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2-Ethyl-6-(*p*-nitrophenyl)dihydro-1,5,2-dioxazine

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Abstract. $C_{11}H_{14}N_2O_4$, $M_r = 238{\cdot}24$, triclinic, $P\bar{1}$, $a = 5{\cdot}383$ (1), $b = 10{\cdot}940$ (2), $c = 12{\cdot}074$ (2) Å, $\alpha = 103{\cdot}09$ (2), $\beta = 100{\cdot}16$ (2), $\gamma = 117{\cdot}59$ (2)°, $V = 580{\cdot}3$ (2) Å^3, $Z = 2$, $D_x = 1{\cdot}37$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 8{\cdot}9$ cm^{-1}, $F(000) = 252$, $T = 293$ K, final $R = 0{\cdot}047$ for 1385 observed reflections. The dioxazine ring is in a chair conformation. The dihedral angle between the phenyl ring and the dioxazine moiety is 142·3 (1)°. The torsion angle C(2′)—C(1′)—C(6)—O(5) is −4·8 (4)°. The ethyl group is in the equatorial position. The torsion angle for C(8)—C(7)—N(2)—O(1) is 78·6 (3)°.

Experimental. The title compound was prepared by thermal rearrangement of the corresponding oxazolidine *N*-oxide. Colorless crystals obtained from a solution of petroleum ether at room temperature; dimensions 0·4 × 0·4 × 0·4 mm. Data collected on a CAD-4 diffractometer, graphite monochromator. Cell parameters measured on the diffractometer using 25 reflections in the 2θ range 20–40°. Range of indices $0 \le h \le 6$, $-10 \le k \le 10$, $-13 \le l \le 13$ ($\theta \le 60°$). Three standards ($\bar{1}1\bar{1}$, $11\bar{2}$, $0\bar{2}4$) measured after every 200 reflections showed a variation of 0·6%. No absorption corrections. Lorentz and polarization corrections. 1709 unique reflections measured. 1385 observed reflections with $I > 3{\cdot}0\sigma(I)$. Direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on *F*) for non-H atoms, isotropic for H atoms. All attempts to refine the coordinates of HC(4) atoms led to meaningless H—C(4) distances. These H atoms were held at a fixed distance, 0·96 Å, with temperature factors of 4·8 Å^2. $\sum w(|F_o| - |F_c|)^2$ minimized. $wR = 0{\cdot}047$, max. $\Delta/\sigma = 0{\cdot}8$. Max. peak height in the final difference Fourier map 0·54 e Å^{-3}, $S = 0{\cdot}51$. Atomic scattering factors from *International Tables for X-ray*

Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	B_{eq}(Å^2)
O(1)	0·0973 (4)	0·1315 (2)	0·3827 (2)	3·64 (5)
O(3)	−0·2476 (6)	−0·4743 (2)	−0·0813 (2)	7·05 (8)
O(4)	−0·5888 (6)	−0·4476 (3)	−0·1744 (2)	6·65 (8)
O(5)	−0·0313 (4)	0·2230 (2)	0·2411 (2)	4·37 (5)
N(1)	−0·3707 (6)	−0·4061 (3)	−0·0902 (2)	4·91 (8)
N(2)	0·2534 (5)	0·2770 (2)	0·4800 (2)	3·68 (6)
C(1′)	−0·0101 (6)	0·0071 (3)	0·1794 (2)	3·40 (7)
C(2′)	−0·2616 (6)	−0·0489 (3)	0·0846 (3)	4·17 (8)
C(3)	0·0711 (7)	0·3401 (3)	0·4530 (3)	4·54 (8)
C(3′)	−0·3831 (7)	−0·1847 (3)	−0·0039 (3)	4·35 (8)
C(4)	0·0784 (7)	0·3609 (3)	0·3341 (3)	4·91 (9)
C(4′)	−0·2464 (6)	−0·2627 (3)	0·0065 (2)	3·87 (8)
C(5′)	0·0024 (7)	−0·2120 (3)	0·1009 (3)	4·49 (8)
C(6)	0·1273 (6)	0·1560 (3)	0·2747 (2)	3·60 (7)
C(6′)	0·1217 (7)	−0·0754 (3)	0·1882 (3)	4·34 (8)
C(7)	0·2383 (7)	0·2400 (3)	0·5891 (3)	4·20 (8)
C(8)	0·4645 (8)	0·2008 (3)	0·6290 (3)	5·91 (9)

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

O(1)—N(2)	1·477 (2)	C(1′)—C(2′)	1·373 (4)
O(1)—C(6)	1·408 (4)	C(1′)—C(6)	1·503 (4)
O(3)—N(1)	1·216 (5)	C(1′)—C(6′)	1·391 (6)
O(4)—N(1)	1·223 (4)	C(2′)—C(3′)	1·381 (4)
O(5)—C(4)	1·434 (3)	C(3)—C(4)	1·507 (5)
O(5)—C(6)	1·421 (4)	C(3′)—C(4′)	1·370 (6)
N(1)—C(4′)	1·482 (4)	C(4′)—C(5′)	1·374 (4)
N(2)—C(3)	1·469 (5)	C(5′)—C(6′)	1·385 (4)
N(2)—C(7)	1·468 (4)	C(7)—C(8)	1·511 (6)

N(2)—O(1)—C(6)	107·7 (2)	N(2)—C(3)—C(4)	107·6 (3)
C(4)—O(5)—C(6)	110·2 (2)	C(2′)—C(3′)—C(4′)	117·9 (3)
O(3)—N(1)—O(4)	124·1 (2)	O(5)—C(4)—C(3)	110·6 (3)
O(3)—N(1)—C(4′)	118·0 (2)	N(1)—C(4′)—C(3′)	118·1 (3)
O(4)—N(1)—C(4′)	118·0 (3)	N(1)—C(4′)—C(5′)	119·1 (3)
O(1)—N(2)—C(3)	104·2 (2)	C(3′)—C(4′)—C(5′)	122·8 (2)
O(1)—N(2)—C(7)	103·6 (2)	C(4′)—C(5′)—C(6′)	118·3 (3)
C(3)—N(2)—C(7)	111·8 (3)	O(1)—C(6)—O(5)	110·3 (3)
C(2′)—C(1′)—C(6)	121·5 (3)	O(1)—C(6)—C(1′)	106·7 (2)
C(2′)—C(1′)—C(6′)	119·6 (2)	O(5)—C(6)—C(1′)	108·4 (2)
C(6)—C(1′)—C(6′)	118·9 (2)	C(1′)—C(6′)—C(5′)	120·2 (3)
C(1′)—C(2′)—C(3′)	121·1 (4)	N(2)—C(7)—C(8)	111·7 (3)

O(1)—N(2)—C(3)—C(4)	−64·0 (3)	C(4)—O(5)—C(6)—O(1)	59·2 (3)
N(2)—C(3)—C(4)—O(5)	58·5 (4)	O(5)—C(6)—O(1)—N(2)	−67·3 (3)
C(3)—C(4)—O(5)—C(6)	−54·3 (4)	C(6)—O(1)—N(2)—C(3)	69·0 (3)

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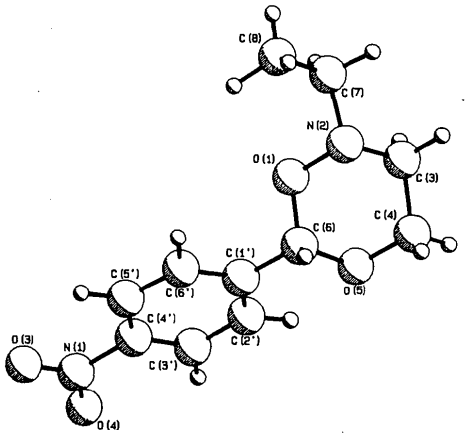

Fig. 1. Numbering of atoms and conformation of the molecule.

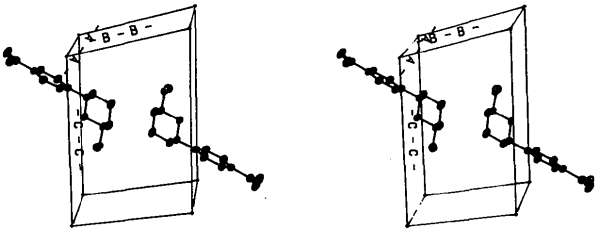

Fig. 2. Stereoscopic view of the unit cell.

Crystallography (1974). Enraf–Nonius *SDP* (Frenz, 1984). Atomic parameters are given in Table 1,* the bond distances, bond angles and relevant torsion angles are presented in Table 2. Atomic numbering is given in Fig. 1 and the packing diagram is shown in Fig 2.

Related literature. A new method for the synthesis of the dihydro-1,5,2-dioxazine ring system bearing simple alkyl groups at the N atom has been recently developed in our laboratories. With the exception of the present paper, there is no information available concerning the conformations of these compounds. In contrast, the conformations of certain *N*-alkyl-1,3,5-dioxazines (Baker, Ferguson, Katritzky, Patel & Rahimi-Rastgoo, 1978) and *N*-alkyl-1,4,2-dioxazines (Jones, Katritzky, Martin & Saba, 1974) have been studied in solution.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-square-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51914 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of α-*N*-Nitroso-1′,2′,3′,4′,10,11-hexahydrocinchonidine Hydrochloride

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Abstract. $C_{19}H_{28}N_3O_2^+.Cl^-$, $M_r = 365{\cdot}9$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}078$ (1), $b = 13{\cdot}893$ (3), $c = 16{\cdot}993$ (2) Å, $V = 1907{\cdot}1$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}27$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ(Cu $K\alpha$) = 1·805 mm^{-1}, $F(000) = 784$, room temperature, $R = 0{\cdot}068$ for 1064 observed reflexions. The protonation occurs at the quinuclidine N atom. The hydrogenated ring has a distorted-sofa conformation. The configuration at the new asymmetric center, C(4′), is *R*. The *N*-nitroso group has the

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