

Powder Diffraction Data from Microfocal-Source Single Crystal X-ray Diffractometers

The Synergy-S and the D8 Venture diffractometers have been designed for collecting single crystal data. However, the multilayer optics deliver a highly focused and concentrated beam at the sample suitable for powder diffraction in transmission mode. Also, we find that there is very small beam divergence away from the sample, and therefore these diffractometers can give good quality powder diffraction data. The primary advantages are the speeds of the data acquisition, that very small amounts are required and of course that a dedicated powder instrument is not required for the applications. Biological materials, paracrystalline and fiber samples can be investigated due to the intense beam.

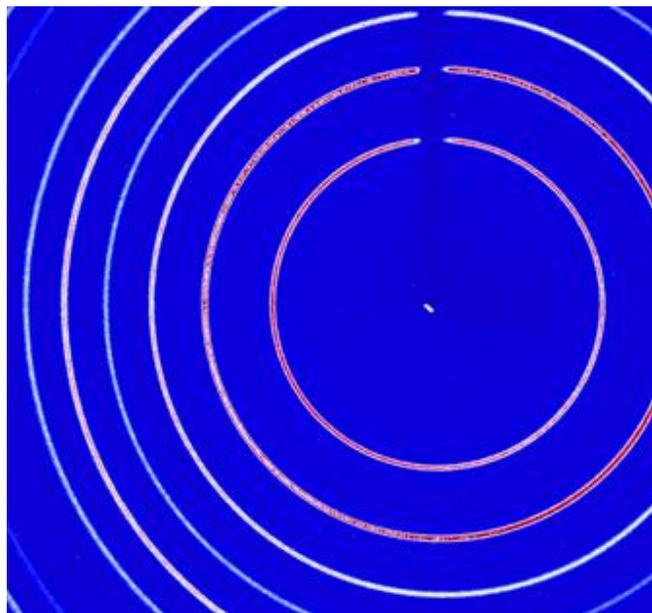


Figure 1 The diffraction pattern from the LaB6 standard using the single crystal instrument.

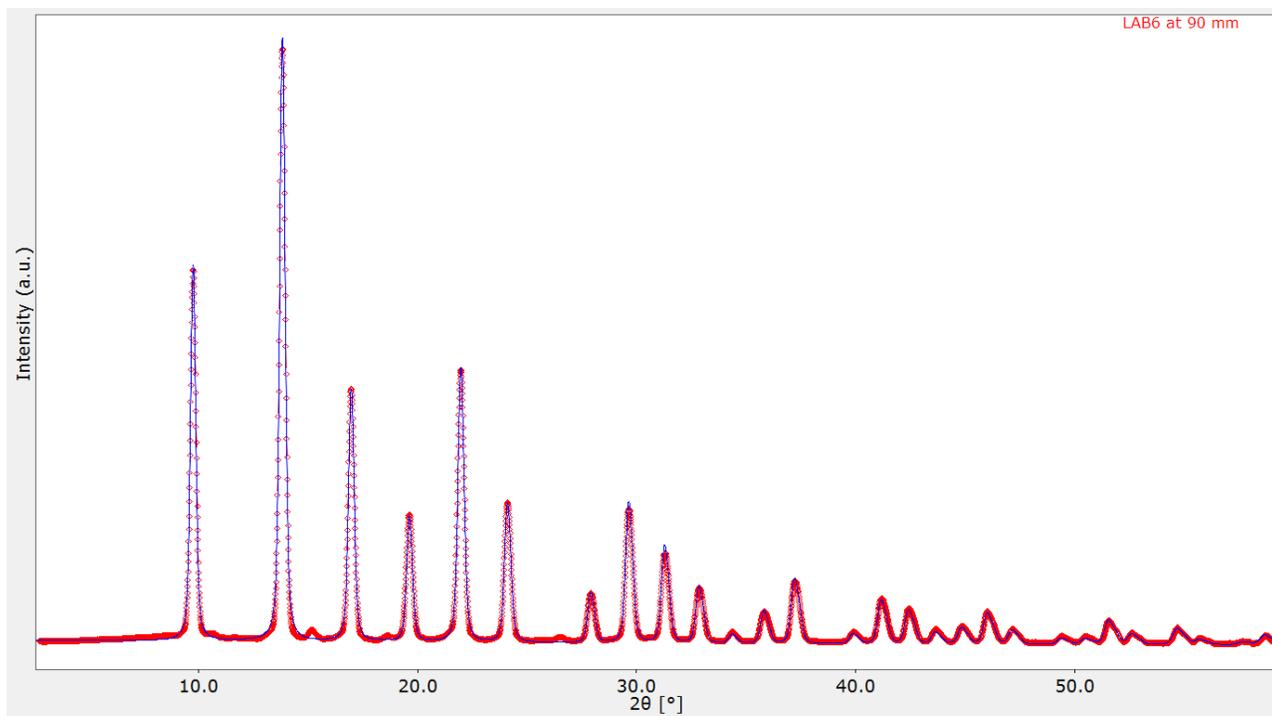


Figure 2 The 1D-diffraction pattern (with the simulated pattern) from the LaB6 powder converted from the 2D diffraction pattern using CrysAlisPro.

Sample preparation

0.025" polyimide capillaries are optimal for sample handling and mounting. The capillary is filled by pressing the capillary onto the sample on a flat surface. As the intensity of the diffraction is dependent on the density of the material, we pack the sample as tightly as possible by pressing the sample between two 0.022 pin gauges. The compacted sample typically forms a plug, so for most samples no further plug is necessary.



The capillary slides into Mitgen B3 goniometer bases or other goniometer pins. The sample should be positioned at the same height as the crystal in a single crystal measurement and the top of the capillary trimmed so that it is not more than 1-2 cm above the sample, to prevent collision with the coldstream nozzle and the beamstop. The capillary is secured with small amount of wax.



Diffractometer calibration

In order to get good, sharp peaks the beam center and the detector distance should be determined. Anticipating that the baseline calibration of the instruments for single crystal analyses (with the YLID test crystal) may not be good enough for powder diffraction, we calibrate by using a good quality crystal with longer unit cell axes, collecting data at the same distance as the powder data. Refinement of the instrument parameters with a complete data set gives a better instrument model for powder diffraction.

Contact Dr. John Bacsa to discuss how the [Emory X-ray Crystallography Center](http://xray.chemistry.emory.edu) can support your research!

- Single crystal X-ray analyses include a publishable quality CIF and a complete structural report. You will receive a complete report of your structure as well as any relevant information that is needed for publication. We routinely determine the absolute configuration of light-atom chiral compounds.
- Our powder X-ray applications include phase identification, unit cell refinements, and particle size analysis.

Service	Emory users	Other academic users
Data collection, data reduction, and standard structure determination	250	300
Unit cell determination, Quick Connectivity Check	75	100
Powder diffraction analysis	75	100