MS44 P01

Kinetics of Crystalline-noncrystalline Phase Transition in Sucrose Taro Fujita and Ken-ichi Ohshima , *Institute of Materilas Science, University of Tsukuba, Tsukuba305-8573, Japan.* E-mail: ohshima@bk.tsukuba.ac.jp

Keywords: sucrose, in-situ X-ray diffraction, melting

Sugar, in which sucrose ($C_{12}H_{22}O_{11}$) is the main ingredients, attracts a great deal of attention in the field of food, preservative and battery material adjusting to surroundings. However, melting points among sucrose crystal with high purity are occasionally different from each other and are given as in the range from 433 to 459 K. It has been thought that the impurity, the water content or the manufacturing method would contribute to such different melting points. However, there are few X-ray diffraction studies for understanding of the microscopic melting phenomena, though the macroscopic observation ha been carried out by an optical microscope and a calorimeter. We have, therefore, performed in-situ X-ray diffraction study for investigating the kinetics of crystalline-noncrystalline phase transition in sucrose.
Crystalline sucrose was obtained from Syowa Chemical Co. at 99.9 % reagent grade quality. X-ray diffraction profiles were collected with a conventional two-axis diffractometer (Philips, X'Pert Pro) where a compact solid-state-array detector (Philips, X'Celerator) was used. High temperature measurements were carried out using a high temperature attachments (Anton Paar, HTK 16).
Time dependency of X-ray diffraction patterns was obtained using the Ni-filtered CuKa radiation from a line focus X-ray generator (Philips, PW3040) at fixed temperatures (425-443 K). Three characteistic times are introduced as follows. T_h, T_d and T_b are defined as times until the (111) Bragg intensity becomes half value, the d_{111}-spacing increases and background intensity increases, respectively. Each of the three times gives a good agreement and has the minimal value at 433 K (~200min.). The Landau-type thermodynamical treatment is introduced for understanding of a so-called "C" shape in the crystalline-noncrystalline phase transition in sucrose.

MS44 P02

Structural basis for photobleaching of a cyan fluorescent protein Antoine Royant[a,b], [a]*European Synchrotron Radiation Facility, &* [b]*Laboratoire de Cristallographie et Cristallogenèse des Protéines, Institut de Biologie Structurale CEA-CNRS-UJF, Grenoble, France.* E-mail: royant@esrf.fr

Keywords: fluorescence spectroscopy, X-ray protein crystallography, crystal spectrophotometry

The cloning of the Green Fluorescent Protein (GFP) gene from jellyfish *Aequorea victoria* has revolutionized many fields of biology by allowing a genetically-encoded labeling of a given protein. Two GFP mutants, ECFP and EYFP (Enhanced Cyan/Yellow Fluorescent Protein) exhibit fluorescence emission maxima that are respectively blue- and red-shifted compared to GFP (476 and 527nm *vs.* 509 nm). A combination of the two proteins is frequently used in FRET (Förster Resonance Energy Transfer) experiments to measure short distance intermolecular interactions. Unfortunately, ECFP present several drawbacks. Its weak level of fluorescence can lead to experimental results with a poor signal-to-noise ratio. Moreover, its fluorescence level decreases with time, due to photoconversion into a non-fluorescent species, which may hinder data analysis [1]. We have initiated the structural study of ECFP photobleaching through a combination of X-ray crystallography and both UV-visible absorbance and fluorescence spectroscopies at the European Synchrotron Radiation Facility. The experiments were performed off-line at the Cryobench laboratory [2], and online at the macromolecular crystallography beamlines. We have observed the photoconversion of the protein into several species, two of which being non-fluorescent. Diffraction data show that the whole structural changes induced by photoexcitation only affect a small number of residues and water molecules in the vicinity of the fluorophore. The observed changes should account for the transient, or permanent, loss of fluorescence.

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MS44 P03

A New Unit Cell Morphologies for Dianin's Compound MW Bredenkamp, LJ Barbour, J Alen, GO Lloyd, T Jacobs and C Esterhuysen, *Department of Chemistry and Polymer Science, University of Stellenbosch, Private Bag X1, MATIELAND 7602,South Africa*
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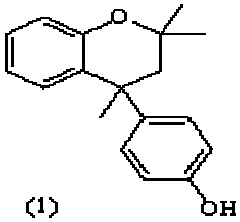
Keywords: crystal engineering, clathrates, hydrogen bonding

Dianin's compound (1) has been known since 1914 and is well known for its clathrating ability. The host to guest ratios are normally 6:1 or 3:1 and with certain bases it sometimes deviates from these clear cut ratios. X-ray crystallography of the clathrates has thus far always revealed an isoskeletal series of host arrangements in the trigonal space-group with a disordered guest in one of two, three or six orientations. We have recently found three new types of crystal structures in which these hosts have different unit cells – triclinic, trigonal and monoclinic – yet two of these new structures (trigonal and triclinic) are closely related to the well known trigonal structure. The triclinic structure has ordered guests and the new trigonal structure is comprised of the cocrystallisation of two supramolecular structures (a novel concept) of which one contains ordered guests and the other disordered guests. All three structures have amine bases as guests/co-crystallising partners in which the host partially or stoichiometrically donates protons to the guest.2 The voided host also proves to be suitable for highly selective absorption of hydrogen.



(1)

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