

Experiments Abating Radiation Damage with Cryogenic Helium

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Helium is a more attractive cryogen than nitrogen for macromolecular data collection at high flux beamlines because of lower deliverable temperatures and better thermodynamic properties. The most salient question about helium use is whether these physical properties can provide improved data quality during the extended data collection periods needed for MAD, MIR or SIRAS phasing, or for ultra-high resolution data collection. Conflicting results have been reported for the usefulness of helium as a cryogen during macromolecular data collection. These observations may be rationalized by the differences in various helium cryostats. We have previously reported on our experiences with an open-flow helium cryostat in macromolecular data collection [1,2]. This device, described as either a Pinkerton-type device [3] or the HeliToledo [2], has been shown to deliver a sufficient cooling to permit collection of a number of large data sets from small molecule crystals at ~16K [3]. The HeliToledo differs from other commercial devices by providing a high flow of cryogen through a narrow aperture resulting in a modest use of helium [4]. In our helium cryogen macromolecular data collection experiments, we noted both an increase in signal level and resolution with crystals of myoglobin, nucleosome core particle and immune proteins. Additional experiments with immune proteins indicated an increase in crystal diffractive lifetime. Recently, we conducted a series of experiments at the IMCA-CAT 17-ID beamline to further examine whether helium provided radiation damage abatement compared with nitrogen. These studies used matched crystals of xylose isomerase (glucose isomerase, kindly provided by Genencor). These crystals diffracted to ~1 Å. Multiple data sets were collected under both helium and nitrogen cryogens. Nitrogen data were collected at 100K using an Oxford Instruments Cryostream. Comparisons were made of the data reduction statistics between the different data sets, and possible temperature related changes in the molecular structures. The results of these experiments will be presented.

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[2] Hanson, B. L., Harp, J. M., Kirschbaum, K., Parrish, D. A., Timm, D. E., Howard, A., Pinkerton, A. A. & Bunick, G. J. (2001) *Journal of Crystal Growth* 232:536-544.

[3] Ribaud, L., Wu, G., Zhang, Y. and Coppens, P. (2001) *Journal of Applied Crystallography* 34: 76-79

[4] Hardie, M. J., Kirschbaum, K., Martin, A. and Pinkerton, A. A. (1998) *Journal of Applied Crystallography* 31: 815-817.