

this compound is crystallized from solutions in acetophenone as a host-guest complex with a ratio 2:3. In the crystal structure DEDA molecules form infinite chains in a direction [01-1] through a pair of centrosymmetric hydrogen bonds. The acetophenone molecules are situated inside different channels formed at stacking of these chains and running along [100] and [0-11] directions.

At decreasing of crystallization temperature until 5°C from the same solution DEDA crystallizes as new complex - hydrate with 1:3 host-guest ratio. In the crystal structure one carboxylic group of DEDA molecules is connected via centrosymmetric H-bonding with the carboxylic group of the other host molecule, while other carboxylic group is deprotonated giving rise to a network of intermolecular H-bonds associating with one ion of hydroxonium (H_3O^+) and two molecules of water. The structure may be described as intercalate type complex with strict separation of the hydrophobic and hydrophilic areas.

L.I. is grateful to NATO Reintegration Grant (Rig.Ref. 980768) for scholarship.

[1] Weber E., Csöregi I., Ahrendt J., Finge S., Czugler M., *J. Org. Chem.*, 1988, **53**, 5831-5839.

Keywords: versatile host, host-guest complex, intercalate

P.06.07.5

Acta Cryst. (2005). A61, C288

Different Building Modes of α -Cyclodextrin/Monoalkyl Amphiphile Complexes

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In this study, the impact of the length of the guest molecule alkyl chain and the crystallization conditions on the structural parameters of α -cyclodextrin (α -CD)/monoalkyl complexes was determined. Several procedures to crystallize those complexes were developed for different alkylalcohols as model guest molecules, as a function of temperature. Three different crystalline structures were identified depending on the alkyl chain length, using synchrotron X-ray diffraction (LURE, Orsay, France). In all cases, complexes crystallize in channel-type structures, where α -CD molecules are stacked like coins in a roll and the alkyl chain of the guest compound is embedded in the tubular cavity of the α -CDs. However, depending on the length of the chains and the crystallization conditions, the channels are organized differently. C_6 - C_8 chains give rise to a pseudo-hexagonal lattice, a packing mode already observed for polyiodide complexes [1]. C_{10} - C_{12} chains crystallize in a triclinic pseudo-monoclinic C2 lattice, while longer chains up to C_{18} form hexagonal crystals with R3 symmetry. These two novel crystal structures are described. Understanding these structures opens new routes to nanotube formation through amphiphile-driven crystallization of cyclodextrin templates.

[1] Noltemeyer M., Saenger W., *J. Am. Chem. Soc.*, 1980, **102**, 8, 2710.

Keywords: cyclodextrin, nanotubes, supramolecular assembly

P.06.07.6

Acta Cryst. (2005). A61, C288

Inclusion Compounds of Isomeric Xanthenol Hosts with Aniline

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Two isomeric xanthenol host compounds have been found to form inclusion compounds with aniline. These hosts are H1 = 9-(4-methoxyphenyl)-9H-xanthen-9-ol and H2 = 9-(3-methoxyphenyl)-9H-xanthen-9-ol. We have elucidated the structures of the inclusion compounds and determined their kinetics of desolvation. H1•½aniline crystallises in the triclinic space group $P\bar{1}$ with the host in general positions and the aniline guest on a centre of symmetry. H2•aniline

was solved successfully in the monoclinic space group $P2_1/c$ with both the host and guest molecules in general positions. For H1•½aniline there is (Host)—OH•••O—(Host) hydrogen bonding whereas in H2•aniline (Host)—OH•••N—(Guest) hydrogen bonding occurs. We have correlated the structures with the thermal stabilities of the compounds.

Keywords: isomeric hosts, aniline, desolvation

P.06.07.7

Acta Cryst. (2005). A61, C288

Structural Features of Some Schiff Base Disulfide Compounds

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Schiff bases bearing imine N and anionic S atoms constitute an important class of polydentate ligands and their metal complexes have previously been used as models for biological systems. The molecule and crystal structure of a new synthesized disulfide compound [$\text{C}_{30}\text{H}_{22}\text{F}_6\text{N}_2\text{O}_2\text{S}_2$] has been undertaken with a view to obtaining accurate structural parameters of interest in disulfide compounds. Crystal data: $M=620.62$, Triclinic, $a=7.639(2)\text{Å}$, $b=8.526(8)\text{Å}$, $c=23.349(5)\text{Å}$, $\alpha=89.04(4)^\circ$, $\beta=89.99(2)^\circ$, $\gamma=63.41(4)^\circ$, $V=1359.6(9)\text{Å}^3$, $P\bar{1}$, $R=0.0538$, $R_w=0.0944$. The structure was solved by direct methods and refined by least squares on F_{obs}^2 by using SHELX-97.

In the second phase of the study, structural results have been compared with the values found in our previous studies related at least four Schiff base disulfides [1-4]. The molecular conformation around central S-S bond has been affected by trifluoromethyl groups in the molecule. High electronegativity in the CF_3 groups has been cause to conformational changes in the torsion angle of C-S-S-C [$77.8(4)^\circ$]. Two strong intramolecular hydrogen bonds [O-H•••N, O•••N: 2.612(9) and 2.612(8)Å] have been observed and cause to increasing of the planarity in the main parts of the molecule.

[1] İde S., Öztaş G., Ancin N., Tüzün M., *Acta Cryst.*, 1997, **C53**, 376-378. [2] Özbey S., Temel A., Ancin N., Öztaş S.G., Tüzün M., *Z. Kristallogr.*, 1998, **213**, 207-208. [3] Candan M.M., İde S., Kendi E., Öztaş G., Ancin N., *Spectroscopy Letters*, 1998, **31(4)**, 891-900. [4] İde S., Ancin N., Öztaş S.G., Tüzün M., *Pharm. Acta. Helv.*, 1998, **72**, 291-294.

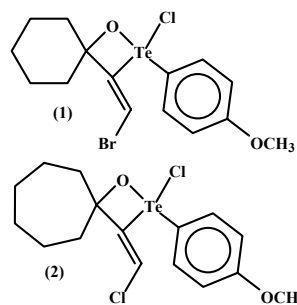
Keywords: Schiff base, disulfides, crystal structure

P.06.07.8

Acta Cryst. (2005). A61, C288-C289

Structural Studies of Human Cathepsin B Inhibitors: Telluroxetanes

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The inhibition of cathepsin B has been postulated to be directly responsible for the abrogation of the invasion process in several tumor cells lines [1], and, as it was shown that AS-101 [2], a Te^{IV} compound, was a cathepsin B inhibitor, compounds (1) and (2) were synthesized and studied.

In both compounds, if intra and two intermolecular secondary bonds and the electron lone pair are considered, then the Te^{IV} is coordinated in a ψ -pentagonal bipyramidal fashion. The secondary interactions join the molecules in chains of centrosymmetric dimers. These compounds, have higher second-order rate constants for the inactivation of cathepsin B, than that of AS-101. Moreover, the compound with a cyclohexane ring is 20-fold more active than (2) and 4-fold than (1), so that it can be

postulated that these differences are due to the nature of the halogens, or to the increase of the cycloalkane ring, which modify their electronic and steric characteristics.

[1]Sinha A.A., Jaumar M.P., Wilson M.J., Rozhin J., Sloane B.F., *Prostate*, 2001, **49**, 172. [2] Albeck A., Weitman H., Sredni B., Albeck M., *Inorg. Chem.*, 1998, **37**, 1704.

Keywords: tellurium complexes, cathepsin, inhibitors

P.06.07.9

Acta Cryst. (2005). A61, C289

Structural Variety in Tris(5-acetyl-3-thienyl)methane (TATM) Inclusion Compounds

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Tripodal host molecules form a versatile class of supramolecular materials which include triphenylmethane and analogues such as TATM. TATM host-guest inclusion compounds display a wide variety of stoichiometry and structural motifs and they have demonstrated a propensity for polymorphism. Indeed we have obtained five different forms of the 2:1 host guest compound of TATM / 1,3 dichloropropane. Four of the five polymorphs can be described as polytypes that share both a similar layer motif and a similar conformation of the TATM's thienyl rings. Over the past several years we have examined the structure of more than 20 TATM inclusion compounds. In all but one example the host TATM molecules adopt one of two distinct conformations as first discussed by Herbstein [1]. The TATM frameworks that share a similar conformation may be described as polytypes composed of common layer motifs. Differences in the layer stacking leads to a variety of space groups.

In the TATM frameworks the host molecules interact through π - π and C-H...O interactions to form layers. In all the studies the host and guest interact very weakly through van der Waals forces. The disordered guest molecules are dynamic and located in channels or cages within or between the framework layers. The long-chain guests span more than one layer. Some of the frameworks appear stable upon removal of up to 90% of the guest species.

[1] Herbstein F.H., *Acta Cryst.*, 1997, **B53**, 168.

Keywords: inclusion compounds, polytypes, molecular packing

P.06.07.10

Acta Cryst. (2005). A61, C289

Molecular Recognition of Racemic Salts by Inclusion Complexation with the Chiral BINOL

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Optically active 2,2'-dihydroxy-1,1'-binaphthyl (BINOL) have found many applications, ranging from chiral ligands in catalysts for asymmetric reactions, to hosts for molecular recognition and enantiomeric separation, and as intermediates for the synthesis of chiral materials.

We report here a chiral resolutions of racemic ammonium salts by the inclusion crystallization with (S)-BINOL (**1**) as the optically pure host. Resolution was very efficient for 3-hydroxyquinuclidinium chloride (**2**), but similar resolution of N-methyl-2-hydroxymethyl-piperidinium chloride (**3**) was failed, although (**3**) formed an inclusion complex with (**1**). X-ray structures of these complexes were studied in order to characterize the intermolecular interactions and recognition scheme.

The crystal structure of the complex (S,S)-**1-2** consists of the hydrogen bonded chains formed by three OH...Cl bonds, two with two different host molecules and one with the guest. There is also an intramolecular N-H...O bond in guest molecule. In the complex (S,S,S)-**1-3** the chloride anion participate in H-bonds in the same manner. However, an extended three dimensional hydrogen bond

networks are obtained through the additional N-H...O bonds with one OH group of the host molecules.

Keywords: inclusion complexes, molecular recognition, H-bond

P.06.07.11

Acta Cryst. (2005). A61, C289

Crystal Structure of a Histidine Schiff Base

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Threonine and histidine are essential aminoacids which have big importance in bioorganic and medicinal chemistry. Their Schiff bases are also important in biological systems because of their functional role in reactions, transaminations, racemizations and decarboxylations [1].

At the beginning of our research program on aminoacid Schiff bases, threonine Schiff base [N-(2-hydroxy-1-naphthylidene)threonine] has been isolated and investigated [2]. In this work, we would like to present an x-ray investigation of a new synthesized histidine Schiff base [N-(2-hydroxy-1-naphthylidene)histidine] as the second part of our studies. The crystal belongs to orthorhombic, space group P2₁2₁2₁ with the following crystallographic parameters: a=6.133(5), b=7.168(5), c=33.639(5) Å, V=1478(2) Å³. The final R factor: R=0.0394, wR=0.0784. These type Schiff bases usually show photochromic or thermochromic characters depending on their tautomeric forms [3,4]. According to the present crystallographic results, histidine Schiff base prefer keto-amine tautomerism rather than enol-imine tautomerism which was observed in the structure of threonine Schiff base. A strong intra-molecular hydrogen bond [N-H...O: 2.592(9)Å] indicated that proton transfer from hydroxy group of naphthalidene to the nitrogen atom is favoured by the charge distribution of the (keto-amin) resonance form.

[1] Wilkins P.C., Wilkins R.G., *Inorganic Chemistry in Biology*, Oxford University Press, Oxford, 1997, 9. [2] Özcan Y., İde S., Şakıyan İ., Logoğlu E. *J. Mol. Struct.*, 2003, **658**, 207-213. [3] Kaitner B., Pavlovic G., *Acta Cryst.*, 1996, **C52**, 2573-2575. [4] Elerman Y., Kabak M., Elmalı A., Suobada I., *Acta Cryst.*, 1998, **C54**, 128-130.

Keywords: schiff base, histidine, X-ray analysis

P.06.07.12

Acta Cryst. (2005). A61, C289-C290

Weak Intermolecular Interactions in Cavitands as Receptors for Mass Sensors

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Cavitands, synthetic organic compounds with enforced cavities of molecular dimensions, are extremely interesting and versatile molecular receptors whose complexation properties mainly derive from H-bonding, CH- π and dipole-dipole interactions [1].

The rational design of organic hosts is particularly appealing to chemical sensor technology, which requires selective, sensitive and stable receptors. The use of supramolecular structures coated as thin layers on quartz crystal microbalances (QCM) has proved to be one of the best approaches to generate new materials with molecular specificity for chemical sensing [2].

Following these studies, convenient phosphorus-bridged cavitands were designed and synthesized to investigate their complexation properties towards linear alcohols [3]. X-ray diffraction on single crystals has been widely used to study the multiple binding interactions in these phosphonate cavitands (hydrogen bonds involving the PO groups and CH- π interactions with the π -basic cavity). It has been thus evidenced that the synergistic behaviour of an increasing number of convergent PO groups enhances the complexation of the guests, giving rise to entropically favoured complexes.

[1] Dalcanale E., Jacopozi P., Uguzzoli F., Mann G., *Supramol. Chem.*, 1998,