Movement of Single-Crystal Protein Samples at Synchrotron Beamlines

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Aside from normal rotational motion during collection of diffraction data from protein single crystals at synchrotron beamlines, additional movement of the nylon cryoloops holding these samples has been observed, due likely to the action of the nitrogen gas stream used to cool the samples cryogenically. A series of experiments was performed on the bending-magnet beamline (19BM) of the Structural Biology Center at Argonne's Advanced Photon Source in an attempt to characterize cryoloop movement. A baseline for the goniometer and timing shutter was established by measuring the profile of the 220 reflection from single-crystal silicon rod during data acquisition. Single-crystal silicon cubes, approximately 200 μm on a side, were mounted in 20-μm, single-fiber Nylon 66 cryoloops, and 220 reflection profiles were recorded. When compared with the silicon rod measurements, movement of the cryoloops was clearly evident. When shutter timing and synchronization among the goniometer, timing shutter and area detector are known and correct, diffraction data from low-mosaicity (less than 0.25° FWHM) lysozyme single crystals are a sensitive probe of cryoloop movement, as manifested in processing statistics resulting from both integration and scaling of the data.

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