Nizwa, Sultanate of Oman, PC 616, Oman, [2]2Department of Chemistry and Biotechnology, School of Engineering, University of Tokyo, Bunkyo-ku, Tokyo 113-8656, Japan, [3]3Department of Chemistry and Biotechnology, School of Engineering, University of Tokyo, Bunkyo-ku, Tokyo 113-8656, Japan, E-mail：emmanuel_jp@yahoo.com

The crystal structure of 6-[4-(trans-4-pentylcyclohexyl)phenoxy] hexane-1,2-diol (hereafter, CP2OH) has been determined by X-ray diffraction techniques. The CP2OH molecule crystallizes in the monoclinic crystal system with space group *C*2/*c* (#15). The asymmetric unit consists of one crystallographically independent molecule of CP2OH, and the unit cell contains eight molecules of CP2OH. The CP2OH molecules crystallize in sheets of layered arrangement along [010] direction. The unit distance of 30 Å exists between neighbouring layers along the [010] direction. In each sheet of the layered structure of CP2OH, the molecules are aligned in an inclined manner in which the terminal hydroxyl groups lie in a head-to-head fashion generated by hydrogen bonding interactions. The driving force behind the generation and stabilization of each sheet of the layered structure is attributed to the hydrogen bonded network between the terminal hydroxyl groups in the self assembled driven interactions amongst the CP2OH molecules. The intermolecular interactions are generally driven by hydrogen bonding and are also orientation dependent.

Keywords: hydrogen bonding, self-assembly, layered structure

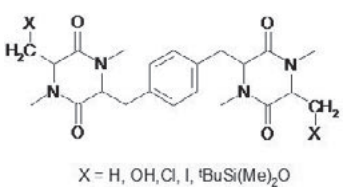
P09.02.14

Acta Cryst. (2008). A**64**, C478

Intermolecular interaction-directed conformations of bridged bis(1,4-piperazine-2,5-diones)

Gary S Nichol, Nathan W Polaske, Lajos Z Szabo, Bogdan Olenyuk
The University of Arizona, Chemistry, 1306 E University Boulevard, Tucson, Arizona, 85721, USA, E-mail：gsnichol@email.arizona.edu

Recent synthetic work to chart a new and efficient route to xylylene-bridged bis(1,4-piperazine-2,5-diones) crossed over into organic crystal engineering, yielding some unexpected and intriguing results. In this class of compounds, molecular flexibility via rotation at the benzyl bridges permits the adoption of several conformers. S and C shapes are possible, depending on the relative position of the piperazinedione rings and the side chain substituent X. An S shape is clearly the least sterically hindered and is observed with both bulky (tert-butyldimethylsilyloxy) and small (hydrogen) substituents. Variation of side chain substituent from hydrogen to hydroxyl and halogen groups, however, showed that several compounds readily crystallize in a potentially energetically unfavourable C shaped conformation. The molecular conformation appears to be directed by hydrogen bonding and halide...halide interactions in the crystal packing, with the stabilization provided by these interactions overcoming steric hindrance. This poster will highlight some of these sterically strained compounds and discuss associated synthetic and crystallographic problems.



X = H, OH,Cl, I, ^{t}BuSi(Me)$_2$O

Keywords: molecular conformation, hydrogen bonding, halide interactions

P09.02.15

Acta Cryst. (2008). A**64**, C478

Polymorphism from a solution perspective: Rationalisation at the molecular level

Vicky M Fawcett[1], Guy A Hembury[1], Ben M Sattelle[1], Mike Sutcliffe[1], Roger J Davey[1], James F McCabe[2], Sven L M Schroeder[1]
[1]The University of Manchester, Chemical Engineering and Analytical Science, D37 Jackson's Mill, PO Box 88 Sackville Street, Manchester, Greater Manchester, M60 1QD, UK, [2]AstraZeneca, Macclesfield, UK, E-mail：v.fawcett@postgrad.manchester.ac.uk

Polymorphism is the formation of different crystal structures by the same chemical compound. It is an important phenomenon associated with crystallization because the crystal polymorphs have different physical properties, especially solubility differences which impact on chemical reactivity and bio-availability. The existence of polymorphic solid forms has far-reaching implications on patent ownership in the pharmaceutical industry. The critical stage in determining the outcome of a crystallization process for a polymorphic system is the nucleation state. This is believed to be a non-crystalline state of a few tens of molecules, formed via self-assembly from solution, present just prior to crystallisation. It is the structure of this state, and how it is influenced, which is key to understanding and controlling polymorph formation. To address this we have developed a protocol that strives to relate experimental and theoretical data, via artificial neural network analysis. This allows both experimental polymorph prediction and the identification of the molecular-level parameters (or combinations of) that influence the polymorph selection process. Our first target system was that of the well-studied polymorphic pharmaceutical carbamazepine. Polymorph screening experiments revealed a strong solvent and temperature dependence on the polymorph obtained from each experiment. Our work has focused on identifying the molecular-level parameters that are influential in the selection process, and consequently developing an ability to determine the polymorphic outcome of a crystallisation experiment.

Keywords: polymorphism, neural networks, molecular modelling

P09.02.16

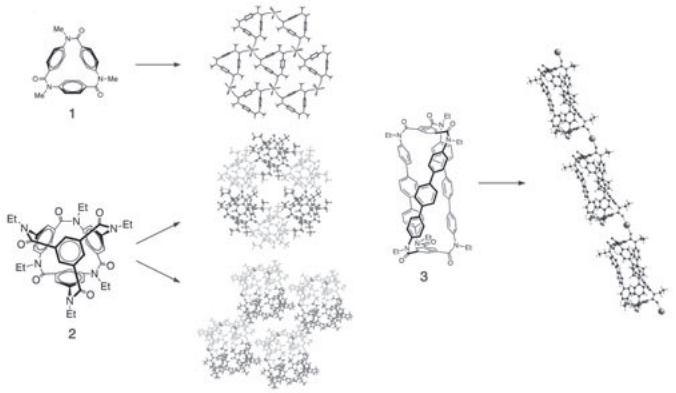
Acta Cryst. (2008). A**64**, C478–479

Characteristic network structure constructed from various block-like molecules

Hyuma Masu, Kosuke Katagiri, Masahide Tominaga, Isao Azumaya
Tokushima Bunri University, Faculty of Pharmaceutical Sciences at Kagawa Campus, 1314-1 Shido, Sanuki, Kagawa, 769-2193, Japan, E-mail：masu@kph.bunri-u.ac.jp

Construction of molecular networks has recently drawn much attention in respect to the useful applications such as catalytic activity, gas adsorption and molecular channels for clathration. It is known that 3-D bulky molecule can construct a highly ordered aggregation in crystalline state. Precise control of topology, size and direction of functional group are required to such block molecules. We synthesized various block molecules based on stereochemistry of tertiary aromatic amide which prefers cis (folded) conformation and studied the crystal structures of them and their metal complexes. The simple cyclic aromatic triamide 1 constructed a 2-D coordination network with lanthanide metal cations in the crystalline state. Spherical aromatic amide 2 aggregated into a channel-shaped network via weak intermolecular interactions. Furthermore, complex

of compound 2 and metal cations constructed more large channel-shaped network. On the other hand, triple helicate 3 was connected by coordination with metal cations to form a 1-D infinite chain. Chirality of each chain was derived from the helicity of the block molecules.



Keywords: helical macromolecules, porous materials, structures of metalloorganic complexes

P09.02.17

Acta Cryst. (2008). A**64**, C479

Self assembled hierarchical nanostructures: Controlling morphology and molecular arrangement

Jangbae Kim[1], Tae Hee Han[2], Ji Sun Park[2], Sang Ouk Kim[2], Hyotcherl Ihee[1]

[1]Center for Time-Resolved Diffraction/KAIST, Chemistry, Dept, of Chemistry, 373-1, Kuseong-Dong, Yuseong-Ku, Taejeon, Taejeon, 305-701, Korea (S), [2]KAIST, Dept. of Materials and Engineering, 373-1, Kuseong-Dong, Yuseong-Ku, Taejeon, 305-701, Korea, E-mail：biomimics@gmail.com

An aromatic dipeptide consisting of the two covalently linked phenylalanine units (FF dipeptide), a key structural motif of Alzheimer's beta-amyloid polypeptide, has been reported to form well-defined nanotubes (NTs) and nanowires (NTs). Despite the enormous interest for their properties and applications, however, the link between the nanoscale morphology (NWs or NTs) and the molecular arrangement has been missing. Here, we demonstrate that both NT and NW morphologies of self-assembled dipeptides interconverted by adjusting the preparing conditions. In addition, we also provide the link between the nanoscale morphology (NWs or NTs) and the molecular arrangement of FF diepeptides. Structural analysis via Rietveld refinement of powder x-ray diffraction patterns show that the molecular arrangements of two different morphology, NWs and NTs, display considerable differences in terms of intermolecular hydrogen bond.

Keywords: peptides, nanotubes, powder X-ray diffraction

P09.02.18

Acta Cryst. (2008). A**64**, C479

X-ray diffraction and small angle X-ray scattering study under high voltage in EHD

Yosuke Michishita[2], Yusuke Imai[2], Hiroshi Abe[2]

[1]National Defence Academy, g46026@nda.ac.jp, Yokosuka, Kanagawa, 239-8686, Japan, [2]National Defense Academy, Yokosuka 239-8686, Japan, E-mail：g46026@nda.ac.jp

Electro hydrodynamics(EHD) phenomenon is well known to be convections of dielectric liquids under the applied voltage. For instance, EHD actuators are designed for cooling system[1]. Nevertheless, few studies associating with molecular aggregations are reported.Here, it is considered that microscopic clusters under high voltage have a relation with macroscopic mechanism of EHD fluids.Dielectric liquids were $C_3F_7OCH_3$, $C_4F_9OCH_3$, $C_4F_9OC_2H_5$, and $C_6F_{13}OCH_3$ *In-situ* observations under high voltage were performed by X-ray diffraction(XRD) and small angle X-ray scattering(SAXS).Data were collected using 18 kW X-ray generator. The wavelength was 0.1542nm. transmission geometry was selected for the measurements.In general, pumping mechanism is divided into ion drag(high current region) and polarization(low current region) pumping. By Fourier transform of XRD, radial distribution function(RDF) is calculated. At 0kV, RDF decreases with increasing molecular weight, M.Also, the intermolecular distance becomes longer at larger M.By *in-situ* observations, the significant point is found. RDF decreased below 5kV(polarization region), though RDF increased above 5kV(ion drag region). Certainly, current density showed the non-linearity at 5kV.In SAXS experiments, scattered intensities were constant below 5kV. In contract, time dependence of the intensities was observed above 5kV. There is a tendency that the size of density fluctuations is inverse proportional to M.It is found that molecular aggregation is caused in ion drag region, While each molecule flows independently in polarization pumping.

[1] J.Darbi and K.Ekula,Microelectro.J.34(2003)1067-1074

Keywords: SAXS, pair distribution function, molecules

P09.02.19

Acta Cryst. (2008). A**64**, C479

Control of stoichiometry and structure: Mechanism of cocrystal formation in mechanochemical synthesis

Shyam Karki, Tomislav Friscic, William Jones

University of Cambridge, Department of Chemistry, Lensfield Road, Cambridge, Cambridgeshire, CB2 1EW, UK, E-mail：sk512@cam.ac.uk

Recent years have seen a rapid progress in the synthesis of supramolecular materials by cocrystallisation. The ability to construct cocrystals following a design based on supramolecular synthons allows controlled and predictable synthesis of new forms of materials with improved physicochemical properties. Several methods of cocrystal synthesis and screening are available including solution crystallisation and solid-state grinding. Our group has been investigating solid-state grinding as an advanced method to control polymorphism and stoichiometric diversity in cocrystals. In this contribution, we will delineate our recent conclusions on the supramolecular mechanism of cocrystal formation by grinding. This will focus on the effects of the competition and hierarchy of supramolecular synthons in the course of mechanochemical synthesis. As model compounds, we have explored cocrystals of nicotinamide with dicarboxylic acids that can exhibit the competition between acid-pyridine, acid-acid, acid-amide and amide-pyridine supramolecular synthons. In addition, we also present a qualitative comparison of liquid-assisted grinding to other mechanochemical and non-mechanochemical synthetic approaches, such as neat grinding, thermal methods and solution growth in terms of screening efficiency and stoichiometric diversity.

Keywords: cocrystals, mechanochemical synthesis, stiochiometric diversity