

Microsymposia

means to manipulate micron sized objects in solution. A laser is focused by a high NA objective lens to produce an optical trap. Trapped crystals can be manipulated within the crystallization drop to both sort and mount them on standard crystal supports used in cryo macromolecular crystallography. The talk will demonstrate several successful examples of this new mounting technique and discuss the limitations like laser beam induced radiation damage.

Keywords: optical trapping, laser tweezers, microcrystals

MS.65.5

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Serial crystallography using protein beams

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By spraying aligned molecules continuously across a synchrotron beam, we hope to develop a new method for solving protein structures which cannot be crystallized (Starodub et al *J. Synch Res* 15, 62 (2008)). We have developed a particle injector which provides a single-file beam of hydrated proteins or cells either continuously or on-demand (for synchronization with a free-electron X-ray laser). We are also developing methods for aligning molecules in a beam, including flow alignment, laser alignment and magnetic field alignment. First results will be presented showing ring diffraction patterns from Photosystem I protein crystallites consisting of just 19 x 19 x 21 unit cells, obtained using the coherent undulator beamline 9.0.1 at the Advanced Light Source at 2nm wavelength (Shapiro et al, *J. Synch Rad.* (2008) submitted). Design details of the aerojet gas-focussing droplet beam generator will be given (DePonte et al (2008), Weierstall et al *Expts in Fluids.* (2008)). Radiation damage aspects of diffractive imaging using femtosecond hard X-ray pulses will also be discussed. A solution to the inversion problem of multiple scattering for soft X-rays will be presented. This group effort (see papers for the many contributors) is supported by NSF, CBST, and ARO awards.

Keywords: femtosecond X-rays, serial crystallography, diffractive imaging

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Multi-component solids in crystal engineering

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Multi-component solids have always been studied as an integral part of structural chemistry. The earliest of these substances is perhaps the quinone-hydroquinone donor-acceptor complexes which were identified more than 150 years ago. In the context of recent developments in crystal engineering, the central issue seems to be whether or not there are any fundamental differences between single-component crystals and multi-component crystals with regard to matters such as crystallisation, design principles, structural specificity, propensity towards polymorphism, crystal structure prediction and property tuning. Kitaigorodskii stated that studies of binary crystals of organic substances are a key for studying intermolecular interactions. We have stated that the very isolation of a two-component solid is evidence of the fact that some intermolecular interactions between the different molecules in the solid are more

significant, stronger or more specific than the interactions that are possible between molecules of the same type. Multi-component crystals can have fixed stoichiometries and well-defined structures, or they can be of the substitutional solid solution type. Both varieties are useful in crystal engineering. These themes will be illustrated with examples taken from our research over the years.

Keywords: mixed crystal, co-crystal, hydrogen bond

MS.66.2

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From a molecular dating agency to successful co-crystal synthesis

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The crystal structure of a compound, the 3-D orientation and organization of molecules in a highly regular manner, ultimately determines most of the fundamental physical properties of that particular material, e.g. thermal stability, hygroscopicity, conductivity, and mechanical strength. Consequently, an ability to control and change the crystalline environment of both known and unknown compounds (without altering the individual molecular properties) would be of enormous significance to both manufacturers and consumers of solid specialty chemicals such as pharmaceuticals, pigments, energetic materials, toxins, foodstuff, and detergents. The deceptively simple act of molecular recognition, which eventually leads to crystallization, is achieved by balancing a range of relatively weak non-covalent, forces, and this presentation will highlight some of our attempts at establishing practical and versatile supramolecular design strategies based on tunable site-specific intermolecular interactions.

Aakeroy, C.B.; Desper, J.; Smith, M.M., *Chem. Commun.* 2007, 3936-3938.

Aakeroy, C.B.; Fasulo, M., Schultheiss, N.; Desper, J.; Moore, C., *J. Am. Chem. Soc.*, 2007, 129, 13772-13773.

Aakeroy, C.B.; Desper, J.; Scott, B.M.T. *Chem. Commun.* 2006, 1445-1447.

Aakeroy, C.B.; Desper, J.; Salmon, D.J., Smith, M.M. *Cryst. Growth & Design.* 2006, 6, 1033-1042

Aakeroy, C.B.; Salmon, D.J. *CrystEngComm*, 2005, 7, 439-448.

Keywords: co-crystal, hydrogen bonding, halogen bonding

MS.66.3

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Multicomponent crystals; Their formation, characterisation and application

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My talk will focus on the role that multicomponent crystals (e.g. solvates, hydrates, cocrystals, salts) play in the design of new organic solids. I will cover mainly applications in the pharmaceutical area, illustrating the various methods of preparing such solids as well as demonstrating some of the resulting benefits. The xanthenes (caffeine, theophylline and theobromine) have been extensively studied within my group and they illustrate many of the important aspects of this area. In addition to describing our experimental

approaches I will also outline the progress we have made with regard to computational methods in multicomponent crystal prediction as well as property prediction/rationalization.

Keywords: multicomponent crystals, cocrystals, crystal structure prediction

MS.66.4

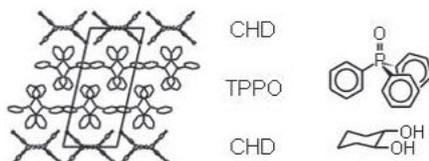
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An unexpected molecular co-crystal with a variable degree of order

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A 1:1 co-crystal of rac-trans-1,2-C₆H₁₀(OH)₂ (hereafter, CHD) and triphenylphosphine oxide (or, TPPO) has been found that is unusual because there are no strong interactions between the two components. Neither CHD nor TPPO has any obvious packing problem that would make formation of an inclusion complex likely. The TPPO layers are very much like those in two of the four known polymorphs of pure TPPO. The H-bonded ribbons of CHD are similar to those found in other vic-diol crystals. The co-crystals are triclinic (*P1*-bar), with the very small deviations from monoclinic symmetry (*C2/c*) arising from incomplete enantiomeric disorder of the R,R and S,S diols. The sizes of the deviations depend on the solvent from which the crystal is grown. Individual CHD layers seem to be at least mostly ordered. Information about R,R/S,S ordering is transmitted from one diol layer to the next through the almost centrosymmetric TPPO layer. The degree of order is unlikely to change with crystal heating or cooling. Thermal data suggest the existence of the co-crystal is a consequence of kinetic factors. Reasons for this surprising failure of fractional crystallization will be discussed.



Keywords: co-crystal, incomplete ordering, crystal growth

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In-situ cocrystallisation combined with Raman spectroscopy

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Fast characterization of the products from cocrystallisation experiments is highly important since there are no general rules for appropriate growth conditions. Therefore a large number of experiments have to be performed to find the right conditions which favor the growth of a cocrystal over the growth of the individual components. For in-situ crystallization on a single crystal diffractometer this is especially important as characterization by diffraction is time consuming. We therefore incorporated a Raman probe into our in-situ laser zone melting apparatus. First results from systems containing acetylene, dioxane, formamide, formic acid and formaldehyde are highly promising. Already in the Raman spectra of the liquid mixtures we found hints to the formation of

molecular aggregates which might be precursors of the cocrystals. After solidification and zone melting growth we used Raman spectroscopy to identify the formation of a cocrystal. Basic research in cocrystal formation is of increasing interest to pharmaceutical science as cocrystals of active pharmaceutical ingredients can have advantageous properties.

Keywords: crystal growth, ocrystallization and complexation of small molecule, raman spectroscopy

MS.67.1

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Magnetic and structural transitions in frustrated magnets

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I will review recent neutron and synchrotron X-ray scattering studies of frustrated spinels and kagomes. The main focus will be to investigate what kinds of magnetic transitions, and lattice and magnetic states can arise when frustrated spins are coupled with lattice in the highly frustrated antiferromagnets.

Keywords: frustrated magnets, neutron scattering, X-ray scattering

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E/T-scaling behavior in the magnetic quasicrystal Zn-Mg-Ho

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Quasicrystals have distinct (quasiperiodic) spatial symmetry characterized by a hidden translational invariance in higher dimensional space, and thus differs both from the periodic crystals and random glasses. Ordering and dynamics of spins in the quasiperiodic structures have been one of fundamental issues. Despite a variety of theoretically expected anomalous properties, earlier macroscopic experiments only exhibited rather normal spin-glass-like behavior. Recently, using the neutron scattering technique, we have revealed non-trivial spin dynamics --temperature independent spin excitation spectrum-- in the magnetic quasicrystal Zn-Mg-Ho, which is, we believe, the most prominent feature ever found. Magnetic excitation spectrum in the Zn-Mg-Ho quasicrystal has only a quasielastic component centered at the elastic position. The quasielastic signal for the positive energy-transfer side is found to be temperature-independent in a wide temperature range $1.5 < T < 200\text{K}$; it may be noteworthy that the excitation spectrum shows little change even below the spin-glass-like freezing temperature $T = 1.95\text{K}$. In the low-energy region $E < 1.5\text{meV}$, imaginary part of generalized susceptibility obtained from the scattering function shows scaling behavior: $\text{Im } \chi(Q, E/k_B T)(k_B T)^{1/3} \propto (E/k_B T)^{-1/3} \tanh(\alpha E/k_B T)$. This is a typical *E/T*-scaling function, where the susceptibility is scaled only by *E/T*, except for the weak prefactor $T^{1/3}$. This *E/T*-scaling is frequently observed in the non-Fermi-liquid (NFL) compounds, such as UCu₄Pd, being in vicinity of