

stoichiometry of the inclusion compounds can be controlled by the composition of the liquid guest mixture. The selectivity of a host molecule towards a given guest in a binary mixture of guests G1 and G2 can be determined by competition experiments, in which the inclusion compounds are crystallised in a mixture of guests of known composition and the ensuing crystals analysed by a suitable analytical technique. The resulting analysis of the enclathrated guests yields the selectivity profile which may be of three main types: (i) Zero selectivity, where the mole fraction of the captured guest = that in the starting solution; (ii) A given guest is strongly favoured over the complete range; (iii) The selectivity is strongly concentration dependent [2]. The question which arises is what is the structure of the harvested crystals? Thus if a host compound H forms inclusion compounds H.G1 and H.G2, what is the structure of the crystals derived from the 50:50 mixture of G1 and G2? Is it a mixture of the original compounds, a new compound, or a combination of both? We have studied this problem using bulky Hosts containing Hydroxyl moieties which capture a variety of guests and have obtained unusual results. Thus, with the host H1 = 1,4-bis(9-hydroxyfluoren-9-yl)benzene dissolved in an equimolar mixture of morpholine (MORPH) and 4-picoline (4PIC) the ensuing crystals contain both H1.2(MORPH) and H1.2(4PIC) in the unit cell, with the host adopting both cis- and trans-configurations. The related host H2 = 2,2'-bis(hydroxydiphenylmethyl)1,1'-binaphthyl, yields three distinct polymorphs of the apohost and nine different inclusion compounds when crystallised from pyridine, morpholine, benzene and their various mixtures. The structures of these inclusion compounds, their thermal stabilities and their kinetics of decomposition will be discussed.

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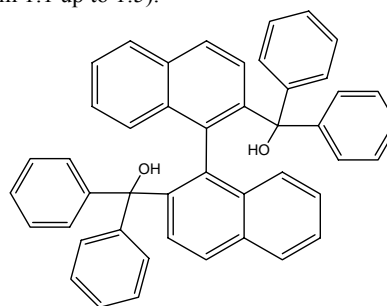
MS18 P05

A bis(hydroxydiphenylmethyl) substituted 1,1'-binaphthyl diol host compound: polymorphism and clathrates with multiple guest species [Hong Su](#) and Luigi R Nassimbeni, *Department of Chemistry, University of Cape Town, Rondebosch 7701, Cape Town, South Africa*. Email: hong.su@uct.ac.za

Keywords: polymorphism, clathrates, single-crystal X-ray analysis

The host compounds based on the scissor-type design proved to be effective clathrate formers[1]. The diol host compound under our study is 2,2'-bis-(diphenylhydroxymethyl)binaphthylene (**H**) (see scheme). Three polymorphous crystalline forms, A, B and C, were discovered during our study, one of which was previously published[2]. All these three forms has been found to co-exist in solution of equal mole ratio of acetone/2-butanol solvent mixture at ambient conditions. Lattice energies were calculated from their respective atomic coordinates using atom-atom pair potentials with Gavezzotti's program OPIX. The results are $-215.4 \text{ kJ mol}^{-1}$, $-232.1 \text{ kJ mol}^{-1}$ and $-210.5 \text{ kJ mol}^{-1}$ for forms A, B and C respectively. This places the stability of these three polymorphous forms in the order B>A>C. This was proved by several slurry experiments using different

solvents, from which form B were constantly obtained. We have carried out competition experiments with three solvents, i.e. pyridine, morpholine and benzene, in different combinations of mixtures at different crystallisation temperatures. A series of clathrates with multiple guest species were obtained: **H**·Morpholine·2Pyridine, **H**·3Morpholine·2Pyridine, **H**·Morpholine·1.5Benzene, **H**·2Morpholine·0.5Benzene, **H**·2Pyridine·Benzene and **H**·Morpholine·Pyridine·Benzene. Analysis of the crystal structures reveals that the molecular structure of **H** is similar in all these compounds. The most characteristic feature of **H** molecule is the presence of an intramolecular hydrogen bond between the two hydroxyls and an intermolecular hydrogen bond to a respective guest. Pyridine is more likely to form hydrogen bond to **H**. Benzene is always hold in the lattice by weak van der Waals interactions. Our study shows a flexible polymorphic host structure with the ability to form clathrates with remarkable high guest ratios (host:guest ratio from 1:1 up to 1:5).



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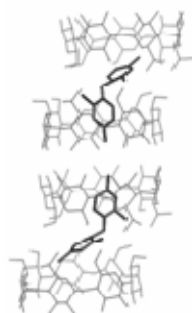
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MS18 P06

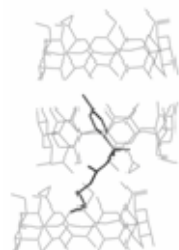
Inclusion complexes of β -cyclodextrin with the Drugs Triclosan and Tolbutamide. [A. Paulidou](#), I. M. Mavridis, *Institute of Physical Chemistry, National Center for Scientific Research "Demokritos", P. O. Box 60228, Aghia Paraskevi 15310, Athens, Greece*
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Keywords: beta-cyclodextrin; triclosan; tolbutamide

Cyclodextrins (CDs) are well known cyclic oligosaccharides, water soluble, used for micro encapsulation of organic molecules inside their relatively apolar cavity, thus they are used extensively to solubilize and/or stabilize apolar/unstable molecules, especially drugs. Triclosan [5-chloro-2-(2,4-dichlorophenoxy)phenol] is a wide spectrum antibacterial agent, infused in an increasing number of consumer products. Inclusion of triclosan in β -cyclodextrin (β CD) cavity finds application in the release of the drug in a controlled way. The β CD/triclosan complex has been crystallized from a water/ethanol mixture and data were collected by synchrotron radiation at 100K (triclinic P1, $a=15.189 \text{ \AA}$, $b=15.23 \text{ \AA}$, $c=16.293 \text{ \AA}$, $\alpha=91.066^\circ$, $\beta=91.049^\circ$, $\gamma=100.709^\circ$). The structure has been refined up to $R_I=0.0658$.



The asymmetric unit contains two β CD molecules and two guest molecules statistically disordered over 2 positions with s.o.f. of 0.5. The stacking of the two host molecules along the *c*-axis generates dimers held together by seven H-bonds via the secondary hydroxyl groups. The geometries of the two guest molecules and their interactions are very similar. The 2,4-dichlorophenyl moiety of the guest is inside the β CD cavity, whereas the 5-chlorophenol expands in the space between the primary sides of two host molecules. The dihedral angle between the two phenyl rings is 81° . Tolbutamide, 1-butyl-3-(*p*-tolylsulfonyl)urea (TBM), is an oral hypoglycemic agent insoluble in water. Complexation of TBM with β CD improves its dissolution rate and solubility, which is believed to be the rate-determining step for its absorption from the gastrointestinal tract.



The inclusion complex of β CD/TBM was crystallized and data were collected at room temperature (monoclinic, *C*2 with $a = 19.225 \text{ \AA}$, $b = 24.547 \text{ \AA}$, $c = 15.708 \text{ \AA}$, $\beta = 109.652^\circ$). The structure has been refined up to $R_f = 0.1054$. Two β CD molecules, related by the 2-fold axis and held together by seven H-bonds via the secondary hydroxyl groups, form also dimers, in the cavity of which a TBM molecule is encapsulated. The aromatic ring of the guest is located in the secondary side of the CD and the chain sticks out between the primary sides of two hosts. The N-atoms of the urea group H-bond to hydroxylic groups of the latter pointing inside. The 2-fold axis of the lattice generates a β CD dimer. However, one guest molecule occupies the dimer cavity in a statistical way (the host: guest ratio is 2:1). The dimers align head-to-head along the *c*-axis forming infinite channels. Channel formation is strengthened by H-bonds between primary hydroxyl groups existing in a variety of orientations.

MS18 P07

Synthesis, characterisation and X-ray study of cobalt (II) complex: [Dimalonato tetraaqua cobalt (II)] $[\text{Co}(\text{C}_4\text{H}_3\text{O}_4)_2(\text{H}_2\text{O})_4]$ Jawher Abdelhak, Sawssen Namouchi Cherni, Mohamed Faouzi Zid & Ahmed Driss *Laboratoire de Matériaux et Cristallographie, Département de chimie, Faculté des Sciences, 2092 El Manar, Tunis, Tunisia.* E-Mail: jawherabd@hotmail.com

Keywords: cobalt compounds, characterization method, magnetic crystal structures.

The investigations of novel complexes represent an intense interest of chemical research; this is driven to a large extent by their interesting properties and their potential practical applications [1-3]. In addition, mixed ligand-metal complexes of some organic oxygen-donors ligands continue to attract attention because they play an important role in biological systems [4].

The title compound was synthesized by slow evaporation of solution at room temperature it has been characterized by X-ray diffraction, IR spectral analysis and UV-vis spectroscopy. The natural elements are detected by the EDX on a scanning electron microscope. The thermal analysis shows that the dehydration of the compound occurs in agreement with the structural features. The temperature dependence of the molar magnetic susceptibility, χ_M , was measured on a polycrystalline sample in the temperature range 5-300 K using a SQUID magnetometer.

The structure was determined by X-ray diffraction on single crystal. This salt crystallizes in the triclinic space group *P*-1 with $a = 5.241(1) \text{ \AA}$, $b = 7.362(1) \text{ \AA}$, $c = 9.268(1) \text{ \AA}$, $\alpha = 109.15(2)^\circ$, $\beta = 104.39(3)^\circ$, $\gamma = 93.15(1)^\circ$, $V = 323.6(3) \text{ \AA}^3$ and $Z = 2$. Crystal structure has been determined and refined to $R = 0.021$ and $wR = 0.057$.

The framework structure of $[\text{Co}(\text{C}_4\text{H}_3\text{O}_4)_2(\text{H}_2\text{O})_4]$ is consisted by Co^{II} ions laying in inversion centers and are six fold coordinated by oxygen atoms from two malonate anions and four water molecules to form a slightly distorted octahedron. The H atoms form hydrogen bonds to neighboring O ensure the cohesion and stability of the molecular packing.

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MS18 P08

Solvent induced transformations in a supramolecular organic zeolite Michela Brunelli^a, Consiglia Tedesco^b, Loredana Erra^b, Ivano Immediata^b, Valeria Cipolletti^b, Carmine Gaeta^b, Placido Neri^b, Andy Fitch^a, *ESRF, BP 200 - 38043 Grenoble, France*, ^b*Department of Chemistry, University of Salerno, Italy.*

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Keywords: calixarenes; supramolecular assemblies; synchrotron powder diffraction

At the Dept. of Chemistry of the University of Salerno we prepared and characterized a new crystalline solid based on *p*-Bu^t-calix[4]dihydroquinone **1** revealing the simultaneous existence of water channels and very large hydrophobic cavities (988 \AA^3). The compound crystallizes from CHCl_3 and ethylacetate with a cubic structure ($a = 36.412(4) \text{ \AA}$) containing 48 calixarene molecules and 155 water molecules in the unit cell. The host supramolecular framework is surprisingly robust and that the material behaves as an organic supramolecular zeolite featuring the simultaneous presence of networked