Multi-temperature Neutron & X-ray Studies of Hydrogen Bonded Molecular Complexes

Martin Adam[1], Ian D. H. Oswald[2], Andrew Parkin[1], Simon Parsons[3], Chick C. Wilson[1], [1]*Department of Chemistry, University of Glasgow.* [2]*ESRF, Grenoble.* [3]*School of Chemistry, University of Edinburgh.* E-mail: maadam@chem.gla.ac.uk

X-ray single crystal diffraction is a commonly used technique in many structural chemistry laboratories to find the structure of large and small molecules. However, neutron diffraction, although less readily accessible, is more sensitive to the determination of detailed hydrogen atom parameters, and this is of particular importance in hydrogen-bonded systems. By application of a multi-temperature approach to both these techniques, it is possible to study in detail the temperature-dependent behaviour of the hydrogen atoms within these hydrogen bonds. Features such as proton disorder and migration can be frequently observed.

Both X-ray and neutron multi-temperature single-crystal data has been collected on the molecular complex of isonicotinamidium formate. Neutron data were collected at four temperatures (40K, 100K, 150K, 200K) on the SXD instrument at ISIS, and X-ray data were collected on a laboratory diffractometer at 50K intervals from 100K-300K.

We will present the initial analysis of the neutron data, including some discussion of the inherent difficulties in processing such datasets and some of the early use in the chemical crystallography area of the new SXD2001 software developed for the instrument, which we are helping to test. We will also present how these initial neutron results compare with the X-ray data.

Keywords: neutron crystallography, hydrogen bonds, variable temperature