Trisaccharide Crystal Structures from X-ray Powder Diffraction and Solution NMR

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Carbohydrates belong to a large class of molecules that are ubiquitous in nature. Crystallographic information on oligosaccharides is limited due to their inherent difficulty to crystallize and especially to grow as single crystals. The direct-space methods are nowadays commonly used to solve crystal structures from X-ray powder data. Prior to calculations, the elaboration of the molecular model is an issue since carbohydrates are especially difficult to model because of their flexibility, polarity, and specific stereoelectronic effects (i.e. anomeric, exo-anomeric and/or gauche effects). Complementary investigations on carbohydrate conformations could be performed in solution by NMR spectroscopy. NMR techniques constitute a valuable tool for pyranose rings flexibility and glycosidic linkages study, and for the distinction of the multiple conformations that will coexist in solution. In the course of our studies on synthetic pentasaccharides active as antithrombin heparin inhibitors, we have determined the structures and conformations of several oligosaccharide precursors that are isolated as crystallized material without single crystals available. In this contribution, we report the crystal structures of two chemically protected trisaccharides, elucidated using X-ray powder diffraction data. These two trisaccharides being highly flexible, we imagined a method consisting of relieving the computational procedure by introducing geometrical features as deduced from 2D NMR solution studies.

Keywords: structure elucidation, powder diffraction, NMR