

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; $\beta=103.92(2)^\circ$; $V=4311.45(10)$ Å³; $Z=8$, $\rho=1.406$ and $\rho=1.415$ g/cm³; $\mu(MoK\alpha)=2.5$ cm⁻¹; $F(000)=1888$ 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.

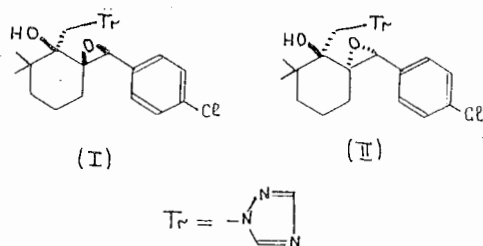
The dioxathiaphosphocin ring shows a boat-chair like conformation in the former where as a boat like conformation in the later and this may be due to different substituents fused to the heterocyclic ring. Details of the geometrical parameters will be discussed.

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 α -METHYL-1,2,3,9,10,10a-HEXAHYDRO-1 β ,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 $\lambda=2.2817$ 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⁻³, $D_m=1.22$ gm cm⁻³, $F(000)=542$, $\rho(Mo-K\alpha)=0.8$ mm⁻¹, $R=0.071$ 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, $P\bar{1}$, $a=9.876(3)$, $b=10.325(4)$, $c=3.870(8)$ Å, $\alpha=93.86(2)^\circ$, $\beta=97.61(2)^\circ$, $\gamma=103.52(3)^\circ$, $V=378.3(4)$ Å³, $Z=2$, $D_{calc}=1.42$ g/cm³, $CuK\alpha$ ($\lambda=1.54178$ Å), $\mu R=3.08$ cm⁻¹.

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°. 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, $U=0.03$ Å².

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⁻³. 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-

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$C_{11}H_{12}N_2O_5$, molecular weight = 204, Monoclinic, space group $P2_1/n$, $a=5.083(2)$, $b=15.551(6)$, $c=13.647(5)$ Å, $\beta=98.94(3)^\circ$, $V=1065.5(7)$ Å³, $Z=4$, $D_{calc}=1.27$ g/cm³, $\mu R=2.26$ cm⁻¹. 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.