

## 06-Crystallography of Organic Compounds

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(II) Crystals of the second compound are colourless from methanol and toluene,  $C_{20}H_{17}O_4PS$ ; monoclinic, Space group  $P2_1/n$ ,  $a=12.842(1)$ ,  $b=20.902(2)$  and  $c=16.548(2)$  Å;  $\beta=103.92(2)^\circ$ ;  $V=4311.45(10)$  Å<sup>3</sup>;  $Z=8$ ,  $\rho=1.406$  and  $\rho=1.415$  g/cm<sup>3</sup>;  $\mu(MoK\alpha)=2.5$  cm<sup>-1</sup>;  $F(000)=1888$  Final  $R=0.039$  &  $R_w=0.046$  for 5452 ( $I \geq 3\sigma(I)$ ) significant reflections.

Structures I & II were solved by direct methods (Shelxs-86) using 1736 and 7562 reflections measured on CAD-4 diffractometer with  $CuK\alpha$  and  $MoK\alpha$  radiations respectively. Both structures were refined by Full-matrix least-squares method using Shelx-76.

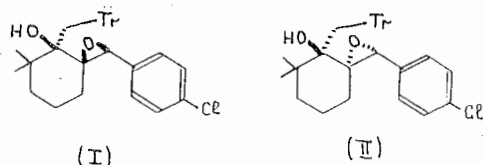
The dioxathiaphosphocin ring shows a boat-chair like conformation in the former where as a boat like conformation in the later and this may be due to different substituents fused to the heterocyclic ring. Details of the geometrical parameters will be discussed.

## PS-06.05.13 STRUCTURE OF STEREOISOMERIC

2-(4-CHLOROPHENYL)-4-HYDROXY-5,5-DIMETHYL-4-(1,2,4-TRIAZOL-1-ILMETHYL)-1-OXASPIRO-[2.5]OCTANES. By Maltnovsky S.T., Krimer M.Z., Tashchik V.P., Dubrovina Ya.A., Komarova O.V., Putsykin Yu.G., Zavodnik V.E.

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We have developed a stereospecific method for obtaining oxirans I and II, to determine why compound I possesses more fungicidal activity than compound II. The X-ray investigation of isomers was undertaken.



Crystals of I and II ( $C_{18}H_{22}ClN_3O_2$ ) are monoclinic,  $P2_1/c$ ,  $Z=4$ . Crystal data: (I)  $a=9.706(3)$ ,  $b=21.254(5)$ ,  $c=9.368(3)$  Å,  $\beta=116.79(2)^\circ$  and (II)  $a=7.477(2)$ ,  $b=15.055(3)$ ,  $c=16.097(4)$  Å,  $\beta=100.42(2)^\circ$ . Final R-factors for I and II are 0.035 and 0.034, respectively. The main conformational difference between the molecules concerns the arrangement of oxiran fragments. The triazol cycle in II is oriented towards the oxiran fragment. In (I) these rings are oriented in opposite directions. The activity of the compounds is thought to depend on the stabilization of the triazol cycle through intermolecular contacts such as those present in isomer I.

## 06.06 - New Structure Reports

PS-06.06.01 CRYSTAL AND MOLECULAR STRUCTURE OF 1 $\alpha$ -METHYL-1,2,3,9,10,10a-HEXAHYDRO-1 $\beta$ ,10 $\alpha\beta$ -11-OXOPROPENO-PHENANTHERENE ( $C_{18}H_{20}O$ ). S.G. Biswas Dept. of Physics, Visva-Bharati University, Santiniketan, W. Bengal, India.

The X-ray investigation of the title compound, a synthesised stereospecific intramolecular rearrangement aiming at synthesising complex diterpenoids, was undertaken to establish its precise molecular architecture.

The intensities of X-ray reflections were measured on a CAD-4 diffractometer using monochromated  $Mo-K\alpha$  radiation with  $\lambda=2.281$  Å. The intensity data was corrected for Lorentz and polarisation factors but no absorption corrections were made. Crystal data. Monoclinic,  $a=9.934(8)$ ,  $b=17.725(11)$ ,  $c=7.607(6)$  Å,  $\beta=97.86(4)^\circ$ ; space group  $P2_1/C$ ,  $Z=4$ ,  $D_c=1.249$  gm cm<sup>-3</sup>,  $D_m=1.22$  gm cm<sup>-3</sup>,  $F(000)=542$ ,  $\rho(Mo-K\alpha)=0.8$  mm<sup>-1</sup>,  $R=0.071$  with 1134 observed reflections. The structure was solved by MULTAN-84 and refined by full-matrix least square method (SHELX-76). The computed bond length and angles are fairly within the range of chemically expected values and the non-bonded contacts were always greater than sum of the Vander Waals radii.

## PS-06.06.02 STRUCTURE OF 2-CYANO-FURFURYLACRYLAMIDE. By R. Pomés\*, J. Duque X-

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$C_8H_6N_2O_2$ , molecular weight = 162, Triclinic,  $PT$ ,  $a=9.876(3)$ ,  $b=10.325(4)$ ,  $c=3.870(8)$  Å,  $\alpha=93.86(2)^\circ$ ,  $\beta=97.61(2)^\circ$ ,  $\gamma=103.52(3)^\circ$ ,  $V=378.3(4)$  Å<sup>3</sup>,  $Z=2$ ,  $D_{calc}=1.42$  g/cm<sup>3</sup>,  $CuK\alpha$  ( $\lambda=1.54178$  Å),  $\mu R=3.08$  cm<sup>-1</sup>.

949 unique reflections measured for two octants,  $3 < 2\theta < 90^\circ$ , with a NICOLET R3M diffractometer four-circle with a graphite monochromator in the incident beam. The data were collected at 25°C, 804 with  $I > 3\sigma(I)$  were used for refinement. range of  $hkl$ :  $-1 < h < 10$ ,  $-10 < k < 10$ ,  $-4 < l < 4$ ,  $R_{int}=0.03$ ,  $\sigma/2\theta$  scan mode, variable scan speed, scan width  $1.0^\circ$ . Intensities of two standard reflections ( $021$ ,  $111$ ), measured at 50 min intervals showed no significant deviations for mean. Only Lorentz-Polarization corrections were applied. Data adjusted to an approximately absolute scale,  $U=0.03$  Å<sup>2</sup>.

The structure was solved by direct methods, using the program MITRIL (Gilmore C.J., Journal of Appl. Cryst., 1984, 17, 42-46), least-squares refinement of all non-H atoms with anisotropic thermal parameters and all of the H atoms were located by a difference Fourier syntheses and refined with isotropic temperature factors. ( $\Delta\rho$ ) from 1.5 to 3 eÅ<sup>-3</sup>. The final R and wR values were 0.064 and 0.067 respectively.

The molecules are held in the crystal by discrete molecules linked by Van der Waals forces.

## PS-06.06.03 STRUCTURE OF N-ISOPROPYL, 2-CYANO-FULFURYLACRYLAMIDE. By J. Duque\*, R. Pomés X-

Ray Laboratory, National Center for Scientific Research, P.O.Box 6990, Havana, Cuba. R. Villena and M. Soriano Institute of Chemistry, UNAM, 04510, Mexico, D.F.

$C_{11}H_{12}N_2O_5$ , molecular weight = 204, Monoclinic, space group  $P2_1/n$ ,  $a=5.083(2)$ ,  $b=15.551(6)$ ,  $c=13.647(5)$  Å,  $\beta=98.94(3)^\circ$ ,  $V=1065.5(7)$  Å<sup>3</sup>,  $Z=4$ ,  $D_{calc}=1.27$  g/cm<sup>3</sup>,  $\mu R=2.26$  cm<sup>-1</sup>. 1421 independent reflections were measured for two octants,  $3 < 2\theta < 90^\circ$ , with a NICOLET R3M diffractometer Four-Circle using  $CuK\alpha$  radiation ( $\lambda=1.54178$  Å) with a graphite monochromator in the incident beam.

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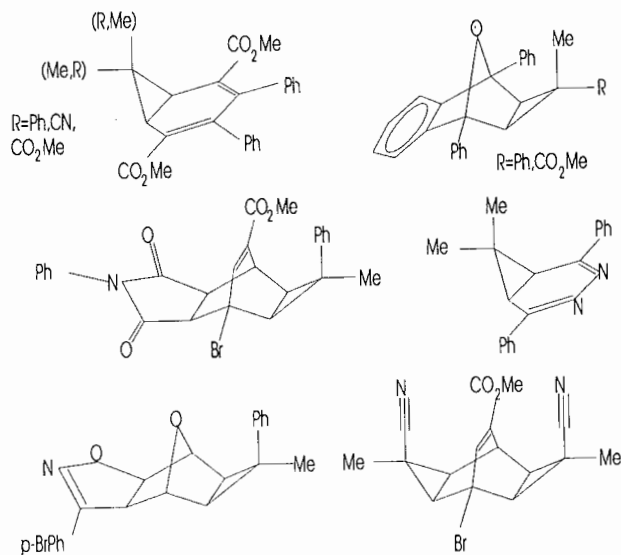
The data were collected at 25 C using the  $\Theta/2\Theta$  scan mode, variable scan speed scan width  $1.0^\circ$ . Two standard reflections (012, 111) monitored every 50 measurements and showed no systematic variation of intensity. Only Lorentz-Polarization corrections were applied, data adjusted to an approximately absolute scale, overall  $U = 0.04\text{\AA}^2$ .

The structure was solved by direct methods, using the program MITRIL (Gilmore C.J., Journal of Appl. Cryst., 1984, 17, 42-46); Least-Squares refinement of all non-H atoms with anisotropic thermal parameters and all of the H atoms were located by a difference syntheses and refined with isotropic temperature factors. ( $\Delta\rho$ ) from  $-0.19$  to  $0.14\text{ e}\text{\AA}^{-3}$ , final  $R = 0.045$ ,  $wR = 0.057$ .

The X-Ray study shows that in this compound there is an intermolecular approach of  $3.4\text{\AA}$  involving non-H atoms:  $N\dots O = 3.018(3)\text{\AA}$ . The molecules are held in the crystal by hydrogen bonds along [100].

**PS-06.06.04** THE STRUCTURES OF THE PRODUCTS OF DIENE CONDENSATIONS WITH PARTICIPATION OF 3,3-DISUBSTITUTED CYCLOPROPENES  
L.A.Aslanov\*, I.G.Bolesov, V.A.Tafeenko & A.V.Yatsenko, Moscow State University

The structures of the products of Diels-Alder reaction between 3-R-3-methylcyclopropenes ( $R = \text{Me, Ph, CN, COOMe}$ ) and dienes were determined by means of X-ray structure analysis (9 structures in all)



The influence of direct substituents' interactions on stabilization of the formed compounds and their spectral properties is discussed.

**PS-06.06.05** CRYSTAL AND MOLECULAR STRUCTURES OF INDIRUBIN MONOOXIME (I) AND INDIRUBIN MONOOXIME ETHYL ETHER (II). By Guo Yunhong, Li Chunmin\*, Wu Shouyu, Zhou Zhonghua, Department of Chemistry, Sichuan University, Sichuan, PRC

In order to search for a new kind of antileukemia drug, a series of derivatives of indirubin have been synthesized. Both indirubin monooxime (I) and indirubin

monooxime ethyl ether (II) have been determined to possess antileukemia activity, the latter being better. In this paper we report their molecular and crystal structures.

On CAD4 diffractometer with  $\text{MoK}\alpha$  radiation and in  $w/2\theta$  mode, in the range of  $2^\circ \leq 2\theta \leq 45^\circ$  for (I) and  $2^\circ \leq 2\theta \leq 50^\circ$  for (II), a total of 4902 and 2937 unique reflections were collected respectively. The 1846 and 1380 reflections having  $I > 3\sigma(I)$  were used in the respective determinations. On a PDP 11/44 computer with SDP Program package, the structures of compounds (I) and (II) were solved by direct methods and Fourier synthesis techniques. The crystallographic data are as follows: crystal (I) (propanone),  $\text{C}_{16}\text{H}_{11}\text{N}_3\text{O}_2 \cdot \text{CH}_3\text{COCH}_3$ ,  $M_r = 335.39$ , monoclinic, space group  $C_2/C$ ,  $a = 2.4697(9)$ ,  $b = 0.7243(2)$ ,  $c = 2.4199(9)\text{ nm}$ ,  $\beta = 129.98(2)^\circ$ ,  $v = 3.3171(2)\text{ nm}^3$ ,  $z = 8$ ,  $D_c = 1.110\text{ g/cm}^3$ ,  $R = 0.0590$ ,  $R_w = 0.0590$ ; crystal (II),  $\text{C}_{18}\text{H}_{15}\text{N}_3\text{O}_2$ ,  $M_r = 305.36$ , monoclinic, space group  $P2_1/c$ ,  $a = 0.9743(1)$ ,  $b = 1.3445(1)$ ,  $c = 1.1807(3)\text{ nm}$ ,  $\beta = 101.06(1)^\circ$ ,  $v = 1.5180(1)\text{ nm}^3$ ,  $Z = 4$ ,  $D_c = 1.336\text{ g/cm}^3$ ,  $R = 0.0393$ ,  $R_w = 0.0418$ . This investigation was undertaken as one part of our ongoing study in the link between the structures and antileukemia activity of derivatives of indirubin.

**PS-06.06.06** CRYSTAL STRUCTURE OF AN INDOLE DERIVATIVE (A DIMER) By S. Eswaramoorthy\*, M.N. Ponnuswamy and K.S. Raju, Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Madras - 600025, INDIA.

The common feature of the naturally occurring antibiotics like proflavine and ethidium is the possession of a planar chromophore with three fused rings. These planar compounds can be inserted in between adjacent base pairs of DNA molecules. The compound, Di[2-(3,4-dimethyl)indole-3-yl methane] is obtained during the synthesis of such planar chromophores. Crystals ( $\text{C}_{35}\text{H}_{34}\text{N}_2\text{O}_4$ ;  $M_r = 558.68$ ) belong to monoclinic space group  $A2/n$ . Unit cell values are  $a = 23.648(2)$ ,  $b = 8.559(3)$ ,  $c = 13.856(4)\text{ \AA}$  and  $\beta = 92.13(1)^\circ$ ;  $V = 2803.6\text{ \AA}^3$ ,  $Z = 8$ ,  $D_x = 1.32\text{ Mg/m}^3$ . Structure is solved by direct methods. One half of the molecule forms the asymmetric unit. The two asymmetric units are connected through a carbon atom which lies at the centre of inversion of the dimer. One monomeric unit is refined by full-matrix least-squares methods. The indole moiety is planar and the phenyl part is perpendicular to the indole moiety. Both of the methoxy groups lie in the plane of the phenyl ring. Four dimeric molecules occupy the unit cell and packed with  $N-H\dots O$  type of hydrogen bonding between the molecules.