

Three refinement approaches to a description of a (3+1)D incommensurately modulated organic zwitterion dihydrate

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Three structural refinements of the incommensurately modulated structure of (2*S*,3*S*)-2-amino-3-hydroxy-3-methyl-4-phenoxybutanoic acid (C₁₁H₁₅NO₄·2H₂O or **I**·2H₂O) are presented: average approximation, commensurate supercell approximations, and the (3+1) superspace refinement. One of the water molecules of crystallization is disordered over two positions. The three approaches produce results of comparable precision and similar disorder ratio of the water molecule.

Whereas the satellite reflections are well observed, Fig. 1, a single-crystal approximation that disregards them yields a high-quality structure in the space group *P*2₁2₁2₁ with one water molecule refined as disordered over two positions in a 0.625:0.375(16) ratio. The absolute structure and absolute configuration are reliably established despite a low Friedel of 30.64.

The refinement in the commensurate three-fold supercell approximation in space group *P*112₁ is also of high quality with the six corresponding water molecules exhibiting three different occupancy ratios averaging 0.635:0.365.

The structure of **I**·2H₂O is correctly described in the (3+1)-dimensional superspace group *P*2₁2₁2₁(0β0)000 (β=0.357). The loss of the three-dimensional periodicity is ascribed to the occupational modulation of the disordered solvent water molecule, with its two positions related by a small translation (ca. 0.666(9) Å) and ~168(5)° rotation about one of its O–H bonds, with an average 0.624(3):0.376(3) occupancy ratio. There is an extensive hydrogen-bonding network formed by neutral and charge-assisted N–H...O and O–H...O interactions, and the water molecule's occupational modulation arises due to the competition between the different hydrogen-bonding motifs associated with each position.

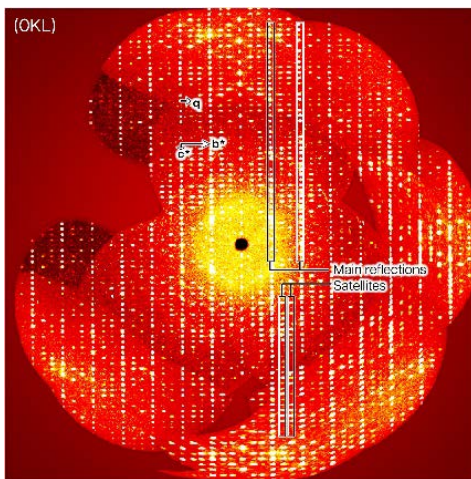


Figure 1. Annotated reconstruction of the *Ok**l* reciprocal space layer for the incommensurately modulated **I**·2H₂O.