensity on the image.

**Acknowledgments**: This work was supported by the Engineering and Physical Sciences Research Council (grant No. EP/N01331X/1)

[1] Probert *et al*. *J. Appl. Cryst.* 2010, **43**, 1415