

Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (\AA^2)
$$U_{\text{eq}} = (U_{11} \times U_{22} \times U_{33})^{1/3}.$$

	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)°. 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 $P2_1$ 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_o , $w = 5.98/\sigma^2(F_o)$. H atoms from molecular geometry, included as fixed isotropic contribution to F_o , 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)		

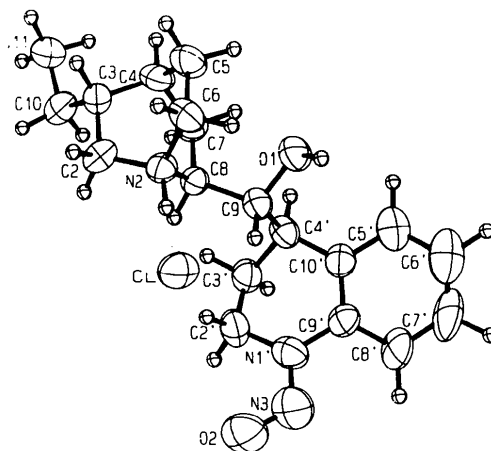


Fig. 1. ORTEP drawing of the cation and anion (50% probability level).

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.

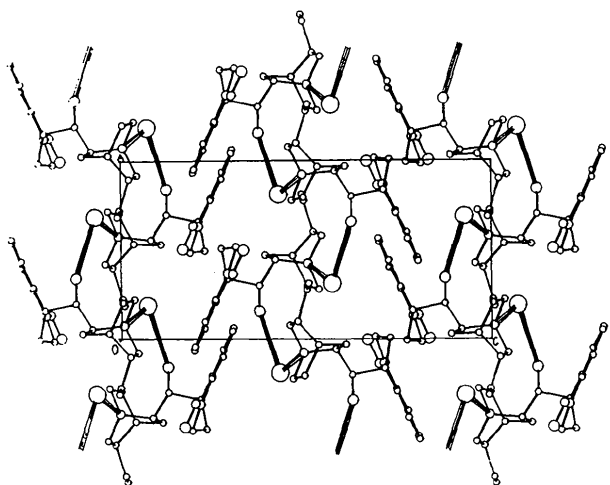


Fig. 2. A view down *b* showing the crystal packing of the title compound. Cl ions are marked with large circles and hydrogen bonds with thick lines. H atoms are omitted for clarity.

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).

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

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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, $\lambda(\text{Cu } K\alpha) = 1.5418$ 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 $3 \leq \theta \leq 70^\circ$ range, using the nickel-filtered Cu $K\alpha$ radiation through a modified version (Belletti, Cantoni & Pasquellini, 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).