

MS86.30.4*Acta Cryst.* (2005). A61, C110**A Relational Database Approach to Report Generation**

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As the throughput of X-ray diffraction laboratories increases and the groups of users diversify, the need for an automatic report generation system becomes more apparent. Although the type of report varies depending on the user's objectives, the data in the report is always based on information gathered throughout the experiment.

We present a relational database approach to report generation. For each step of the structure determination the relevant pieces of information are recorded, such as the type of operation, input and output data, software version, user name, date and time. Furthermore, by linking the input of an operation to the output of the preceding steps, each piece of information is put into context as and when it becomes available. From the emerging tree structure it is possible to create a report from the beginning of the experiment to any step in the experiment. Wrong paths that did not lead to the final result are preserved too.

Reports come in different shapes and sizes: various formats (e.g. CIF, mmCIF, HTML, and proprietary formats), various purposes (e.g. publication, archiving, or visualization), and varying degrees of detail. In order to serve this wide spectrum of needs we have developed a template language that describes which data is extracted from the database and how it is formatted.

Keywords: report generation, databases, CIF**MS86.30.5***Acta Cryst.* (2005). A61, C110**Structure Integration with Function, Taxonomy and Sequences (SIFTS)**Ganesh Jawahar Swaminathan^a, Sameer Velankar^a, Phil McNeil^a, Antonio Suarez^a, Daniel Barrell^b, Jules Jacobsen^b, Kim Henrick^a, ^aMacromolecular Structure Database. ^bUnProtein Resource, European Bioinformatics Institute, EMBL Outstation, Hinxton, Cambridge CB10 1SD, United Kingdom. E-mail: msd@ebi.ac.uk

One of the major obstacles to the improved integration of structural databases such as MSD (<http://www.ebi.ac.uk/msd>) and sequence databases like UniProt is the absence of up to date and well maintained mapping between corresponding entries. MSD and UniProt have worked closely to clean up the taxonomy and sequence cross-reference, a vital step in integrating sequence family databases such as Pfam and Interpro with structure-oriented databases (SCOP and CATH). This information has been made available to the eFamily group (<http://www.efamily.org.uk/>), and now forms the basis of the regular interchange of information between the member databases (MSD, Uniprot, Pfam, Interpro, SCOP and CATH). This work was carried out under the SIFTS initiative (<http://www.ebi.ac.uk/msd-srv/docs/sifts/>). As well as the domain boundary definitions, the schema also allows any associated sequence or structural alignments to be encapsulated in XML. Since the eFamily schema is complex, an API has been developed for easy integration into BioPerl. For example, the API allows a Pfam alignment wrapped up in the eFamily XML to be returned as a BioPerl alignment object and *vice versa*. The use of such APIs in the development of production quality webservices will also be discussed.

Keywords: data integration, webservices, data exchange**MS87 REACTIONS IN MOLECULAR SOLIDS****Chairpersons:** Leonard Richard MacGillivray, Fumio Toda**MS87.30.1***Acta Cryst.* (2005). A61, C110**Mechanically Induced Reactivity of Molecular Crystals: Chemistry (almost) without Solvents**

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Reactions between solids, such as those activated by mechanical co-grinding of different solid materials, or those between a solid and a gas, are attracting the interest of scientists engaged in the quest for sustainable chemical processes. In Bologna we have been actively involved in the exploration of solvent-free mechanochemical reactions as a means to prepare novel mixed molecular crystals. The basic idea is that molecular diffusion by contact between two molecular crystals can lead to formation of supramolecular bonds and co-crystals or adducts without solvent participation. Under this point of view solvent-free reactions between or within molecular crystals can be regarded as a *green way* to crystal engineering.^[1]

In this contribution we report that co-grinding of silver acetate with *trans*-1,4-diaminocyclohexane [H₂NC₆H₁₀NH₂] in 1:1 ratio generates novel one- and two-dimensional coordination networks based on Ag--[H₂NC₆H₁₀NH₂] coordination bond and on hydrogen bonds between the acetate anions and the bis-amine ligand. The bis-amine [H₂NC₆H₁₀NH₂] has been little exploited in crystal engineering experiments and only few examples are known of its use for the construction of hydrogen bonded adducts or coordination networks.

[1] Braga D., Grepioni F., *Angew. Chem. Int. Ed.*, 2004, **43**, 4002.**Keywords:** crystal engineering, hydrogen bonding, supramolecular mechanochemistry**MS87.30.2***Acta Cryst.* (2005). A61, C110**Solid-Solid Reactions of Xanthenols with Unsaturated Hydrocarbons**Luigi R Nassimbeni^a, Mino R Caira^a, Elizabeth Curtis^a, Hong Su^a and Benjamin Taljaard^b, ^aDepartment of Chemistry, University of Cape Town, Rondebosch, 7701, South Africa. ^bDepartment of Chemistry, University of Port Elizabeth, Port Elizabeth, 6000, South Africa. E-mail: xrayluig@science.uct.ac.za

The structures of the inclusion compounds of the host H = 9-(4-methoxyphenyl)-9H-xanthen-9-ol with a series of aromatic guests: naphthalene, anthracene, phenanthrene, pyrene and α -naphthol have been elucidated. The structures are similar, crystallise in the space group P(-1), and are characterised by pairs of hydrogen-bonded host molecules, with the guests located at centres of inversion.

The kinetics of the solid-solid reactions between the host, H, and naphthalene and α -naphthol were monitored by X-Ray powder diffraction at 25°C and their rate constants established.

Linear relationships were derived for unit cell volumes versus the number of guest atoms in this series as well as from the structures containing benzene, toluene and the isomers of xylene as guests.

Lattice energy calculations for the naphthalene and α -naphthol structures were reconciled with the results of thermal analysis obtained by DSC.

Keywords: solid-solid reaction, clathrate, kinetics**MS87.30.3***Acta Cryst.* (2005). A61, C110-C111**Structural and Thermochemical Studies of Lattice Inclusion Hosts**

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Lattice inclusion hosts are molecules that form cage or channel type frameworks in the solid-state having Å to nanometer-sized voids [1]. Recent crystal engineering strategies for the design of open-framework solids, inclusion of small guest/solvent molecules in the voids, and thermo-chemical behavior of host-guest complexes will be discussed. Significant structural and functional differences in channel and cage type host structures are due to differences in hydrogen bonding / intermolecular interactions, van der Waals close packing, and the strength of host lattice in trapping guest species. Structural control of the cage/channel lattice and modification of pore size/shape through functional group and guest selection is illustrated in robust organic host systems. Certain volatile guests are tightly enclathrated in the host framework as measured by high T_{onset} values in DSC measurements. Selective guest inclusion is monitored by TG-IR and explained through hydrogen bonding in X-ray crystal structures. Some

host scaffolds assembled via O-H...O, N-H...O hydrogen bonds and halogen...halogen, pi-pi stacking interactions based on T-shaped, H-shaped and trigonal tectons have appeared in recent publications from our group [2-4].

[1] Nangia A. in *Nanoporous Materials: Science and Engineering*, Eds. G. Q. Lu and X. S. Zhao, World Scientific, Singapore, 2005, 165-187. [2] Reddy C. M. et al., *CrystEngComm*, 2005, 7, 44-52. [3] Aitipamula S., Nangia A., *Supramol. Chem.*, 2005, 17, 17-25. [4] Saha B. K. et al., *Cryst. Growth Des.*, 2005, 5, ASAP.

Keywords: crystal engineering, guest-host structures, thermal analysis

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Photochemical Reactions in Inclusion Compounds

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In inclusion compounds the guest molecules occupy space formed by the host molecules. Carrying out photochemical reactions in inclusion compounds proved to be a unique method for the synthesis of a large variety of compounds. The research deals with unimolecular and bimolecular photochemical reactions in inclusion compounds. In a recent publication the effect of the reaction core on the homogeneity/heterogeneity of the reaction was studied. A question arises from this recent study: what happens if the volume of the product is smaller than that of the reactant? Free volumes are not anticipated to exist. Therefore it is expected that the following possibilities will occur: either destruction of the crystal as a result of the collapse of the cavity's walls (heterogeneous reaction) or that some other molecules will occupy the free space. We have encountered for the first time few examples where a photochemical dimerization reaction is taking place in a single crystal of inclusion compound and at the end of the reaction water molecules penetrate into the free space without destruction the crystal lattice. Moreover, at the end of the dimerization, the orientation of the dimer with respect to the host molecules is different than that prior to the reaction. Evidently the dimer is rotating during or after the photoreaction.

The aim of the unimolecular study is to examine to what extent the conformation adopted by N,N disubstituted- α -oxoamides determines its photochemical reaction. We have used different host molecules in order to control the conformation of the guest. It was found that indeed the N,N disubstituted- α -oxoamides show different photochemical behavior which depends on their conformation.

Keywords: host-guest complexes, homogeneous reactions, photochemistry

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Confinement of Gases and Polymers in Van Der Waals Crystals

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The exploitation of weak interactions for the fabrication of binary adducts by self-assembly is receiving a great interest. We could obtain high-melting crystalline materials with macromolecules by simple mechanical mixing and grinding of D₃ and D_{3h} molecules with linear polyethylene or several polyconjugated oligomers [1]. A number of weak CH... π and π ... π interactions cooperatively promote the stabilization of the architecture as recognized by solid state 2D MAS NMR. In particular, tris-(*o*-phenylenedioxy)cyclotriphosphazene (TPP) organizes the aromatic paddles parallel to the nanochannels encasing the elongated molecules. The guest hydrogens are subjected to the strong magnetic susceptibility of the surrounding aromatic groups that demonstrate the topology of the guest hydrogen above the π -system. The favorable intermolecular interactions compensate the loss of entropy necessary for constraining the macromolecules to the stretched geometry. However, ²H NMR and ¹³C(T₁) show diffusional fast spinning motion ($\tau_c \approx 10^{-10}$ s) about the polymer axis. The exceptional mobility of the polymer chains in the fully aromatic environment creates unique examples of molecular rotors and

gyroscopes stabilized up to 350°C by soft interactions. The anisotropic alignment of polyconjugated molecules is suitable for growing crystal for electronic applications. The same kind of driving forces determine effective enclathration of light molecules and gases and promote absorption properties from the gas phase [2].

[1] Sozzani P., Comotti A., Bracco S., Simonutti R., *Angew. Chem. Int. Ed.*, 2004, 43, 2811. [2] Sozzani P. et al., *Angew. Chem. Int. Ed.*, 2005, 44, 2.

Keywords: self-assembly, weak interactions, gas storage

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Homo- and Heterochirality in Crystal

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In the present communication the methods for the recognition of homo (Ho)- and heterochiral (He) crystals are considered [1], as well as the conditions for the transformations Ho→He or He→Ho crystallization [2], the directed construction of Ho-crystals [3], solid-phase reactions Ho \leftrightarrow He, synthesis of Ho-crystals [4], isomerizations during the formation of the solid phase [5] and the spontaneous resolution of Ho-crystals [6].

On the example of glycoluriles the methodology for the development of chiral drugs via the spontaneous resolution is presented [7].

The differences in physical properties of enantiomers and racemates are demonstrated by distinct differences in ESR spectra of polycrystalline samples of the enantiomers and racemates of nitroxide radicals [8]. Recently it has been found that the enantiomers of a series of compounds are effective organogelators, whereas their racemates do not show that property [9].

For the first time the supramolecular H-bonded catenanes and polycatenated structures in crystals have been synthesized [10].

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[1] *Mendeleev Commun.*, 2003, 97, 106. [2] *Tetrahedron: Asymmetry*, 2003, 14, 2059. [3] *Mendeleev Commun.*, 2002, 4. [4] *Tetrahedron: Asymmetry*, 2004, 15, 1445. [5] a) *Tetrahedron*, 1982, 38, 549; b) *Tetrahedron*, 1984, 40, 2151. [6] a) *Mendeleev Commun.*, 2000, 16, 83; b) *Mendeleev Commun.*, 2001, 1; c) *Mendeleev Commun.*, 2003, 111; d) *Tetrahedron: Asymmetry*, 2001, 12, 2721. [7] a) *Mendeleev Commun.*, 1998, 231; b) *Mendeleev Commun.*, 2001, 134; c) *Mendeleev Commun.*, 2002, 6. [8] *Mendeleev Commun.*, 2004, 318. [9] *Mendeleev Commun.*, 2005, in press. [10] a) *Tetrahedron: Asymmetry*, 2002, 13, 2697; b) *Mendeleev Commun.*, 2005, in press.

Keywords: chirality, solid phase reactions, recognition of chirality

MS88 ELECTRONIC EXCITATIONS

Chairpersons: John J. Rehr, Keijo Hamalainen

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Electronic Excitations: What Inelastic X-ray Scattering can Reveal

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A review of inelastic x-ray scattering (IXS) as a method to investigate electronic excitations in solids is presented, where also some aspects of synchrotron radiation based instrumentation are touched. The direct access to the polarizability of electrons in solids by measuring the dynamic structure factor using non-resonant inelastic scattering is stressed. The weight of different terms in the diagrammatic expansion of the proper polarizability can be tested. Special lattice effects on electron correlation as zone boundary collective states, plasmon Fano resonances and the plasmon band structure will be represented, where the role of coherent inelastic x-ray scattering is stressed. The increasing importance of resonant inelastic x-ray scattering (RIXS) is emphasized. It is the sensitivity of this