09.2-13 THE CRYSTAL STRUCTURE OF THE FUROIN.

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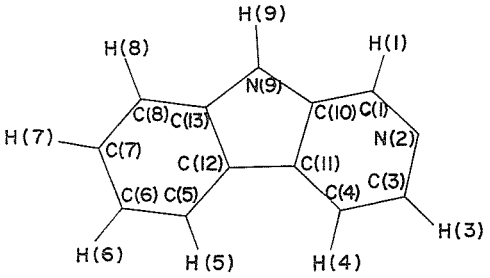
The crystal structure of $C_{10}H_8O_4$ (furoin) has been determined from diffractometer data by direct methods. This work is part of a research program about the structure of some derivatives of furfural.
Crystals are colourless prisms and belong to the monoclinic system, space group $P2_1/c$, with: a = 10.57 Å, b = 16.48 Å, c = 5.56 Å, γ = 110.3° and z = 4.
620 F(hkl) were recorded on a manual diffractometer with MoKα radiation. The structure has been solved using the Röntgen-75 program (Tarnopolskii and Andrianov, 75). The refinement was carried out by full-matrix least-squares methods to R = 3.7 %.
The hydrogen positions are being determined and further refinement is in progress (22 atoms).
In the structure two molecules of $C_{10}H_8O_4$ form an interesting double hydrogen bond with distances: H ..... O = 2.09 Å, H - O = 0.87 Å, O ..... O = 2.868 Å and ∠ O...H - O= 150.6°.



while those obtained from microwave spectra are 1.370 ± .003 Å (Nygaard, L.et al, Molec. struct.(1969) 3, 491). The C(11) - C(12) bond linking the six membered rings has a length of 1.44 Å. The preconception that the lengthening of the C-C contact bond in a phenanthrenoid arrangement of rings is due to steric repulsion between an overcrowded hydrogen pair seems to be of doubtful validity since in the case of norharmane this lengthening is observed even though the molecule is free from steric hindrance. The ring attached to the indole frame is essentially a pyridine configuration. The relatively shortened C-N bond distance is in agreement with that observed in pyridine (Bak, B., Nygaard, L. & Rastrup-Anderson, J. J.Mol.Spectry. (1958) 2, 361). Pyrimidine (Wheatley, P.J., Acta Cryst. (1960) 13, 80) and phenanthridine (Roychowdhury, P., Acta Cryst (1973) B29, 1362) also exhibit similar C-N bond characteristic. This may be due to higher electronegativity of nitrogen.

09.2-14 CRYSTAL STRUCTURE OF NORHARMANE $C_{11}H_8N_2$.

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Norharmane crystallizes from a solution of the compound in a mixture of acetone and alchohol in the orthorhombic spacegroup $P2_12_12_1$ with 4 molecules in the unit cell of dimensions a=9.825, b=14.629, c=5.876 Å. Five very strong reflexions showed the c-axis projection of the structure. The expected NH...N hydrogen bond was utilised to pack the molecule in the unit cell. The derived structure was put through the least squares refinement. The final cycle of full matrix anisotropic refinement of the structure with the inclusion of hydrogen atoms whose temperature factors were only refined isotropically converged at R=.034 for 887 independent reflexions measured on a four circle diffractometer. The standard deviations in bond lengths involving non-hydrogen atoms lie in the range .002 to .003 Å and a mean standard deviation of .024 Å for bond lengths involving hydrogen atoms. The average standard deviation in the bond angles involving non-hydrogen atoms is .1° .and the average e.s.d. for bond angles involving hydrogen is .5°. The average C-C length of the benzenoid ring in the present molecule is 1.394±.002 Å and the mean bond angle is 120°. Two base angles of the pyrrole ring were around 106° while the other three ring angles were approximately 109°. The angles centred at the ring atoms are nearly trigonally symmetric. In the pyrrole ring the two C-N bonds are nearly equal having an average value of 1.371±.002 Å

09.2-15 CRYSTALLOGRAPHIC STUDIES ON SOME CYCLOALKANONE-PHENYLPYRIDINE DERIVATIVES.

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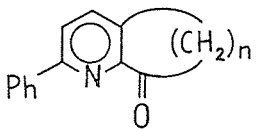
This work was undertaken in order to establish the relation between the number of C atoms in the cycloalkane ring and the planarity of the conjugated system carbonyl-pyridine. It was of interest as well, to determine the conformations of the cycloalkane rings.
Four compounds have been investigated:



n = 2(I), 3(II), 4(III), 5(IV)

In (I) and (II) the carbonyl group is coplanar (in the limits of error) with the pyridine ring while in (III) and (IV) it is at an angle of 32.1° and 60.2° respectively.
The conformations of the cycloalkanone ring are as follows: cyclohexanone - chair, cycloheptanone - twist-boat, cyclooctanone - twist-crown.
The phenyl rings are twisted about the bond by which they are linked to the pyridine ring. The maximum twist-angle is 28.4°.