

interactions and crystal packing motifs.

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Keywords: high-pressure polymorphism, crystal growth from solution, antibiotics

### P05.03.04

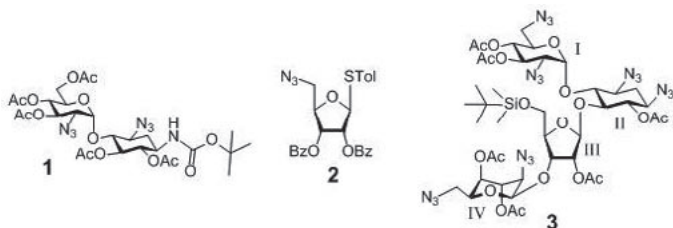
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#### Behaviour of the azido group in crystal structure of the intermediates of aminoglycoside antibiotics

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Aminoglycoside antibiotics are function through binding to specific sites in prokaryotic ribosomal RNA and affecting the fidelity of protein synthesis. Compounds **1-3** are the precursors of new derivatives of neomycin B and paromomycin. Conformational disorders of the azide groups at these compounds were observed. Compound **3** consists of pyranose (I and IV), furanose (III) and an aminocyclitol (II) rings joined together via glycosidic linkages. Compounds **1** and **2** are smaller by the number of the rings and of the azide groups. All azido groups are in a linear arrangement. In **2** it has usual geometry. The azide groups in **1** and **3** are significantly disordered. In **1** one of the azide group gave the ratio of 0.65:0.35 between the two positions, which lead to a significant distortion from the linearity. In **3** at least three from the six N<sub>3</sub> groups are disordered: in two the ratio between two possible conformations are 0.60:0.40 and in one- 0.80:0.20. For all of them the N=N=N angle is smaller than the literature value and most of the N=N bonds are shorter than usual bonds. Such a flexibility of the azido groups suggests that they are present in two resonance structures N=N=N and N-N=N.



Keywords: difficult structures, antibiotics, azides

### P05.04.05

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#### Crystallography of steroids: A comparative analysis of geometrical features and hydrogen bonding

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Steroids are a class of biologically important organic compounds which have extensively been studied crystallographically for the last over fifty years. In view of the striking properties as exhibited by this

class of materials, we have classified the cholestane (C27) into four types, viz. cholane, pregnane, androstane, oestrane, etc. and have carried out a detailed study on each of its classes to (i) compare selected bond distances and angles and their deviations from the accepted values vis-a-vis the substitutional group and X-H...A intra and intermolecular interactions, (ii) study asymmetry parameters and the importance of hybridization and ring fusions for the conformation of individual ring systems, (iii) investigate the incidence and role of X-H...A intra- and intermolecular interactions in molecular entities of this kind, (iv) prepare a database on d(H...A), D(X...A) and theta(X-H...A) range based on the cut-off criterion as proposed by Desiraju(1999), (v) study the effect of solvent on the properties of steroidal molecules and investigate the solvent-solute/solute-solvent and solvent-solvent interactions in a hydrogen bonded network. Some part of the work has been published in *Acta Cryst B* 2007, *Z.Kristallographie*, 2007., *Journal of Chemical crystallography*, 2007., *Ind. J. Biochemistry and Biophysics* 2007. A comprehensive presentation of the entire work will be made.

Keywords: crystallography, steroids, molecular structures

### P05.05.06

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#### Crystal and molecular structure of the diethyl ester of rhodoporphyrin

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Natural product plant porphyrins are a relatively underrepresented class in the structural databases. The current work reports the crystal and molecular structure of ethyl 8,13-diethyl-18-ethoxycarbonyl-3,7,12,17-tetramethylporphyrin-2-propionate (the diethyl ester of rhodoporphyrin) isolated from the leaves of the Thai traditional medicine plant, *Bridelia ovata* Decne, also commonly called *maka* in Thai. The structure determination was straightforward with a minor disorder of one ethyl group (refined occupancy ratio 0.77(1):0.23). The ethoxycarbonyl group is fixed to be coplanar with the porphyrin core by a concerted group of five intramolecular C – H...O interactions, while the ethyl propionate groups are oriented perpendicular to the porphyrin cores forming ribbons of intra- and inter-molecular C – H...O interactions. The other dominant packing feature is  $\pi\cdots\pi$  stacking of the porphyrin planes, which are required to be parallel by symmetry. Experimental Data: From the leaves of *Bridelia ovata* Decne; intense violet plate crystals, recrystallized from hexane-dichloromethane solution; MP 194-196 °C . Crystal data: C<sub>36</sub>H<sub>42</sub>N<sub>4</sub>O<sub>4</sub>, *M*<sub>w</sub>=594.74; 0.4 x 0.3 x 0.05 mm; triclinic *P* – 1 (No. 2), *a*=9.409(2), *b*=12.589(2), *c*=14.683(2) Å,  $\alpha$ =103.19(2),  $\beta$ =107.49(2),  $\gamma$ =103.40(2)°, *V*=1528.3 Å<sup>3</sup>, *Z*=2,  $\mu_{\text{Cu}}$ =0.68 mm<sup>-1</sup>;  $d_{\text{calc}}$ =1.292 Mg m<sup>-3</sup>; *T*=113 K; *R*<sub>1</sub>(*F*)=0.084, *gof*=1.12.

Keywords: porphyrin, supramolecular structure, natural product

### P05.05.07

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#### Potassium salts of some ribonucleotides: AMP, IMP, CMP and UMP

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Most of nucleoside 5'-monophosphates have been extensively investigated as regards their structure for long, but potassium salts of them have never been studied by the use of X-ray crystallography. The results of structural investigations on five mono- and dipotassium salts of two purine [ $K_2(AMP)6H_2O$ ,  $K(IMP)3H_2O$ ] and two pyrimidine [ $K_2(CMP)6.25H_2O$ ,  $K(UMP)$ ,  $K_2(UMP)5H_2O$ ] ribonucleotides will be presented. The  $K^+$  salts crystallize with  $Z' = 1-4$  for the respective crystal. In all the mono-/dianions, the orientation of the base with respect to the sugar ring is *anti*, which is typical for nucleotides. The ribose rings in AMP and UMP salts exist in ideal envelope conformations:  ${}^2E$  in two dianions in  $K_2(AMP)6H_2O$  and two dianions in  $K_2(UMP)5H_2O$ , and  ${}^3E$  in the monoanion in  $K(UMP)$ . The sugar rings in the remaining crystals are slightly deformed from the ideal pucker, with the nearest descriptors being:  ${}^3T_2$  for the IMP monoanion,  ${}^2T_1$  for two CMP dianions and  ${}^3T_4$ ,  ${}^2E$  for two remaining CMP dianions. The orientation of the phosphate relative to the ribose ring is the same in all the anions: *gauche-gauche*, which is characteristic of the 5'-nucleotides. The arrangement of the terminal phosphate atoms relative to the ester C5' is *antiperiplanar (ap)* for one of O atoms and *+/-synclinal (+/-sc)* for the others in most of the presented nucleotides. However the phosphate group in the IMP monoanion is disordered and exists in two orientations: typical *ap/sc* and quite uncommon *sp/ac*.

Keywords: nucleotides, ribonucleotides, biological compounds

## P05.05.08

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### Sesquiterpene lactones isolated from a medicinal plant of the Venezuelan Andes

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Sesquiterpene lactones are perhaps the most studied members of the terpenes because a large number of them exhibit pharmaceutical activity. The structures of two sesquiterpene lactones, Tenuline and Gaillardiline, isolated from a medicinal plant of the Venezuelan Andes (*Ambrosia cumanensis*) will be presented in this contribution. Tenuline,  $C_{17}H_{22}O_5 \cdot 2(H_2O)$ , crystallizes in the orthorhombic system, space group  $P2_12_12_1$ , with unit cell parameters  $a=6.9501(2)$ ,  $b=9.5145(3)$ , and  $c=25.7739(8)$  Å,  $V=1704.34(9)$  Å<sup>3</sup>,  $Z=4$ ,  $d_c=1.334$  g cm<sup>-3</sup>. Due to the presence of the water molecules in the structure, there is an extensive hydrogen bonding network which helps stabilize the structure. There are eight inter- and one intra-molecular hydrogen bond. Gaillardiline,  $C_{17}H_{22}O_6$ , crystallizes in  $P2_12_12_1$  with unit cell parameters  $a=9.0749(3)$ ,  $b=11.1830(3)$ ,  $c=15.8804(4)$  Å,  $V=1611.62(8)$  Å<sup>3</sup>,  $Z=4$ ,  $d_c=1.329$  g cm<sup>-3</sup>. Three inter- and four intramolecular hydrogen bonds help to stabilize the structure. Both hydrogen bond networks can be described using graph set theory. They will be discussed in detail. We thank R. Benson (Rigaku-USA), for support with data collection using Cu radiation. This work was funded by FONACIT-Venezuela, through grant LAB-97000821.

Keywords: natural products, sesquiterpene lactones, crystal structure

## P05.06.09

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### Organic two-photon absorbing materials for technological applications and photodynamic therapy

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Two-photon absorbing (TPA) chromophores are used in the technology for three-dimensional microfabrication by lithographic process and in biomedical applications as fluorescent markers for TPA microscopy and as photosensitizers in PTA photodynamic therapy. Advantage of TPA chromophores in comparison with conventional one-photon absorbing chromophores is an opportunity to achieve a very high spatial resolution in three dimensions and so treat tumors without damage to neighboring healthy organs. We studied structures of a large series of substituted benzylidenepiperidones, organic chromophores with general formula Donor- $\pi$ -bridge-Acceptor- $\pi$ -bridge-Donor. Structure – properties relations between geometric and spectroscopic characteristics of materials, important for their PDT applications, have been established. Dark toxicity and toxicity under irradiation have been characterized and discussed in connection with spectroscopic properties of materials.

Keywords: two-photon absorbing materials, photodynamic therapy, spectroscopy

## P05.07.10

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### The structure of water nanopipe, a hollow-type water nanowire, in crystal host

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One-dimensional (1D) water nanostructures are important for better understanding the structure and behavior of water molecules at the atomic level. Previously we reported the first water nanowire in the crystal structure of L-tryptophan pyridoxal-5-phosphate Schiff base, obtained from its aqueous solution [1,2]. Here, we report the second water nanowire, called water nanopipe, in the crystal obtained from another batch of aqueous solution of the Schiff base. This nanopipe is a hollow-wire type nanowire, where the diameter and unit length (equals to the length of c-axis) are 1.649 nm and 0.473 nm. The structure is constructed in such a way that five water molecules form a small cluster unit, and three of the small units are linked around a three-fold axis to form a large cluster unit consisting of 15 water molecules. These large units are then piled up by nine hydrogen bonds parallel to c-direction and form an infinite water nanopipe. In this structure, seven kinds of hydrogen bonds are formed by five independent water molecules: the O--O distances are 0.246, 0.247, 0.272, 0.281, 0.290, 0.293 and 0.295 nm and the hydrogen-bonded O-O-O angles range from 82 to 135°. This nanopipe structure is assumed to be much more stable than the nanowire structure, because the more hydrogen bonds are formed along to the piling-direction in the nanopipe than the nanowire, and the very long needle crystals up to 2.6 cm are obtained for the nanopipe structure. Therefore, it would be considered that the present nanopipe structure provides a structural scaffold between the nanoworld (nm) and the real world (cm) in the