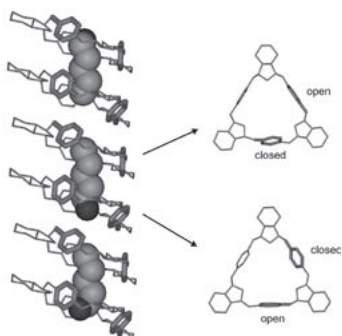


intramolecular inclusion of simple solvent molecules. Bridging of the neighboring nitrogen atoms by the methylene groups gives rise to more rigid macrocycle with a well defined inner cavity. We shall demonstrate the ability of this molecule to include in its inner cavity simple alcohol molecules belonging to the homologous series (ethanol, 1-propanol, 1-pentanol and 1-hexanol). Of interest in the investigated crystal structures is a concerted host/guest disorder and the ability of the host molecules to switch in between the various conformations which either allow or prevent the solvent molecules to enter the intramolecular cavity. Our studies also demonstrate that the inner cavity of trianglamines is not large enough to accommodate any aromatic guest molecule.



Keywords: inclusion, macrocycles, triaglamines

### P06.07.39

*Acta Cryst.* (2008). A64, C394

#### Molecular complexes of 4,4'-dinitrophenyl

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Complexes of para disubstituted and 4-monosubstituted biphenyl formed with 4,4'-dinitrophenyl (DNBP), demonstrate intense colours, from pale yellow to dark red, upon formation. These colours are dissimilar to the colour combination of the parent compounds. The focus of this study was to investigate the nature of these molecular donor-acceptor interactions in the solid state, using spectroscopic techniques such as IR, Raman, UV-Vis, NMR and X-ray crystallography. Typical interactions observed in such molecular complexes include pi-pi interactions, hydrogen bonding, charge transfer and van der Waals interactions. Complexes of DNBP, as acceptor, studied included a variety of mono- and disubstituted donors, such as dihalo, diamino, di- and monohydroxy groups. The crystal structures of these complexes showed retention of the non-planar conformation of DNBP with a dihedral angle of around 35 deg. This conformation for DNBP has also been confirmed using density functional theory (Guassian) calculations that showed good agreement between the theoretically calculated and experimentally observed IR and Raman spectra in the solid state. It appears as if the packing of the complexes in the solid state is directed mainly by the similar packing of DNBP units in these complexes. Some of the molecular ratios for these complexes that vary, depending on the electronic properties of the donor molecules, were determined using NMR spectroscopy.

Keywords: molecular complexes, molecular cocrystals, crystallographic analysis

### P06.07.40

*Acta Cryst.* (2008). A64, C394

#### (Z)-Ethyl 4-chloro-2-[(4-chlorophenyl)-hydrazino]-3-oxobutanoate

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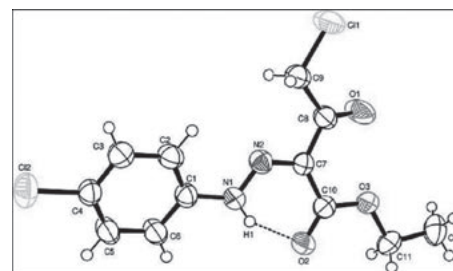
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The title compound, (Z)-Ethyl 4-chloro-2-[(4-chlorophenyl)-hydrazino]-3-oxobutanoate, crystallizes as a nonmerohedral twin with a twinning ratio of 0.51:0.49. The molecule adopts a keto-hydrazo tautomeric form stabilized by an intramolecular N-H...O hydrogen bond. The configuration around the N-N bond is trans. The overall view and atom-labelling of the molecule are displayed in Fig.1. The molecule is approximately planar with dihedral angle between the aromatic C1-C6 ring and the plane of the C7-C12/O1-O3/C11 aliphatic chain being 19.71(12) degrees. Intramolecular N-H...O hydrogen bond generate S(6) ring motif (Bernstein et al., 1995).

Reference:

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* 34, 1555 – 1573.

Fig.1.



Keywords: single-crystal structure determination, tautomerism, small organic molecules

### P06.08.41

*Acta Cryst.* (2008). A64, C394–395

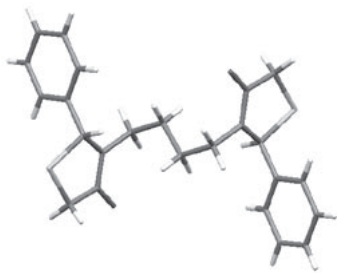
#### Synthesis and structural characterization of 1,4-bis-(2-phenyl-4-oxo-1,3-thiazolidin-3-yl)butane

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Thiazolidinones are important heterocyclic compounds, which exhibit a broad range of biological activities, including interesting profile as fungicidal, pesticide, antibacterial, anticonvulsant, antihistaminic, antioxidant, anti-inflammatory and antinociceptive agents. As a consequence many different protocols allowing the synthesis of 4-thiazolidinone skeletons have been developed. However, few are known about a,w-bis-(2-hetaryl-4-oxothiazolidin-3-yl)alkanes. Even though, several studies have revealed their anti-

inflammatory, antihistaminic and analgesic activities. Moreover, recently, it was found that some bis-(2-aryl-4-oxothiazolidin-3-yl)ethanes act as good cyclooxygenase-2 inhibitory agents. The crystallographic characterization of the bis-heterocycle 1,4-bis-(2-phenyl-4-oxo-1,3-thiazolidin-3-yl)butane belonging to a family of compounds synthesized through a one pot three component condensation methodology, with acetonitrile as solvent are reported herein. This compound crystallizes in a monoclinic cell with the cell parameters  $a=5.7452\text{\AA}(11)$ ,  $b=27.065\text{\AA}(5)$   $c=7.1157\text{\AA}(14)$  and  $\beta=105.53^\circ(3)$ , Space group  $P2_1/c$  [No 14],  $V=1066.05\text{\AA}^3$  and  $Z=2$ .



Keywords: single-crystal, thiazolidinone, structural characterization

### P06.09.42

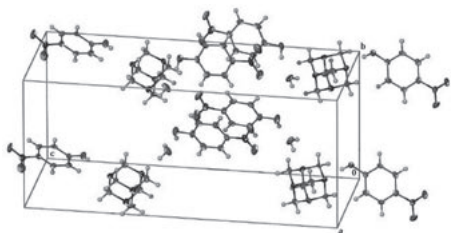
*Acta Cryst.* (2008). A64, C395

#### Hexamethylenetetramine bis(*p*-nitrophenol) monohydrate clathrate

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Hexamethylenetetramine bis(*p*-nitrophenol) monohydrate,  $(\text{CH}_2)_6\text{N}_4 \cdot 2\text{HO}-\text{C}_6\text{H}_4-\text{NO}_2 \cdot \text{H}_2\text{O}$ , crystallizes as four independent formula units in the  $P1$  space group [ $a$  6.9325(1),  $b$  11.6867(2),  $c$  25.0826(5) Å;  $\beta$  96.728(1),  $\beta$  92.449(1),  $\beta$  89.971(1)°]. The eight *p*-nitrophenol and four water molecules each function as a hydrogen-bond donor to two acceptor (nitrogen and oxygen) sites. Only two of the four nitrogen sites of each hexamine molecule are involved in hydrogen bonding. The hydrogen bonds connect the component molecules into a linear chain. Diffraction measurements were made at  $-173^\circ\text{C}$ . A previous room-temperature study has found a monoclinic  $C2$  polymorph [ $a$  49.989(4),  $b$  5.901(1),  $c$  7.056(1) Å;  $\beta$  92.423(1)°]. The *p*-nitrophenol molecule is disordered about a two-fold axis; the *p*-nitrophenol, hexamine and water molecules are also linked by hydrogen bonds into a linear chain (Ng et al. (2001). *J. Mol. Struct.* 595, 189-194].



Keywords: clathrate, polymorph,  $P1$  space group

### P06.09.43

*Acta Cryst.* (2008). A64, C395

#### Structure of the anhydrous form of gossypol - Dianhydrogossypol

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Gossypol,  $\text{C}_{30}\text{H}_{30}\text{O}_8$ , is yellow pigment containing in glands of various species of cotton plants. By chemical modification of gossypol a great number of gossypol derivatives - its ethers, Schiff's bases, etc. - have been obtained. Dianhydrogossypol which is formed by dehydration of gossypol is of a special interest for our studies. The crystals of dianhydrogossypol were obtained from saturated solution in dichloromethane. Data collection has been carried out on a Gemini R X-ray diffractometer equipped with an Oxford Cryosystems open-flow cryostat at 100 K. The crystallographic parameters are:  $a=33.8265(4)$ ,  $b=33.8265(4)$ ,  $c=9.1497(2)\text{\AA}$ ,  $V=4677\text{\AA}^3$ , Sp.gr.-  $I4_1/a$ . The results of the X-Ray studies show that molecular structure confirms anticipated one. The crystal structure has wide channels in the direction of  $c$ -axis and resembles the channel structure found earlier in one of gossypol polymorphs (zeolite-like structure). The packing factor of these crystals is very low and equal to 0.59. The volume of empty space of the an elementary cell is equal to 1641 Å<sup>3</sup> or 15,7 % of the total volume. The dianhydrogossypol demonstrates good inclusion ability for molecules of many small volatile compounds which requires further detailed investigations in relation to potential new organic zeolite.

Acknowledgements. This work was supported by STCU/ARS USDA grant - P181

Keywords: X-ray structural crystallography, inclusion compounds, zeolites

### P06.10.44

*Acta Cryst.* (2008). A64, C395-396

#### A study on the effect of substitutions and intermolecular interactions in thiophene 3-carboxamides

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The study of interactions involving halogens, particularly fluorine is a major interest in crystal engineering<sup>1</sup>. Conventional hydrogen bonding is the significant interaction in many crystal structures but a number of weak interactions have been also shown to play a role in stabilization of crystal structure. In view of these, as a part of our research on structural studies on biologically active molecules, we have reported intermolecular interactions in fluorinated compounds and some substituted thiophene 3-carboxamide derivatives<sup>2</sup>. These compounds were found to exhibit broad spectrum of biological activities such as antibacterial, antifungal and anti-inflammatory activities<sup>3</sup>. Our present research work describes the crystal structure and conformational studies of 2-amino thiophene 3-carboxamides and Schiff bases of thiophenes<sup>4</sup>, which serve as starting material for a number of intermediate derivatives. It is noticed from the comparative study that the chloro substitution in the aryl amide group had a significant effect on crystal packing. The ortho -chloro group reversed the orientation of the amide linkage and favoured the formation of more intra molecular hydrogen bonds. The para -chloro