MS15 Mineralogical and inorganic crystallography

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Accurate Crystal Structure of Ice VI from X-Ray Diffraction with HAR
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Abstract
Water is an essential chemical compound for living organisms, and twenty of its different crystal solid forms (ices) are known. Still, there are many fundamental problems with these structures such as establishing the correct positions and thermal motions of hydrogen atoms. The list of ice structures is not yet complete as DFT calculations have suggested existence for additional as of yet unknown phases. In many ice structures, neither neutron diffraction nor DFT calculations nor X-ray diffraction methods can easily solve the problem of hydrogen atom disorder or accurately determine their atomic displacement parameters. Here we present accurate crystal structures of H2O, D2O and mixed (50%H2O/50%D2O) ice VI obtained by Hirshfeld Atom Refinement (HAR) against high pressure single crystal synchrotron and laboratory X-ray diffraction data. It was possible to obtain O-H bond lengths and anisotropic atomic displacement parameters for disordered hydrogen atoms which are in excellent agreement with the corresponding results of single crystal neutron diffraction data. Our results show that Hirshfeld atom refinement against X-ray diffraction data is a tool which can compete with neutron diffraction in detailed studies of polymorphic forms of ice and crystals of other hydrogen rich compounds. As neutron diffraction is relatively expensive, requires larger crystals which might be difficult to obtain, and access to neutron facilities is restricted, cheaper and more accessible X-ray measurements combined with HAR can facilitate the verification of the existing ice polymorphs and the quest for the new ones.

References
[1] M. L. Chodkiewicz, R. Gajda, B. Lavina, S. Tkachev, V. B. Prakapenka, P. Dera, K. Woźniak, Accurate crystal structure of ice VI from X-ray diffraction with Hirshfeld Atom Refinement, IUCRJ, 2022, Submitted
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Examples of water clusters considered.