

## **NEUTRON POWDER DIFFRACTION WITHOUT RESORT TO DEUTERATION: USE IN MOLECULAR STRUCTURE REFINEMENT**

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Advances in structure determination from powder diffraction methodologies have meant that increasingly complex structures are being solved from X-ray powder diffraction data alone. Often, the molecular packing and conformation obtained from a structure solution and refinement is sufficient for the purposes required. However, due to the limited resolution of the powder data and the problem of reflection overlap, there are frequently ambiguities in the final structure that have to be resolved by applying prior chemical knowledge or must be 'flagged up' as being unresolved. By way of example, the rotation of an SO<sub>2</sub>NH<sub>2</sub> group, critical for determining H-bonding within a crystal, may be left unresolved due to the negligible difference in X-ray scattering power between oxygen and nitrogen. Similarly, the orientation of the H-atoms around the N can normally only be implied from potential H-bonding considerations.

Neutron powder diffraction (NPD), with its ability to distinguish light elements (particularly hydrogen) by virtue of their different scattering lengths is potentially a very useful complementary technique for resolving such ambiguities. Unfortunately, its application to organic materials is severely hindered by the incoherent scattering exhibited by hydrogen; put simply, this incoherent scattering generates a background signal that makes it difficult to detect coherent Bragg scattering from the sample. Deuteration is an effective but normally impractical remedy to this problem and so the routine application of NPD to fully hydrogenous materials remains an important goal. Recent experiments on the GEM diffractometer at ISIS have shown that it is possible to collect good quality NPD data from hydrogenous materials such as chlorothiazide (C<sub>7</sub>H<sub>6</sub>ClN<sub>3</sub>O<sub>4</sub>S<sub>2</sub>), hydrochlorothiazide (C<sub>7</sub>H<sub>8</sub>ClN<sub>3</sub>O<sub>4</sub>S<sub>2</sub>) and carbamazepine (C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>O) and to use this data in combination with XRPD data to allow unrestrained refinement of molecular structures. The high count rates achievable on GEM are critical in overcoming the incoherent scattering problem and data suitable for refinement purposes can be collected in typically 8-12 hours. This presentation will show some successful applications of NPD and discuss the pros and cons of the approach.