MS10 H-bonding & weak interactions in crystals: neutrons and X-rays

Chairs: Boris Zakharov, Amber Thompson

MS10-P1 Crystal structure and hydrogen bonds pattern of a new hydrated hexachloridoindate based on piperazinium

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A new hybrid compound based on piperazinium and hexachloridoindate(III) was synthesized by aqueous solution reaction and characterized by X-ray diffraction.

The asymmetric unit of (C4H12N2)3-[InCl6]2_4H2O, consists of one and half independent piperazinium cations, and hexachloridoindate anion and two molecules of water.

The In(III) ion is six-coordinated and forms a quasi-regular octahedral arrangement. In the crystal, alternating layers of cations and anions are arranged parallel to (101) and are linked with the water molecules via intra- and intermolecular N—H…O, O—H…Cl, C—H…O and N—H…Cl hydrogen bonds, forming a complex three-dimensional network. Additional stabilization within the layers is provided by weak intermolecular C—H…Cl interactions.

Keywords: crystal structure , piperazinium cation, hexachloridoindate anion, hydrogen bond.

MS10-P2 An *ab-initio* fully deuterated tiny crystal (1x0.25x0.20mm) allows Neutron data collection at room temperature up to 1.90Å

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The crystal structure of Heart Fatty Acid Binding Protein (H-FABP) (M.W. = 15 kDa) complexed with its natural oleic acid ligand was jointly refined with X-ray and Neutron diffraction data measured both at RT up to 0.98 and 1.90Å respectively. The H---H contacts (as D---D) occurring in the binding of the long fatty acid (16 C) to the highly conserved hydrophobic protein residues, together with the ordered water cluster (as D_2O) present inside the large internal protein cavity could be analyzed in fine details, [*E. I.Howard et al., (2016). IUCrJ. 3, 115-116., PDB entry 5ce4*]. The perdeuterated protein has been over-expressed in the Deuteration Laboratory at ILL, Grenoble-France using d_8-glycerol as a carbon source [*J. B. Artero et al., (2005). Acta Cryst. D61, 1541-1549.*]. Subsequent crystallization was achieved in D_2O.These perdeuterated crystals must be sheltered against air moisture (to avoid replacement of deuterium by hydrogen atoms) especially during their mounting in quartz capillaries, by using protecting D_2O amounts on each side of the crystal within the capillary, which is finally closed tightly with a two-component glue. The quasi-Laue neutron data were collected at RT at ILL, Grenoble-France on the new LADI-III beamline [*M. P. Blakeley et al., (2010). Acta Cryst. D66, 1198-1205.*]. Interestingly, the ratio of our tiny crystal volume ($0.05mm^3$) *vs.* the crystal asymmetric unit-cell volume ($34.000\ Å^3$), is the smallest ratio ($14x10^{14}$) of any Neutron Protein Crystallography (NPC) study published so far [*M. P. Blakeley et al., (2015). IUCrJ, 2, 464-474.*]. X-ray diffraction data from another perdeuterated crystal of the same crystallization batch were collected also at RT at the Swiss Light Source (SLS) Synchrotron on the X06SA beamline. Both data processing statistics are given in the poster.

Keywords: Neutron protein crystallography, hFABP, perdeuteration