

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

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

* 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.

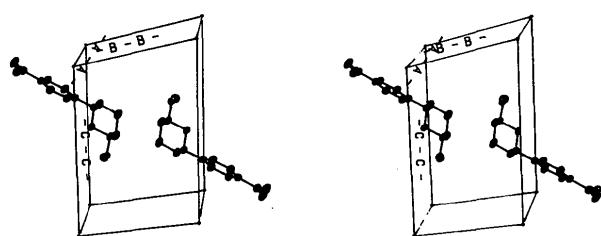


Fig. 2. Stereoscopic view of the unit cell.

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.

References

- BAKER, V. J., FERGUSON, I. J., KATRITZKY, A. R., PATEL, R. & RAHIMI-RASTGOO, S. (1978). *J. Chem. Soc. Perkin Trans. 2*, pp. 377–381.
 FRENZ, B. A. (1984). *Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 JONES, R. A. Y., KATRITZKY, A. R., MARTIN, A. R., & SABA, S. (1974). *J. Chem. Soc. Perkin Trans. 2*, pp. 1561–1563.
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray diffraction Data*. Univs. of York, England, and Louvain, Belgium

Acta Cryst. (1989). **C45**, 1656–1658

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

BY M. GDANIEC AND Z. KOSTURKIEWICZ*

Department of Crystallography, Faculty of Chemistry, A. Mickiewicz University, 60-780 Poznań, Poland

AND B. GOLANKIEWICZ

Institute of Bioorganic Chemistry, Polish Academy of Science, 61-704 Poznań, Poland

(Received 19 January 1989; accepted 18 May 1989)

Abstract. $C_{19}H_{28}N_3O_2^+ \cdot Cl^-$, $M_r = 365.9$, orthorhombic, $P2_12_12_1$, $a = 8.078(1)$, $b = 13.893(3)$, $c = 16.993(2)$ Å, $V = 1907.1(5)$ Å 3 , $Z = 4$, $D_x = 1.27$ Mg m $^{-3}$, $\lambda(Cu K\alpha) = 1.54178$ Å, $\mu(Cu K\alpha) =$

1.805 mm $^{-1}$, $F(000) = 784$, room temperature, $R = 0.068$ 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

* To whom correspondence should be addressed.

Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (\AA^2)

	x	y	z	U_{eq}
Cl	0.6385 (3)	0.2960 (2)	-0.0717 (2)	0.078 (1)
N(1P)	0.632 (1)	0.3757 (6)	0.2100 (5)	0.072 (4)
C(2P)	0.485 (1)	0.3183 (7)	0.1874 (7)	0.073 (4)
C(3P)	0.493 (1)	0.2215 (7)	0.2267 (5)	0.068 (4)
C(4P)	0.661 (1)	0.1720 (6)	0.2114 (5)	0.058 (4)
C(5P)	0.939 (1)	0.1946 (9)	0.2755 (5)	0.072 (4)
C(6P)	1.063 (2)	0.254 (1)	0.3037 (7)	0.091 (6)
C(7P)	1.045 (2)	0.351 (1)	0.3011 (8)	0.101 (7)
C(8P)	0.902 (1)	0.3934 (9)	0.2738 (6)	0.092 (5)
C(9P)	0.781 (1)	0.3347 (8)	0.2430 (6)	0.063 (4)
C(10P)	0.793 (1)	0.2341 (7)	0.2440 (6)	0.060 (4)
O(1)	0.8427 (6)	0.0857 (4)	0.1245 (3)	0.070 (2)
O(2)	0.504 (1)	0.5017 (6)	0.1673 (4)	0.106 (4)
N(2)	0.5767 (8)	0.0978 (5)	-0.0071 (4)	0.055 (3)
N(3)	0.636 (1)	0.4712 (7)	0.1965 (6)	0.095 (5)
C(2)	0.422 (1)	0.0615 (6)	-0.0436 (5)	0.062 (4)
C(3)	0.400 (1)	-0.0470 (6)	-0.0224 (5)	0.063 (4)
C(4)	0.534 (1)	-0.0751 (7)	0.0347 (6)	0.066 (4)
C(5)	0.703 (1)	-0.0631 (6)	-0.0050 (6)	0.073 (4)
C(6)	0.721 (1)	0.0394 (6)	-0.0361 (6)	0.070 (4)
C(7)	0.530 (1)	-0.0083 (6)	0.1078 (5)	0.060 (4)
C(8)	0.557 (1)	0.0972 (6)	0.0807 (5)	0.049 (3)
C(9)	0.399 (1)	0.1483 (6)	0.1243 (5)	0.056 (3)
C(10)	0.223 (1)	-0.0666 (6)	0.0040 (6)	0.074 (4)
C(11)	0.187 (1)	-0.1738 (6)	0.0091 (7)	0.104 (5)

following geometry: N—N = 1.35 (1), N—O = 1.25 (1) \AA , N—N—O = 112.4 (9) $^\circ$. Hydrocinchoninium cations and Cl anions form a hydrogen-bonded chain: the hydroxyl group of one cation is joined to a Cl anion which is also bonded to the protonated N atom of another cation related to the previous cation by a 2_1 axis.

Experimental. Crystals from ethanol, space group from Weissenberg photographs, crystal $0.1 \times 0.1 \times 0.4$ mm, Syntex P2₁ diffractometer, cell parameters from least-squares treatment of setting angles of 15 reflexions with $14 \leq 2\theta \leq 25^\circ$. 1458 reflexions measured in range $h 0 \rightarrow 8$, $k 0 \rightarrow 14$, $l 0 \rightarrow 18$. No significant intensity variation for two standard reflexions measured every 1 h. Maximum value of $\sin\theta/\lambda = 0.55 \text{ \AA}^{-1}$. Peak profile analysis according to Lehmann & Larsen (1974); no absorption correction. 1064 reflexions with $I \geq 2\sigma(I)$. Structure solved by direct methods using MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinement on F , $w = 5.98/\sigma^2(F)$. H atoms from molecular geometry, included as fixed isotropic contribution to F_c , anisotropic thermal parameters for non-H atoms, empirical isotropic extinction parameter x refined to $63 (8) \times 10^{-8}$, $R = 0.068$, $wR = 0.058$, $S = 3.748$, $(\Delta/\sigma) < 0.1$, $(\Delta\rho)_{\text{max}} = 0.24$, $(\Delta\rho)_{\text{min}} = -0.27 \text{ e \AA}^{-3}$. Computer programs: MULTAN80 (Main *et al.*, 1980), SHELX76 (Sheldrick, 1976) and local programs (Jaskólski, 1982). Molecular illustrations drawn using ORTEP (Johnson, 1976) and PLUTO (Motherwell & Clegg, 1978). Atomic scattering fac-

Table 2. Bond lengths (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

N(1P)—C(2P)	1.48 (1)	C(9)—O(1)	1.45 (1)
C(2P)—C(3P)	1.50 (1)	C(9)—C(8)	1.54 (1)
C(3P)—C(4P)	1.54 (1)	C(8)—C(7)	1.55 (1)
C(4P)—C(10P)	1.48 (1)	C(7)—C(4)	1.55 (1)
C(10P)—C(9P)	1.40 (2)	C(4)—C(5)	1.53 (1)
C(9P)—N(1P)	1.44 (1)	C(5)—C(6)	1.53 (1)
N(1P)—N(3)	1.35 (1)	C(6)—N(2)	1.50 (1)
N(3)—O(2)	1.25 (1)	N(2)—C(2)	1.49 (1)
C(9P)—C(8P)	1.38 (2)	C(2)—C(3)	1.56 (2)
C(8P)—C(7P)	1.38 (2)	C(3)—C(4)	1.51 (1)
C(7P)—C(6P)	1.36 (2)	C(3)—C(10)	1.52 (1)
C(6P)—C(5P)	1.38 (2)	C(10)—C(11)	1.52 (1)
C(5P)—C(10P)	1.41 (1)	N(2)—C(8)	1.50 (1)
C(4P)—C(9)	1.55 (1)		
O(2)—N(3)—N(1P)	112.4 (9)	C(4P)—C(9)—C(8)	114.2 (6)
N(3)—N(1P)—C(2P)	120.4 (8)	C(9)—C(8)—N(2)	113.5 (6)
N(3)—N(1P)—C(9P)	115.8 (8)	N(2)—C(8)—C(7)	108.4 (6)
N(1P)—C(2P)—C(3P)	109.5 (7)	C(9)—C(8)—C(7)	113.5 (6)
C(2P)—C(3P)—C(4P)	111.1 (8)	C(8)—C(7)—C(4)	108.9 (6)
C(3P)—C(4P)—C(10P)	108.1 (7)	C(7)—C(4)—C(5)	107.8 (7)
C(4P)—C(10P)—C(9P)	121.8 (8)	C(4)—C(5)—C(6)	110.0 (7)
C(10P)—C(9P)—N(1P)	117.1 (8)	C(5)—C(6)—N(2)	108.3 (6)
C(9P)—N(1P)—C(2P)	123.8 (7)	C(6)—N(2)—C(8)	113.9 (6)
N(1P)—C(9P)—C(8P)	120.4 (8)	C(8)—N(2)—C(2)	109.0 (6)
C(9P)—C(8P)—C(7P)	118.1 (10)	N(2)—C(2)—C(3)	109.0 (6)
C(8P)—C(7P)—C(6P)	121.6 (11)	C(2)—C(3)—C(4)	108.6 (7)
C(7P)—C(6P)—C(5P)	120.3 (11)	C(3)—C(4)—C(7)	110.2 (7)
C(6P)—C(5P)—C(10P)	120.6 (9)	C(3)—C(4)—C(5)	109.1 (7)
C(5P)—C(10P)—C(9P)	116.7 (8)	C(4)—C(3)—C(10)	116.0 (7)
C(5P)—C(10P)—C(4P)	121.4 (8)	C(2)—C(3)—C(10)	110.2 (7)
C(10P)—C(4P)—C(9)	109.7 (7)	C(3)—C(10)—C(11)	112.0 (7)
C(3P)—C(4P)—C(9)	115.7 (6)	C(2)—N(2)—C(6)	109.5 (6)
C(4P)—C(9)—O(1)	106.6 (6)	N(1)—C(9P)—C(10P)	117.1 (8)
O(1)—C(9)—C(8)	108.7 (6)		

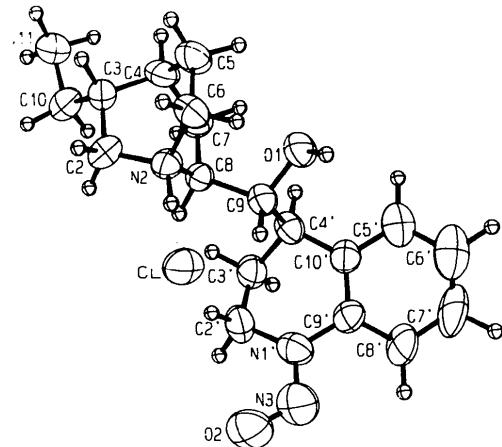


Fig. 1. ORTEP drawing of the cation and anion (50% probability level).

tors from International Tables for X-ray Crystallography (1974). Atomic parameters are given in Table 1, bond lengths and angles in Table 2.* An ORTEP

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51929 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

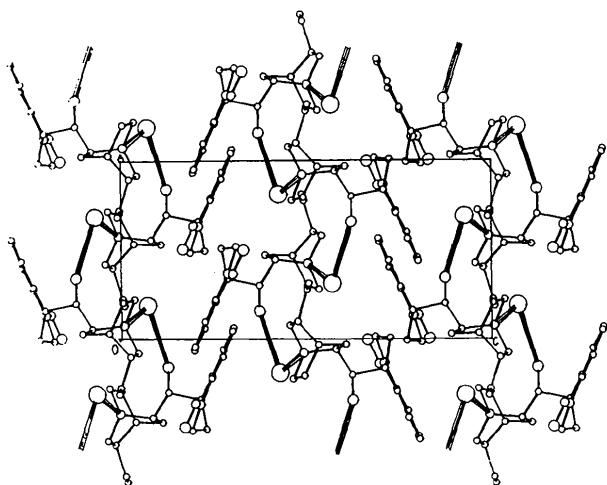


Fig. 2. A view down b showing the crystal packing of the title compound. Cl ions are marked with large circles and hydrogen bonds with thick lines. H atoms are omitted for clarity.

drawing showing the conformation of the cation and its atom-labelling system is presented in Fig. 1. A view of unit cell down the b axis is shown in Fig. 2.

Related literature. Crystal structures containing hexahydrocinchonine moieties have been reported by Golankiewicz, Gdaniec, Jaskólski & Kosturkiewicz (1981) and Gdaniec, Kosturkiewicz & Golankiewicz

(1987). The geometry of Cinchona alkaloids is discussed by Oleksyn (1987).

The work was supported by the project RP.II.13.2.13.(MEN).

References

- GDANIEC, M., KOSTURKIEWICZ, Z. & GOLANKIEWICZ, B. (1987). *Acta Cryst. C43*, 1951–1954.
 GOŁANKIEWICZ, B., GDANIEC, M., JASKÓLSKI, M. & KOSTURKIEWICZ, Z. (1981). *Pol. J. Chem. 55*, 2279–2287.
International Tables for X-ray Crystallography. (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 JASKÓLSKI, M. (1982). *Fourth Symposium on Organic Crystal Chemistry*, Poznań, September 1982, edited by Z. KAŁUSKI, pp. 70–71. A. Mickiewicz Univ., Poland.
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 LEHMANN, M. S. & LARSEN, F.K. (1974). *Acta Cryst. A30*, 580–584.
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
 OLEKSYN, B. (1987). *Habilit. Dissertation*, Jagiellonian Univ., Poland.
 SHELDICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1989). **C45**, 1658–1660

Stereochemical Studies of Oligomers. XXV.* *N-(o-Tolyl)phthalimide*

BY GABRIELE BOCELLI AND ANDREA CANTONI

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Viale delle Scienze, 43100 Parma, Italy

(Received 10 May 1989; accepted 24 May 1989)

Abstract. $C_{15}H_{11}NO_2$, $M_r = 237.3$, monoclinic, $P2_1/c$, $a = 10.010$ (2), $b = 16.250$ (2), $c = 7.658$ (2) Å, $\beta = 104.73$ (3)°, $V = 1204.7$ Å³, $Z = 4$, $D_x = 1.31$ g cm⁻³, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 6.71$ cm⁻¹, $F(000) = 496$, room temperature, $R = 0.070$ for 888 observed reflections. Each individual ring is planar but the two rings of the phthalimide group are not perfectly coplanar, the dihedral angle between the mean ring planes being 1.2 (3)°. The O1 and O2 atoms deviate from the mean pyrrole ring by 0.019 (7) and 0.011 (6) Å. The geometrical values for the methyl-substituted ring are normal, the mean C—C distance and C—C—C internal angle being 1.382 (7) Å and

120.0 (9)° respectively. This ring forms a dihedral angle of 70.7 (2)° with the mean phthalimide moiety. The molecules are joined together in the crystals by van der Waals forces only.

Experimental. Colourless prismatic crystal, recrystallized by slow evaporation from methanol, of 0.4 × 0.5 × 0.8 mm used for data collection on a Siemens AED single-crystal diffractometer equipped with an IBM PS2/30 personal computer, in the $3 \leq \theta \leq 70$ ° range, using the nickel-filtered Cu $K\alpha$ radiation through a modified version (Belletti, Cantoni & Pasquinelli, 1988) of the Lehmann & Larsen (1974) procedure. 28 reflections employed to obtain accurate cell dimensions by least-squares fit. The range in

* Part XXIV: Bocelli, Rizzoli & Ori (1989).