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4,8-Dimethoxy-1,5-naphthyridine

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(*Received* 16 *September* 1976; *accepted* 2 *April* 1977)

Abstract. $C_{10}H_{10}N_2O_2$, M_r = 190·20, hexagonal, $R\bar{3}$ (No. 148), Z = 9. At −35 °C, a = 24·314 (8), c = 4·005 (1) Å, V = 2050 Å^3, D_x = 1·386 g cm^{-3}. Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 0·77 cm^{-1}. Full-matrix least-squares refinement using 793 reflections [$I > 2\sigma(I)$] collected with ω scans on a Syntex $P2_1$ diffractometer converged at a final R of 0·041. The center of the molecule is coincident with a crystallographic center; the methoxy substituent is in the same plane as the naphthyridine ring with the methyl group directed away from the lone pair of the ring N atom.

Introduction. Crystals were grown by vacuum sublimation and found to be hygroscopic when exposed to the atmosphere at room temperature. The colorless prisms clearly showed the presence of a threefold rotation axis. The possible space groups, $R3$ and $R\bar{3}$, were determined from oscillation and Weissenberg photographs. The crystal selected for data collection had approximate dimensions of 0·32 mm perpendicular to (001) and 0·29 mm perpendicular to the {100} form.

Mounted on a Syntex $P2_1$ diffractometer equipped with a low-temperature apparatus, the crystal was cooled to −35 °C. Mo $K\alpha$ radiation, monochromated with a graphite crystal, was used throughout this study. The unit-cell dimensions were refined from the Bragg angles (as determined by the Syntex centering routine) of 15 reflections.

Intensity data for 1041 unique reflections (4° < 2θ < 55°) were collected using the ω-scan technique. Scans of 1·0° were employed with scan rates which ranged from 1·0 to 5·0° min^{-1} depending on the number of counts measured in a rapid preliminary scan. Background measurements were taken at both ends of the scan with ω displaced 1·0° from the $K\alpha$ peak; the time for each measurement was one-half of the scan time. The intensities of four standard reflections were monitored after every 96 reflections; only statistical variations were noted. The intensities were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods (*MULTAN*); the E statistics suggested, and the structure was successfully solved in, the centrosymmetric space group, $R\bar{3}$. The molecular center coincides with a crystallographic center of symmetry. Refinement of the structure by the full-matrix least-squares method was carried out using only those 793 reflections for which $I > 2\sigma(I)$. The non-hydrogen

Table 1. *Final positional parameters with estimated standard deviations*

(*a*) Atomic coordinates for the non-hydrogen atoms

	x	y	z
N(1)	0·57417 (6)	0·52658 (6)	0·7416 (3)
C(2)	0·58478 (8)	0·58394 (8)	0·8126 (5)
C(3)	0·54744 (8)	0·60975 (8)	0·7149 (4)
C(4)	0·49422 (7)	0·57316 (7)	0·5267 (4)
C(5)	0·47908 (7)	0·51022 (7)	0·4426 (4)
O(6)	0·45379 (5)	0·59170 (5)	0·4141 (3)
C(7)	0·46871 (11)	0·65510 (10)	0·4958 (7)

(*b*) Atomic coordinates for the H atoms

	x	y	z
H(2)	0·6221 (9)	0·6107 (8)	0·947 (5)
H(3)	0·5603 (7)	0·6516 (8)	0·778 (4)
H(7*A*)	0·4328 (10)	0·6603 (9)	0·405 (5)
H(7*B*)	0·4691 (9)	0·6612 (10)	0·772 (6)
H(7*C*)	0·5087 (10)	0·6869 (9)	0·407 (5)

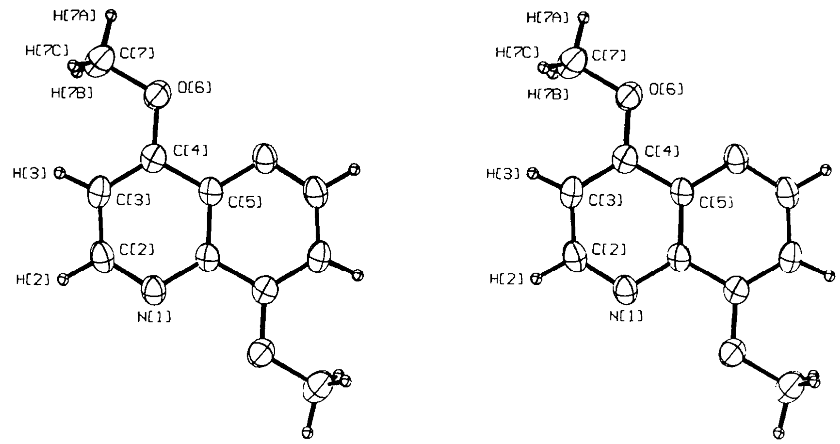

Fig. 1. Atom-numbering scheme for the asymmetric unit. The two halves of the molecule are related by a crystallographic center of symmetry.

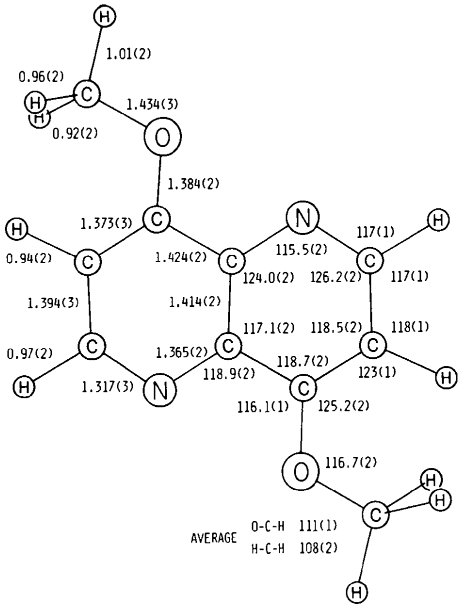

Fig. 2. Bond distances (Å) and angles (°) with estimated standard deviations.

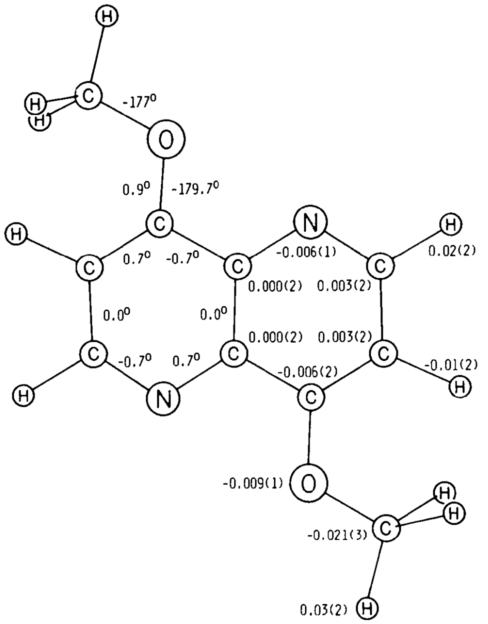

Fig. 3. Left: selected torsion angles (°). Right: deviations (Å) from the mean plane calculated for the ten non-hydrogen atoms of the naphthyridine moiety.

atoms were refined with anisotropic thermal parameters. The H atoms, located from a difference-density Fourier map, were refined with isotropic thermal parameters. The refinement of the 84 variables converged at a conventional R of 0·041 (observation-to-variable ratio = 9·4) and R_w = 0·042. The parameter shifts during the final cycle of refinement were negligible in comparison with the standard deviations of the parameters. The final positional parameters are given in Table 1.[*] The largest peak in the final difference map had a magnitude of 0·18 e Å^{-3} and was located between atoms C(5) and C(5)′. The mathematical and computational details are noted elsewhere (Harlow & Simonsen, 1976).

Discussion. This is the first in a series of substituted naphthyridine compounds to be studied by X-ray diffraction methods in an effort to understand better the chemical and spectral properties of these compounds (Brown, 1976). Fig. 1 is a stereodrawing of the molecule with the atom-numbering scheme. Details of the molecular geometry are given in Figs. 2 and 3. Structural studies of several naphthyridines have been

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[*] Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32613 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

discussed by Clearfield, Sims & Singh (1972), and the present compound falls within their general conclusions.

As expected, the methyl group is oriented away from the lone pair of the N atom (Sax & Desiderato, 1967). The fact that the methyl C atom is nearly situated in the plane of the naphthyridine ring implies that some π bonding occurs between the O atom and the ring. The conformation of the methyl group and the distortion in the exocyclic angles result from the minimization of the steric interactions between the H atoms of the methyl group and atom H(3).

The authors would like to thank the Robert A. Welch Foundation for support of this work (Grant F-0170).

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Acta Cryst. (1977). B**33**, 2664–2667

7-Nitro-1,3-dihydro-5-phenyl-2*H*-1,4-benzodiazepin-2-one (Nitrazepam)

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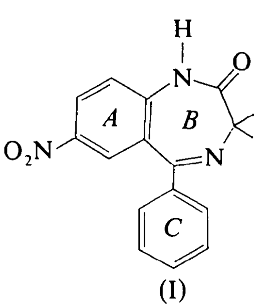
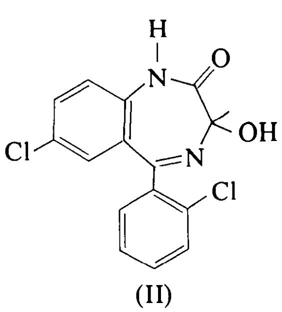
(*Received* 27 *March* 1977; *accepted* 4 *April* 1977)

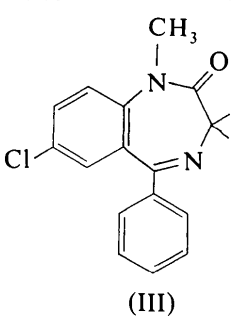
Abstract. Monoclinic, $C2/c$ (No. 15), $a = 27{\cdot}752$ (8), $b = 8{\cdot}251$ (3), $c = 13{\cdot}033$ (4) Å, $\beta = 116{\cdot}93$ (3)°, formula $C_{15}H_{11}N_3O_3$, $Z = 8$, $D_c = 1{\cdot}40$ g cm^{-3}, μ(Cu $K\alpha$) = 8·5 cm^{-1}. The structure consists of pairs of molecules linked by two hydrogen bonds through a crystallographic centre of symmetry. The seven-membered ring is in a boat conformation and a statistical comparison of the geometries of the benzodiazepine group in nitrazepam (I), lorazepam (II) and diazepam (III) has been carried out.



(I)



(II)



(III)

Introduction. The benzodiazepines were introduced as psychotherapeutic agents about fifteen years ago and are still widely used for this purpose. Moreover some of them, markedly diazepam, chlordiazepoxide, oxazepam and nitrazepam, have been shown to have anticonvulsant activity.

In recent years Camerman & Camerman (1970, 1974) have suggested that the biological activity of many anticonvulsant drugs is strictly related to particular steric features of the molecules or, in other words, to the similar arrangement in three-dimensional space of particular atoms or groups of atoms. They based their theory on the similarity of the space-filling characteristic of diazepam with those of other anticonvulsant drugs like diphenylhydantoin, phenobarbital, ethylphenacemide, procyclidine and trihexyphenidyl. On the other hand Sternbach, Sancilio & Blount (1974) have recently determined the crystal structure of two benzodiazepines (4′-fluorodiazepam