

water structure.

References

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Keywords: water, nanopipe, nanowire

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### H-bonds in andrographolide and related molecules: Neutron diffraction, database and modeling studies

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The biologically active natural product andrographolide has OH and CH<sub>2</sub>OH groups at adjacent positions on a decalin ring. Our neutron diffraction study confirms that the OH group is the donor and O(H)CH<sub>2</sub> the acceptor in an intramolecular hydrogen bond (IHB) with H ··· O 1.743(6) Å and O-H ··· O 153.9(5)°, the latter group hydrogen bonding to the OH group of a nearby molecule. A priori this scheme seems preferable since with two rotatable bonds CH<sub>2</sub>OH should adapt better than OH to the vagaries of crystal packing. However, an X-ray study of a derivative acetylated at a remote OH group showed the reverse pattern. A CSD search with normalized H atoms for cyclohexane rings with adjacent OH and CH<sub>2</sub>OH groups gave 34 matching fragments: 7 with OH ··· O(H)CH<sub>2</sub> ≤ 2.3 Å, 13 with CH<sub>2</sub>OH ··· OH ≤ 2.3 Å, the remainder with weaker or no IHB. The former class has 'better' IHB with <H ··· O> 1.82 Å and <O-H ··· O> 142° vs. 1.90 Å and 137°, yet is less numerous. Molecular dynamics suggests a swap to the former when cyclohexane changes to decalin. Cluster analysis with dSNAP classifies mainly IHB type but also substituent location. Work supported by the U.S. Department of Energy, Office of Basic Energy Sciences, under contract DE-AC02-06CH11357, and the Malaysian Ministry of Science, Technology and Innovation (MOSTI) grant no. 02-01-04-SF0089.

Keywords: neutron diffraction, hydrogen bonding, natural products

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### Crystal structure of trehalose dihydrate by X-ray and neutron diffraction experiments

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Trehalose is a non-reducing disaccharide that is a frequently synthesized in living organisms as an intracellular protectant or co-protectant against harsh conditions, permitting organisms to survive dehydration or freezing. As this characteristic property is still open to argument, a large number of studies on biopreservation have been performed. However, there are few reports on the crystallographic investigation for trehalose. We have, therefore, tried to determine

both the crystal structure and the electron density distribution (EDD) of trehalose dihydrate using the single-crystal X-ray and neutron diffraction data for understanding of the structural characteristics. The crystal structure of trehalose dihydrate was refined with the data at room temperature. Furthermore, in order to reduce an influence of atomic displacement parameters on the structure refinement, X-ray diffraction intensity has been measured at low temperature. Both the refinement and MEM analysis were performed for obtaining the EDD again. From the results, the covalent bonding was confirmed to exist between the trehalose and the internal water. The EDD, thus obtained, was compared with that calculated using the full-potential linealized augmented plane wave (FLAPW) method.

Keywords: absolute structure determination, electron density distribution, trehalose dihydrate

## P06.01.02

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### Temperature-dependent structural change of trehalose dihydrate and anhydrate crystals

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Thermal analysis method is widely used in order to determine the phase transition temperature for the easier technique, but it is difficult to obtain a detailed information of the temperature. On the other hand, *in-situ* high-temperature powder X-ray diffraction technique is a powerful tool to investigate a detailed structural change. We have, therefore, determined both the crystal structures and the phase transition temperature of trehalose dihydrate and anhydrate crystals using the high-temperature *in situ* powder X-ray diffraction data and the detailed temperature-dependent structural change was investigated. Trehalose anhydrate was obtained by dehydrating from trehalose dihydrate. The melting and crystal-amorphous phase transition peaks of trehalose anhydrate by DSC and TG-DTA were confirmed that it is around 453K. The crystal structure of trehalose anhydrate was refined with the use of powder X-ray diffraction data between 373K and 467K. From this result, trehalose anhydrate have crystal-amorphous phase transition at 461K. For trehalose dihydrate, the same experiment was performed and crystal-amorphous phase transition temperature was confirmed to be around 353K. The structural parameters, i.e. changes of atomic distance in trehalose molecule, were obtained at this temperature. The result, thus obtained, was compared with that calculated using molecular dynamics method.

Keywords: Rietveld structural refinement, *in-situ* powder diffraction, phase transitions

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### HTXRD and XRD-DSC study of crystalline-noncrystalline phase transition of sucrose crystals

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