[2]*State University of Campinas / Synchrotron Light National Laboratory (LNLS), BRAZIL.* E-mail: cavalcanti@esrf.fr

The present work shows a structural study on the process of incorporation of a hydrophobic drug, Ellipticine (ELPT), into lipid model membranes for drug targeting purpose. The ELPT is an alkaloid that showed an anti proliferation activity against several types of tumour cells and against the HIV1 virus. In the context of drug targeting, there are several important processes and parameters to be studied. For instance, the drug loading efficiency into the lipid matrix, the order into the lipid system that encapsulates the drug, the lipid-carrier critical size and stability to transport the drug and the releasing mechanisms. We used the zwitterionic lipid dipalmitoylphosphatidylcholine (DPPC) and some other phopholipids with different size of head and tail and/or different net electronic charge both on a Langmuir monolayer and deposited on a solid substrate. First results appointed toward a strong increase in drug loading efficiency into DPPC lipid systems mixed with charged lipids. However, this increasing in loading efficiency was accompanied by a disturbance in the ordering of the bilayers. To characterize these systems we used Grazing Incidence X Ray Diffraction and also specular X Ray Reflectivity technique with synchrotron radiation at Troika II beamline-ESRF, France and also a rotating anode set-up at State University of Campinas, Brazil to monitor structural changes of loaded and non-loaded lipid systems.

Keywords: lipid mesophases, drug interaction, diffraction

P.06.02.2

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Crystal Structure of Chocolate from Powder Diffraction Data

Henk Schenk, René Peschar, Mihaela M. Pop, Dirk J.A. De Ridder, Jan B. van Mechelen, René A.J. Driessen, *Laboratorium voor Kristallografie, HIMS, FNWI, Universiteit van Amsterdam, Valckenierstraat 65, 1018XE Amsterdam, The Netherlands.* E-mail: schenk@science.uva.nl

We solved the crystal structure of the β(V)-form of chocolate and cocoa butter (CB). Chocolate is a made from cocoa and sugar in a complicated process. At room temperature it consists of a well-crystallised continuous CB matrix in which fine cocoa powder and sugar particles are dispersed. In good quality consumer chocolate, the CB is crystallised in one of the two highest melting forms, β(V) or β(VI). Poor storage or improper production may result in fat bloom, the whitish layer on chocolate that is commonly associated with the phase transition from β(V) to β(VI). Any CB consists for 75% of three triacylglycerols, SOS (1,3-distearoyl-2-oleoylglycerol), POS (2-O-1-palmitoyl-3-S-glycerol) and POP. In particular SOS is known to play a major role in the β-crystallisation of CB.

The powder patterns of chocolate, CB and SOS are very similar suggesting a close structural relation. Unit cells were obtained with an indexing routine written specially for this purpose by RP. The cells of β(V)-CB and β_2-SOS are very similar and, surprisingly, so are the indexing figures of merit M_{20}. We solved SOS (63 unique non-H atoms), using the programs FOX and ORGANA. After refinement we used this structure as a starting model to solve and refine the structure of β(V)-CB, employing partial occupancies (57%) for the two end-carbon atoms of both stearin chains. Our results show a considerably different packing as postulated earlier. Moreover the crystal structure gives rise to the explanation of the mechanism of the β(V) to β(VI) phase transition of CB. This is supported by the XRD of β-POS.

Keywords: SDPD, chocolate, structure of cocoa butter

P.06.03.1

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X-ray Analysis as the Important Tool in Controlling Stereoselective Synthesis of Drugs

Jan K. Maurin[a,b], Zbigniew Czarnocki[c], Krzysztof Wieteska[a], Wojciech Wierzchowski[d], *[a]Institute of Atomic Energy, Otwock-Swierk, Poland. [b]National Institute of Public Health, Warsaw, Poland. [c]Department of Chemistry, University of Warsaw, Warsaw, Poland. [d]Institute of Electronic Materials Technology, Warsaw, Poland.* E-mail: maurin@il.waw.pl

Alkaloids form a group of natural heterocyclic compounds exhibiting valuable pharmacological properties: from painkilling and antihypertensive, through antidepressant and antipsychotic to anticancer. Some of them have dangerous narcotic, hallucinogenic and paralysing action. The bioactivity of compound, in great degree, depends on its stereochemical constitution and hence much effort has been made to develop new, bio mimetic methods of stereoselective synthesis of alkaloids and other heterocyclic compounds. Although the results of chemical synthesis are usually widely documented by many physicochemical methods, the final proof for stereochemistry is possible only after crystal structure determination. Here we propose a new method of synthesis using natural amino acids as building blocks which define three-dimensional structures of products. The case of 16 isoquinoline and β-carboline alkaloids and the application of X-ray methods to determine stereochemistry of products serve as an example. Quantum chemical calculations suggest thermodynamically controlled reaction.

Keywords: alkaloids, structure, modelling

P.06.03.2

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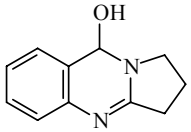
Structures of Cocrystals of Peganole with 6-Brompeganole

Kambarali K. Turgunov, Akmal Tojiboev, Nuritdin Mukarramov, Bahodir Tashkhodjaev, Khusnuddin M. Shakhidoyatov, *S. Yunusov Institute of Chemistry of Plant Substances, Tashkent, Uzbekistan.* E-mail: kk_turgunov@rambler.ru

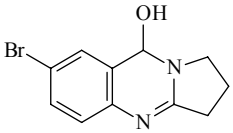
The crystal structures of two polymorphic cocrystals (I, II) of peganole (1) with 6-brompeganole (**2**) have been determined. Both forms are in the monoclinic system with space group of P2(1)/n. Cell parameters of I are a=8.232(4), b=11.768(8), c=10.156(6)Å, β=98.12(4)°, V=974.0(10) Å^3 and II are a=7.995(6) , b=15.501(8), c=8.816(6), β=112.25(5)°, V=1011.2(11) Å^3.

It is interesting to note that asymmetric unit of cell in both case consist of one molecule where this molecule at the same time can be **1** or **2**. Site occupation factors of molecules **1** and **2** are ~0.7 and ~0.3 (in I) and ~0.3 and ~0.7 (in II) respectively.

As well, tests on composition of single crystals by High-Performance Thin-Layer Chromatography (CAMAG, Switzerland) confirms the X-ray results.



1



2

Keywords: cocrystals, polymorphs, small-molecule single crystals

P.06.04.1

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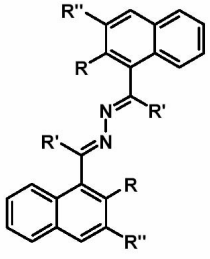
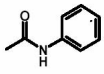
Crystal Structures of Fluorescent Bisazomethine Pigments

Jürgen Brüning, Juste E. Djanhan, Michael Bolte, Martin U. Schmidt, *Institute of Inorganic and Analytical Chemistry, University of Frankfurt, Marie-Curie-Str. 11, D-60439 Frankfurt am Main, Germany*. E-mail: jbruening1@gmx.de

Pigment Yellow 101 (**1**) is a well-known greenish-yellow fluorescent organic pigment. The compounds **1-5** were synthesized and crystallized from DMSO, xylene and CH_3OH/C_2H_5OH mixtures



Compound	1 P.Y.101	2	3	4	5
R	OH	OCH_3	OH	H	OH
R'	H	H	CH_3	H	H
R''	H	H	H	H	
Solid state fluorescence	yes	no	yes	no	yes

resp. The crystal structures of compounds **1-4** were determined by single crystal X-ray structure analysis. Compound **5** was determined from scratch by X-ray powder diffraction.

From the crystal structures it is not evident why the compounds **1**, **3**, and **5** are fluorescent but **2** and **4** are not. Thus extensive quantum mechanical calculations have been made and the reason for the fluorescence quenching of **2** and **4** was finally found [1].

[1] Dreuw A., Wachtveitl J., Brüning J., Schmidt M. U., *in preparation*.

Keywords: organic pigments, crystal structures, fluorescence

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Structure of 19-Hydroxyneohopane

Kenneth J. Haller, *School of Chemistry, Institute of Science, Suranaree University of Technology, Nakhon Ratchasima, Thailand*. E-mail: haller@ccs.sut.ac.th

Several fused multicyclic natural product ring systems, especially those that are saturated or nearly saturated, are poorly represented in the Cambridge Structural Database of crystallographic determinations of organic compounds.

19-hydroxyneohopane, $C_{30}H_{48}O$, is one such compound consisting of a five fused ring system (rings 1 to 4 containing six carbons and ring 5 containing five carbons) with two double bonds trans across the 2-3 ring junction. The compound was obtained from the rhizome of *Davallia solida* Sw and crystallizes in the monoclinic space group, $P2_1$, with two molecules in a cell of dimensions: a = 12.587(3), b = 7.558(3), c = 13.620(3) Å, and β = 102.68(3)° at T = 113(2) K.

Crystal Data: $C_{30}H_{48}O$, MW = 424.68, clear colorless plate crystal, 0.50 x 0.50 x 0.02 mm, monoclinic, $P2_1$, a = 12.587(3), b = 7.558(3), c = 13.620(3) Å, β = 102.68(3)°, V = 1264.10, Z = 2 T = 113(2) K, d_{calc} = 1.116 Mg m^{-3}, μ = 0.48 mm^{-1}, CuK$_\alpha$ radiation, F(000) = 472., sinθ/λ_{max} = 0.545 Å^{-1}, R_{int} = 0.0688, 3876 unique data, 3404 observed $F_o > 4s(F_o)$, R_1 = 0.0737, goof = 1.126.

Keywords: fused ring system, natural product, hopane

P.06.04.3

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X-ray Investigations of Bicyclic α-methylene-δ-valerolactones

Jakub Wojciechowski[a], Henryk Krawczyk[b], Marcin Śliwiński[b], Wojciech M. Wolf[a], [a]*Institute of General and Ecological Chemistry.* [b]*Institute of Organic Chemistry, Technical University of Łódź, Łódź, Poland.* E-mail: wmwolf@p.lodz.pl

The α-methylene-δ-valerolactones moiety is present in various biologically active natural compounds, *e.g.* vernolepin, vernomenin, pentalenolactone *E*, teucriumlactone, artemisitene and crassin. However, work on isolation and synthesis of new α-methylene-δ-valerolactones has not led to a significant number of crystal structure investigations. A search of the CSD (version 5.26) shows that system in which δ-valerolactone ring is condensed with the cyclohexane moiety along the individual C_δ-C_γ single bond is unique among crystal structures examined to date. Investigated compounds represent a novel group of the optically active α-methylene-δ-valerolactones synthesized in a highly stereoselective Michael reaction. Recently we reported crystal structures of two compounds *i.e.* the 3-metylene-2-oxohexahydrochromene-4a-carbozylic acid ethyl ester [1] and the 4a-methyl-3-metylene-octahydro-chromen-2-one [2]. The six following crystal structures will be shown in detail. In all compounds the δ-valerolactone rings adopt a half-chair conformation. The highly polar character of the carbonyl group hinders π electron density delocalization within the O=C-C=C moiety. In the crystal, molecular conformation is stabilized by attractive interactions between the oppositely charged atoms. The mechanism of interactions has been investigated using *NBO* theory at the *MP2*/6-31+*G*(*d*,*p*) level.

[1] Krawczyk H., Śliwiński M., Wolf W.M., Bodalski R., *Synlett*, 2004, 1995.
[2] Krawczyk H., Śliwiński M., Wolf W.M., *Acta Cryst.*, 2004, **C60**, o897.

Keywords: δ-valerolactone, crystal structure, NBO

P.06.04.4

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Conformation of Dioxaphosphopin Ring – Structures of 6-Substituted Benzo and Dibenzo [d,f] [1,3,2] Dioxaphosphopin 6-oxide (I) and Sulphide (II)

Musali Krishnaiah[1], J. Radha Krishna[1], Vedavathi G., Purank[2], [1]*Department of Physics, Sri Venkateswara University, Tirupati-517502.* [2]*Center of Material Characterization, National Chemical Laboratory, Pune – 411 008, INDIA*. E-mail: mkphysvu@yahoo.co.in

The hetro cyclic form of organophosphorous compounds containing phosphoryl unit with suitable substitution exhibits significant physiological activity and they have unique multifaceted applications. Structural studies of organophosphorous compounds have gained considerable importance recently because of their use as insecticides, anti-cancer agents, lubricating oil additives and polymer stabilizers. As part of our continuing investigations on this molecules, we have investigated the structures of 6-substituted benzo and dibenzo [d,f] [1,3,2] dioxaphosphopin 6-oxide and sulphide to know the dependence of substituents on the conformation and geometrical parameters of dioxaphosphin hetro ring. compound (I): C15 H15 O4 P, colourless crystals grown from methanol are Monoclinic $P2_1/c$ with a = 9.441(1) ; b = 15.202(2) and c = 9.746(1)Å ; β = 95.8(2)°; V = 1391.5(3) Å^3; Z = 4; F(000) = 608; ρ_c= 1.385 g cm^{-3}; μ(M_0 Kα) = 2.08 cm^{-1}; R=4.96 and R_w = 0.1157 for 2457 unique reflections. compound (II): C18 H11 O3 Cl2 P S, colourless crystals obtained from 2-proponal, Monoclinic $P2_1/n$ with a = 10.816(6) ; b = 13.615(8) and c = 12.321(7)Å ; β = 99.6(9)°; V = 1789.5(2) Å^3; ρ_c = 1.519 g cm^{-3}; Z = 4; F(000) = 832; λ(Mo Kα) = 0.71073 Å ; R=0.048 and R_w = 0.130 for 2410 unique reflections. based on intensity data collected on Bruker Smart Apex diffractometer using Monochromated M_0Kα radiation, structures were solved by the direct methods and refined by full-matrix least squares methods. The seven membered dioxaphosphin ring exhibits a pseudo- chair form for the former where as a distorted boat like conformation for the later. This is evident for the structural changes with different substituents fused to the hetro ring and also attached to the phosphorous.

Keywords: organophosphorous compounds, conformation of dioxaphosphopin ring, seven membered hetro ring

P.06.04.5

Acta Cryst. (2005). A**61**, C281-C282

***N*-isopropylamidino-substituted Derivatives of Benzo[b]thiophene-2-carboxanilides and Benzo[b]thieno[2,3-*c*]quinolones: DNA Binding by Intercalation**

Gordana Pavlović[a], Ivana Jarak[b], Grace Karminski-Zamola[b], Zora Popović[c], [a]*Faculty of Textile Technology, University of Zagreb, Pierottieva 6, HR-10000, Zagreb, Croatia.* [b]*Department of Organic Chemistry, Faculty of Chemical Engineering and Technology, University of Zagreb, Marulićev trg 20, P. O. Box 177, HR-10000 Zagreb, Croatia.* [c]*Chemistry Department, Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, Ul. kralja Zvonimira 8, HR-10000 Zagreb, Croatia.* E-mail: pavlovic@chem.pmf.hr

Recently, we published syntheses, characterization and antitumor evaluation of series of cyano- and *N*-isopropylamidino-substituted derivatives of benzo[b]thiophene-2-carboxanilides and benzo[b]thieno[2,3-*c*]quinolones [1]. Aromatic surface of such aromatic compounds, usually built of three or more condensed aromatic units, is more than large enough for intercalation with the DNA. On the other hand, organic cations (*i.e.* amidinium cation) are known to bind in the DNA minor groove showing various biological activities, especially anticancer properties. The X-ray crystal structure study of 4'-carbmethoxy *N*-phenyl-3-chlorobenzo[*b*]thiophene-2-carboxamide and *N*-[4'-(*N'*-isopropylamidino)-phenyl]-3-chlorobenzo[*b*]thiophene-2-carboxamide hydrochloride is undertaken in order to compare their sterical properties with some classical intercalators and to give an answer if insertion between basepairs of DNA/RNA is possible.

[1] Jarak I., Kralj M., Šuman L., Pavlović G., Dogan J., Piantanida I., Žinić M.,