X-RAY POWDER DIFFRACTION FOR AB INITIO STRUCTURE DETERMINATION USING 2D DETECTORS. [I] A SIMPLE BUT EFFECTIVE DETERMINATION OF THE 2D IMAGE CENTRE

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For the Powder diffractionist, one obvious reason to make use of a 2D detector often found, but not exclusively, at synchrotron macromolecular beamlines is to provide direct visualization and measurement of the anisotropy of the X-ray scattering within each one of the Debye-Scherrer rings. Our own motivation lies elsewhere: to make use of the much smaller exposure time [< 2 mn] to prevent the decomposition of the pharmaceutical or biological samples that might occur at a dedicated High Resolution powder diffraction beamline where a typical 1D histogram at least requires a 10-fold increase in the exposure time.

The transformation of a 2D digitized frame as obtained using a 2D detector into a usable 1D powder diffraction histogram requires a very accurate determination of its centre, i.e. of the point of impact of the direct X-ray beam onto the detector. Whilst this determination could in principle be achieved experimentally by the beamline scientist, this information may not always be available nor suitably updated and most especially so at a macromolecular beamline where this requirement is less stringent.

This situation has prompted us to look for and eventually develop a strategy that would yield a good enough estimate of the centre position (in pixel coordinate units) using only those obtained frames pertaining to the samples of biological or pharmaceutical interest. Our suggested procedure involves a three-step determination, the first one of which is mandatory and the next two allow for fine-tuning. Our procedure has been demonstrated by using two macromolecular beamlines [HZB/MX–BL14-1][1-2] and [SOLEIL / PROXIMA2][3-4], respectively endowed with a DECTRIS / PILATUS 6M and a DECTRIS / EIGER 9M detector, and producing 2D frames in CBF and HDF5 formats. These frames were obtained on a newly characterized triclinic pharmaceutical compound ISOXICAM. In both instances, the only requirement is the use of the freely available DECTRIS / ALBULA[5] software.

How the ensuing 1D histograms are further independently processed to first provide indexing of the triclinic unit cell and then ab initio structure solution is addressed in our second poster [II]: "Indexing and Ab Initio Structure Determination of Isoxicam from XRPD at Macromolecular Beamlines".

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