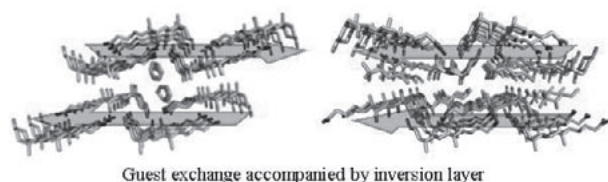


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Solid-state dynamic properties contribute to development of gas sorption and storage materials with microporous coordination polymers. Organic crystals function as dynamic materials due to their flexibility and diversity. We study on steroidal bile acid derivatives which serve as host components and form dynamic inclusion crystals for intercalation and enantioresolution of guest molecules. So far, we reported that cholamide has various types of flexible bilayers where secondary aliphatic alcohols are accommodated. Among them, 2,2-dimethyl-3-hexanol induces a rare bilayer structure which is responsible for high enantioselectivity. Here we present intercalation and enantioresolution of 2,2-dimethyl-3-hexanol by using inclusion crystals of cholamide with 1,4-dioxane. It was found that guest exchanges took place with retention of the crystalline state in appearance. The crystal structures were determined before and after the intercalation by means of powder X-ray diffraction, indicating that the intercalation accompanied layer inversion on the lipophilic sides of the bilayers. Moreover, it was found that the resulting crystals include (*S*)-2,2-dimethyl-3-hexanol in over 95% ee yield.



Keywords: inclusion compound, chiral recognition, intercalation

## P06.07.28

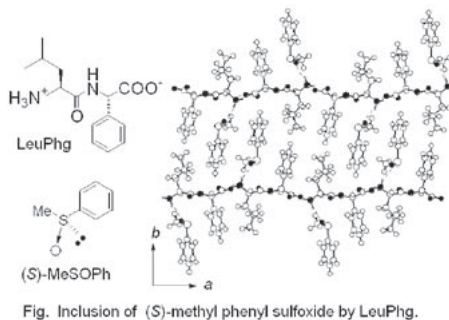
*Acta Cryst.* (2008). A64, C391

### Enantioselective inclusion of methyl phenyl sulfoxides by (*S*)-alkylglycyl-(*S*)-phenylglycine

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As dipeptide host molecules, (*S*)-alkylglycyl-(*S*)-phenylglycines were examined in terms of enantiomeric inclusion for racemic methyl phenyl sulfoxides. Among them, (*S*)-leucyl-(*S*)-phenylglycines (LeuPhg) and (*S*)-isoleucyl-(*S*)-phenylglycines (IlePhg) mainly included *S*-form of methyl phenyl sulfoxides with high enantioselectivity. By single crystal X-ray analyses of these inclusion compounds, it was elucidated that the dipeptide molecules self-assembled to form layer structures and included the sulfoxides between these layers by hydrogen bonding between the proton of  $^+NH_3$  and the oxygen of the sulfoxide. In the cavity, *C*-terminal phenyl group of the dipeptide interacts



with the phenyl group of sulfoxides. In addition to these host-guest interactions, the two homochiral sulfoxides belonging in upper and lower layers make a pair having 2-fold rotation axis or 2-fold screw axis along the channel cavity. In other words, the self-recognition of sulfoxides made a homochiral pair to achieve high enantioselectivity.

Keywords: inclusion compounds, dipeptides, molecular recognition

## P06.02.29

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### Structure and polymorphism of trans mono-unsaturated triacylglycerols

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Trans fats are in a natural way in small quantities present in animal foods. Moreover, unsaturated plant fats are often partially hydrogenated to raise melting temperatures for preparation of foods, and then, as a minor side reaction, a small part of the *cis*-bonds will change into *trans*-configuration. Like saturated fats, fats with *trans* fatty acid residues will have negative effects for human health (1). The presence of the elaidoyl chain, one of the major *trans* fatty-acid chains, is suspected of increasing health risks because it resembles the stearoyl chain. When incorporated in biological membranes elaidoyl chains will be influencing the physical-chemical properties of the membranes. Thus insight in the influence of the differences between the fatty-acid composition of *trans* and saturated triacylglycerols (TAGs) on the conformation and packing, on polymorphic stability, and on phase-transition behavior, will be useful. By combining X-ray powder diffraction (XRPD) techniques a better understanding of *trans* mono-unsaturated TAGs and their related saturated ones can be obtained. Synchrotron and advanced laboratory time- and temperature-resolved XRPD reveal the stability and phase-transition behavior of the important polymorphs like the  $\beta$  and  $\beta'$ . These results can be related to the underlying crystal structure packing that can be obtained from XRPD data using direct-space search techniques. Our recent results will be discussed including novel meta-stable  $\beta'$  polymorphs and the structure of one them, methyl-end plane packing analysis in relation to observed melting points for various subgroups of TAGs, and the difference in  $\beta'$  to  $\beta$  phase-transition behaviour of symmetric versus asymmetric TAGs.

(1) The EFSA Journal, 2004, 81, 1-49

Keywords: *trans* mono-unsaturated triacylglycerols, time-temperature-resolved diffraction, powder structures

## P06.10.31

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### Polymorphism and structure solution from powder data of *N,N'*-1,4-phenylene-bis(3-oxobutanamide)

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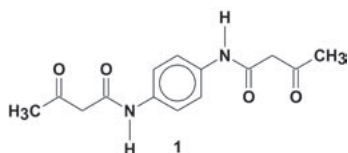
*N,N'*-1,4-Phenylene-bis(3-oxobutanamide) (**1**) is an industrial intermediate which is used as a coupling component in the synthesis

of organic azo pigments; it serves as a linker between two azo moieties [1]. Despite of its commercial importance, crystal structures of **1** are not known up to now. We were able to grow crystals from 1-butanol using re-crystallisation techniques. The X-ray structure analysis showed that the crystal was a monohydrate of **1**. We dried the powder of **1** at 120°C and measured its X-ray powder pattern. The powder was monophasic and we were able to determine the structure from the powder pattern using DASH [2] and TOPAS [3]. This polymorph turned out to be the anhydrous form. A third polymorphic form was obtained by drying the mono-hydrate at 70°C. The resulting powder pattern contained eight reflexions only, and the crystal structure could not be solved yet.

[1] W. Herbst, K. Hunger, *Industrial Organic Pigments*, 3rd ed., Wiley-VCH, Weinheim, 269-270 (2004).

[2] W. I. F. David, K. Shankland, J. van de Streek, E. Pidcock, W. D. S. Motherwell, J. C. Cole., *J. Appl. Cryst.*, **39**, 910-915 (2006).

[3] A. A. Coelho, TOPAS Academic, version 4.1, 2007.



Keywords: polymorphism, organic compounds, powder structure determination

### P06.10.32

*Acta Cryst.* (2008). A64, C392

#### Crystal structures of two insect recognition pheromones

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Social insects distinguish their nest mates from aliens via the recognition pheromones that are part of the complex mixture of waxy chemicals found on the cuticle. Among these, methyl-branched alkanes appear to act as pheromones, whereas straight-chain alkanes do not. Thus the stereochemistry associated with the methyl group, or the conformation of the molecule imposed by this group, may affect the binding of the molecule to the receptors in an insect's antennae. Enantiomerically pure methyl-branched alkanes have been synthesised and their crystal structures determined by high resolution powder diffraction, or by single crystal diffraction, using synchrotron radiation. 11-methyl nonacosane and 7-methylnonacosane are both monoclinic, space group  $P2_1$ ,  $Z = 2$ ,  $a = 15.582 \text{ \AA}$ ,  $b = 7.144 \text{ \AA}$ ,  $c = 14.096 \text{ \AA}$ ,  $\beta = 114.913^\circ$  and  $a = 28.172 \text{ \AA}$ ,  $b = 4.935 \text{ \AA}$ ,  $c = 10.375 \text{ \AA}$ ,  $\beta = 90.30^\circ$ , respectively. The two compounds have distinct molecular packing arrangements in the structure. Moreover, molecules in 11-methylnonacosane are very straight, whereas 7-methylnonacosane molecules show a slight bend in the chain near the methyl group.

Keywords: alkanes, powder diffraction, synchrotron diffraction

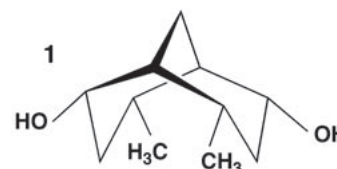
### P06.09.33

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#### Five alternative crystal forms produced by crystallisation of a simple alicyclic dialcohol

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The simple racemic dialcohol **1** formed inclusion compounds in all recrystallisation experiments attempted by us. Many of these crystals are isostructural clathrates of stoichiometry 3(host).1(guest) that are formed in the cubic space group  $Ia\bar{3}$ . However, two further inclusion crystal forms are also produced, namely 6(host).1(*p*-xylene) and 2(host).1(water), from these specific solvents. If the cubic crystals are sublimed, then the guest molecules are lost and solvent-free apohost crystals are formed instead in the monoclinic space group  $P2_1/c$ . Finally, resolved **1** crystallises solvent-free in the tetragonal space group  $P4_122$  (or its enantiomorph). All of these five very different crystal forms utilise different hydrogen-bonded supramolecular synthons to create their structures. These arrangements will be described and contrasted. In particular, it will be argued that the most common cubic crystal form is a supramolecular cousin of the well-known host Dianin's compound, despite the very different molecular structures of the two compounds.



Keywords: hydrates and clathrates, hydrogen bonding in organic crystals, inclusion compounds polymorphism

### P06.07.34

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#### <sup>1</sup>H NMR application to inclusion chemistry: Selectivity profiles of a xanthenol-related host

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10,11-Dihydro-5-(1-naphthyl)-5H-dibenzo[a,d]cyclohepten-5-ol (H) is a derivative from the xanthenol family. We obtained its alpha-structure by recrystallisation at room temperature in ethanol. Subsequently, we crystallised it at room temperature from pyridine (Pyr), 1,4-dioxane (Diox) and DMSO. In all cases, we obtained 1:1 host:guest inclusion compounds displaying hydrogen bonding between the host and the guest molecules. The three structures belong to very different space groups ( $C2/c$ ,  $P-1$  and  $P2_1/c$  respectively for pyridine, 1,4-dioxane and DMSO) which was interesting to consider when carrying out competition experiments between the solvents with H. We characterized the selectivity profile of our host H with the three solvents using <sup>1</sup>H NMR. We have carried out competition experiments between pairs of guest molecules and characterized the ensuing inclusion compounds formed. We rationalise the structural results with the molecular properties of the guests in terms of their donor/acceptor capacities.

Keywords: inclusion compound, host-guest chemistry, crystal structure