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The Crystal Structure of Aspartame Anhydrate from Powder Diffraction Data

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Aspartame (L-aspartyl-L-phenylalanine methyl ester) is a dipeptide sweetener, ~200 times sweeter than sucrose. Because of its dietary and pharmaceutical usefulness, various characteristics of aspartame, including the model of its receptor site and its conformation in aqueous solution, have been studied extensively [1, 2]. Aspartame is known to grow in different pseudo-polymorphic forms, IA, IB, IIA and IIB, each containing a different amount of water and all having a needle-like morphology. Although a lot of information on aspartame can be found in literature, no clear and complete picture can be obtained of the dehydration process at the molecular level. One object of our work was to obtain the ″missing″ crystal structure of the anhydrate form and the study of its relation to the other forms. The employed technique was X-ray Powder Diffraction (XRPD), using a Bruker AXS D8 ADVANCE X-ray Powder Diffractometer in transmission capillary geometry. The crystal structure of the aspartame anhydrate was solved using the DASH software [3]. The final Rietveld refinement was performed with the Topas software [4]. The anhydrate crystallizes in the monoclinic system with space group $P2_1$ and cell parameters: a=19.4103(11) Å, b=4.9608(3) Å, c=15.6565(9) Å, β=94.875(2)°, V=1502.14(15) Å^3 (Fig. 1). Comparison of the structures of the hydrates and the anhydrate reveals remarkable similarity between the structures of IA and IB on the one hand and between IIA and IIB on the other hand.

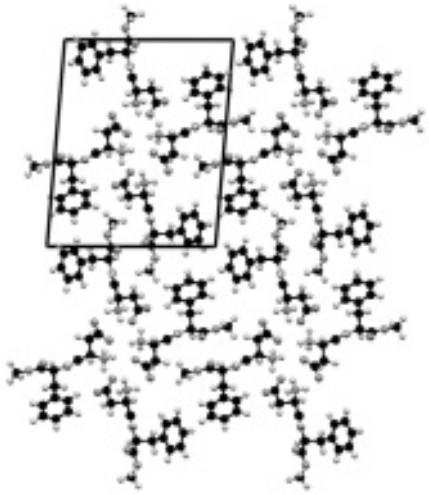

Figure 1. Crystal structure of aspartame anhydrate; b-projection of four unit cells

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Protein powder diffraction at cryocooled conditions

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Modern developments of the powder diffraction technique have allowed the investigations of systems with large unit cells like proteins [1-3]. In previous investigations into protein powder diffraction, the resolution and quality of the data has been limited mainly by rapid deterioration of the protein crystal structure during exposure to the intensed synchrotron X-ray beam. In a typical single crystal diffraction experiment radiation damage can be minimized by collecting diffraction data at low temperatures (typically 100K) which requires the addition of a cryoprotecting agent to the protein sample in order to avoid freezing of the mother liquor. The present study aims in the optimisation of protein cryocooling methods and the enhancement of powder diffraction data when applied to radiation sensitive materials such as proteins. Remarkable variation of the lattice parameters and peak widths with the type and concentration of cryoprotecting agent has already been observed and will be presented. Preliminary data interepretation correlating these changes with the structural and microstructural characteristics of the systems under study will be shown.

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