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A-type Amylose single crystals: unit cell refinement from synchrotron radiation microdiffraction data

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The difficulty of obtaining sufficiently large polymer single crystals limits usually X-ray structural studies to fibre diffraction. Progress in synchrotron radiation microdiffraction now allows the collection of X-ray data sets on micrometer-sized crystals. In the present note we report the first study of a polysaccharide single crystal by low temperature synchrotron radiation microdiffraction. Amylose is a linear molecule of (1-4)-linked α-D-glucopyranosyl units, which is found in native starch granules. Needle-shaped A-amylose single crystals of a few μm length and about 1 μm thickness have been crystallized from diluted aqueous solutions of amylose fractions [1]. Diffraction experiments were performed at the ESRF-ID13 beamline at a wavelength of λ=0.0947 nm. The beam was focused by parabolic Be-refractive lenses and collimated to 10 μm at the ID13 microgoniometer [2]. Diffraction patterns were recorded at 100 K by a MAR165 CCD. The diffraction patterns were processed using the XDS program package [3]. The two crystals studied were rotated in steps of 2^0 and 4^0 with 1 and 2 sec exposure time per step correspondingly. Radiation damage restricted the total number of reflections ($I/(\sigma \geq 3)$ 332 and 353 correspondingly. The highest resolution obtained was 1.51 Å. In a previous structural analysis [4], a clear-cut difference between orthorhombic and monoclinic symmetry could not be firmly established for A-amylose. The present X-ray microdiffraction data allows analyzing a larger set of symmetry equivalent reflections. The space group was tested using the XDS program package by calculating the merging factor R_{int} for the statistically significant reflection intensities. In the monoclinic setting a value of R_{int} = 7.6% (expected R-value from intensity statistics: R_{exp}=7.0%). In an orthorhombic setting a value of R_{int} = 19.8% (R_{exp}=21.9%). A systematic change of unit cell parameters was observed with increasing X-ray exposure and hence increasing radiation damage at 100 K. We also noted that the monoclinic unit cell got closer to a pseudo-orthorhombic cell with increasing exposure. This phenomenon is described using the parameter $p=b+a\cos\gamma$, which becomes 0 for a pseudo-orthorhombic lattice.

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Non-covalent interactions in 2-phenylethylammonium perhalometallates

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The aim of crystal engineering is the design of crystal structures, and as a result, materials with desired properties. A fundamental requirement of crystal engineering is the understanding of the role of non-covalent interactions present in the solid-state structure.
This study focuses on the identification of non-covalent interactions in a family of 2-phenylethylammonium perhalometallate compounds, containing the metals Zn, Cd and Hg. A number of novel crystal structures, some isostructural, will be reported. In the compounds investigated, each ammonium group possesses three hydrogen atoms, all participating in hydrogen bonding. The hydrogen bonding- and aromatic interactions present in these crystal structures will be highlighted, and their influence on the molecular geometry and packing illustrated. Crystal engineering synthons will be identified, and compared to synthons identified in related structures.