**MS18 P01**

**A Comparison of the Inclusion Ability of Two Related Hosts** Ayesha Jacobs[a], Kanyisa L. Nohako[a], Luigi R. Nassimbeni[a], Jana H. Taljaard[b], *Department of Chemistry, Cape Peninsula University of Technology, Cape Town, South Africa, [b]Nelson Mandela Metropolitan University, Port Elizabeth, South Africa.* E-mail: jacobsa@cput.ac.za

**Keywords: inclusion compounds, kinetics, organic compounds**

The inclusion ability of two host compounds H1, 9-(1-naphthyl)-9H-xanthen-9-ol, and H2, 9-(1-naphthyl)-9H-thioxanthen-9-ol was studied. We have previously shown that H1 successfully forms inclusion compounds with 1,4-dioxane[1] and acetone[2]. Here we report on the structures of **H1•DMF** and **H1•DMA** where DMF = N,N-dimethylformamide and DMA = N,N-dimethylacetamide. Numerous attempts at forming inclusion compounds between H2 and selected small organic guests including DMF and DMA were unsuccessful and yielded powders of the host alone. Both **H1•DMF** and **H1•DMA** successfully solved in P(-1), the host: guest ratios were 1:1 and their structures were stabilized by hydrogen bonding of the type (Host)-OH•••O-Guest.
Sulphur is less electronegative than oxygen and is less likely to be involved in hydrogen bonding. Interestingly even though the hydrogen bonding in the inclusion compounds of H1 does not involve the ether oxygen of the host, still no inclusion is found for H2.
The kinetics of desolvation for **H1•DMF** and **H1•DMA** were analysed as well as competition experiments.

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**MS18 P02**

**Clathrates with Mixed Guests** Tanya le roex[a], Luigi R. Nassimbeni,[b] Edwin Weber[c] [a]*Department of Chemistry & Polymer Science, University of Stellenbosch, South Africa.* [b]*Department of Chemistry, University of Cape Town, South Africa.* [c]*Institut für Organische Chemie, Technische Universität Bergakademie Freiberg,Germany.* E-mail: tlr@sun.ac.za

**Keywords: clathrates, molecular recognition, crystal structures**

Investigation of the selectivities of two diol organic hosts for pairs of small organic guests gave very interesting results which could be correlated with the crystal structures of the inclusion compounds containing mixtures of the two guests. This study comprises two separate investigations and in the first investigation the selectivity of the host 9,9'-(biphenyl-4,4'-diyl)difluoren-9-ol (**H1**) for a pair of guests with very similar chemical compositions but significantly different boiling points was examined. The second investigation involved a pair of guests with very similar boiling points and which form inclusion compounds with a particular host which have different host:guest ratios when crystallised at different temperatures. We thus investigated the selectivity of the host 9,9'-(ethyne-1,2-diyl)difluoren-9-ol (**H2**) for these guests and carried out the competition experiments at two different temperatures in order to see whether the selectivity would change with changing temperature. Clathrate compounds are an important aspect of supramolecular chemistry and this study displays some novel results showing interesting selectivity profiles, and more importantly giving new insight into clathrates with mixed guests through crystal structure analysis, which in each case could be correlated with the selectivity profiles.

**MS18 P03**

**Inclusion Complexes of β-Cyclodextrin with N-acetyl-L- Tryptophan and N-acetyl-D-Tryptophan** I. M. Mavridis, S. D. Chatziefthimiou, *Institute of Physical Chemistry, National Center for Scientific Research "Demokritos", P. O. Box 60228, Aghia Paraskevi 15310, Athens, Greece.* Email: mavridi@chem.demokritos.gr

**Keywords: beta-cyclodextrin; N-acetyl-tryptophan; crystal structure**

Natural cyclodextrins (CDs), cyclic oligosaccharides consisting mainly of six (*α*CD), seven (*β*CD) or eight (*γ*CD) α-(1,4)-linked D-glucopyranosyl residues have a hydrophobic, relatively rigid cavity able to host a plethora of molecules. Being chiral compounds, CDs can, in principle, discriminate between enantiomers (guests) forming preferentially with one of the two a more stable inclusion complex [1,2]. X-ray crystallography can provide details of the factors that lead to chiral recognition. Structures of CD inclusion complexes with each enantiomer separately suggest that not only the cavity, which undergoes "induced fit" by the presence of the guest, but the guest's H-bonding potential, solvent interactions and packing forces also play a role in chiral discrimination [3].
The crystal structures of the complexes of *β*CD with N-acetyl-L-Tryptophan (triclinic *P*1, a=17.760 Å, b=15.158 Å, c=15.237 Å, *α*=102.774°, *β*=99.346°, *γ*=112.997°) and N-acetyl-D-Tryptophan. (Orthorhombic $C222_1$, a=19.162 Å, b=23.965 Å, c=32.597), with data collected by synchrotron radiation at 100K, are reported presently. In both complexes the *β*CD forms dimers accommodating two molecules of the guest. The differences in the arrangement of the two enantiomers with respect to the host, as well as the impact of the different packing modes are discussed with respect to chiral discrimination.

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**MS18 P04**

**Polymorphism, selectivity and reactivity of inclusion Compounds** Luigi Nassimbeni[a], Hong Su[a], Edwin Weber[b], [a]*Department of Chemistry, University of Cape Town, South Africa,* [b]*Institut fur Organische Chemie, Technische Universitat Bergakademie Freiberg, D-09596 Freiberg, Germany.* E-mail: luigi.nassimbeni@uct.ac.za

**Keywords: inclusion, selectivity, reactivity**

Host molecules which are exposed to a mixture of guests form inclusion compounds whose Guest:Host ( G/H ) ratio depends on the conditions of the crystallisation experiment. Thus the G/H ratio tends to decrease with rising crystallisation temperature [1] while the

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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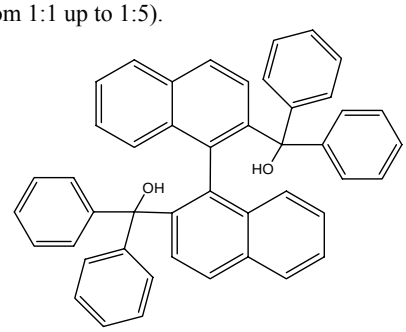
## MS18 P05

**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 kJ $mol^{-1}$, -232.1 kJ $mol^{-1}$ and -210.5 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 crytsal structures reveals that the molecular structure of **H** is similar in all these compounds. The most characteritic 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 remarkeable high guest ratios (host:guest ratio from 1:1 up to 1:5).

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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*
Email: paulidou@chem.demokritos.gr

**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 *P*1, a=15.189 Å, b=15.23 Å, c=16.293 Å, *α*=91.066˚, *β*=91.049˚, *γ*=100.709˚). The structure has been refined up to $R_1$= 0.0658.