(Ul-Haque & Caughlan, 1969); structure of plenolin (11,13-dihydrohelenalin) as *p*-iodobenzoate (Lee, Ibuka, McPhail, Onan, Geissman & Waddell, 1974); structure of autumnolide (Von Dreele, Petit, Cragg & Ode, 1975); structure of mexicanin-E as monobromide (Ul-Haque & Caughlan, 1967); various plant sources (Fischer, Olivier & Fischer, 1979); conformational asymmetry parameters (Duax & Norton, 1975).

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Acta Cryst. (1987). C**43**, 360–361

Structure of a Local Anaesthetic: Dyclonine Hydrochloride*

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(*Received* 26 *March* 1986; *accepted* 2 *September* 1986)

Abstract. 3-Piperidino-4′-butoxypropiophenone hydrochloride, $C_{18}H_{28}NO_2^+.Cl^-$, $M_r = 325{\cdot}9$, monoclinic, $P2_1/a$, $a = 9{\cdot}1084$ (3), $b = 14{\cdot}3242$ (7), $c = 14{\cdot}8573$ (5) Å, $\beta = 106{\cdot}523$ (3)°, $V = 1858{\cdot}39$ (4) Å^3, $T = 295$ K, $Z = 4$, $D_x = 1{\cdot}165$ (2), $D_m = 1{\cdot}16$ (3) Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 18{\cdot}8$ cm^{-1}, $F(000) = 704$, final $R = 0{\cdot}0346$, $wR = 0{\cdot}0327$ for 2720 significant reflections ($I_{net} \geq 2{\cdot}5\sigma$). The piperidine ring is in a distortion-free chair conformation. The phenyl ring makes an angle of 29·9 (2)° with the best plane through the piperidine ring. The C—C—C—C—N group linking the benzene and the piperidine ring is in *trans–trans* conformation. The N atom of the piperidine ring is hydrogen-bonded to the Cl atom, N···Cl 3·062 (2) Å, N—H···Cl 170·2 (2)°.

Experimental. The title compound is a local anaesthetic. The X-ray analysis of the compound was undertaken as a part of a project on the crystal structure and conformation of local anaesthetics. The sample was obtained from Sigma Chemical Co. Colourless needle-shaped crystals, 0·3 × 0·25 × 0·15 mm, from a mixture of methanol + ether. D_m by flotation. Preliminary data from oscillation and Weissenberg photographs, cell constants refined using 70 reflections, $110 \leq 2\theta \leq 120°$ and Cu $K\alpha$ ($\lambda = 1{\cdot}54051$ Å), $\theta/2\theta$ scan with line profile analysis (Grant & Gabe, 1978), Picker four-circle automatic diffractometer, graphite-monochromated Cu $K\alpha$ radiation, no absorption or extinction corrections, data corrected for polarization and Lorentz effects (Le Page, Gabe & Calvert, 1979), 3309 reflections measured, $2\theta_{max} = 120°$. $-10 \leq h \leq 9$, $0 \leq k \leq 16$, $0 \leq l \leq 16$, 2896 independent reflections, 2760 observed with $I \geq 2{\cdot}5\sigma(I)$. Agreement between equivalent reflections 0·7% on intensity. Three standard reflections monitored, variation <2%. Structure solution by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Initial $R = 0{\cdot}35$, after few cycles of refinement $R = 0{\cdot}15$. Hydrogen atoms from $\Delta\rho$ synthesis, full-matrix least-squares refinement on $|F|$, non-hydrogen atoms aniso-

* DCB Contribution No. 690.

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0108-2701/87/020360-02$01.50

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms with e.s.d.'s in parentheses*

$B_{eq} = \frac{8}{3}\pi^2(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)$.

	x	y	z	B_{eq}(Å^2)
Cl	0·27803 (5)	0·16383 (3)	0·98317 (3)	4·82 (3)
N	0·25964 (16)	0·37564 (10)	0·95431 (10)	3·46 (7)
C(1)	0·10667 (23)	0·41024 (15)	0·89737 (14)	4·28 (10)
C(2)	0·0705 (3)	0·37536 (18)	0·79778 (15)	5·34 (12)
C(3)	0·1919 (3)	0·40158 (18)	0·75160 (17)	6·20 (15)
C(4)	0·3460 (3)	0·36871 (20)	0·81049 (18)	6·13 (14)
C(5)	0·3809 (3)	0·40355 (18)	0·90973 (17)	5·19 (12)
C(6)	0·30204 (24)	0·40725 (14)	1·05446 (13)	3·96 (10)
C(7)	0·19417 (25)	0·37226 (15)	1·10698 (13)	4·09 (9)
C(8)	0·24743 (22)	0·39999 (12)	1·20895 (12)	4·14 (9)
O(1)	0·37545 (16)	0·43178 (10)	1·24194 (9)	6·14 (8)
C(9)	0·14039 (21)	0·38918 (11)	1·26674 (12)	3·74 (9)
C(10)	0·17973 (24)	0·42581 (13)	1·35776 (13)	4·28 (9)
C(11)	0·08248 (23)	0·42098 (13)	1·41233 (14)	4·45 (9)
C(12)	−0·05952 (22)	0·37819 (12)	1·37834 (12)	4·04 (9)
C(13)	−0·09957 (24)	0·33901 (13)	1·28945 (13)	4·35 (9)
C(14)	−0·00069 (23)	0·34555 (12)	1·23459 (13)	4·12 (9)
O(2)	−0·15110 (15)	0·38039 (9)	1·43625 (8)	5·06 (7)
C(15)	−0·3034 (3)	0·34212 (18)	1·40183 (16)	5·28 (11)
C(16)	−0·3881 (3)	0·36387 (18)	1·47297 (17)	5·74 (12)
C(17)	−0·3202 (3)	0·32281 (20)	1·56791 (19)	6·43 (15)
C(18)	−0·4175 (5)	0·3416 (3)	1·6347 (3)	8·19 (21)

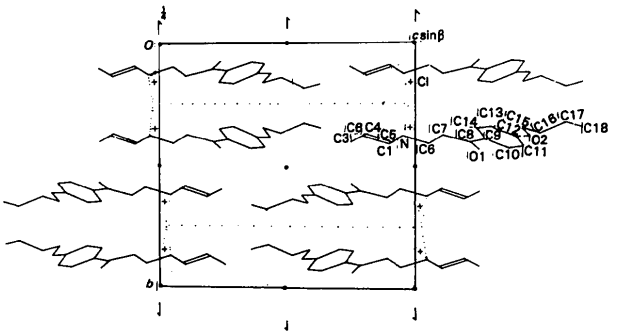

Fig. 1. Packing diagram of the molecules down the *a* axis.

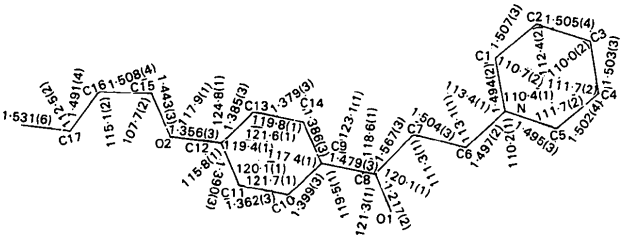

Fig. 2. Atom numbering scheme. Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses.

tropic and hydrogen atoms isotropic refinement in the last few cycles. Individual weighting scheme where $w = 1/\sigma^2(F_o)$ based on counting statistics. Final $R(F)$ for all reflections 0·038, $wR = 0·036$; for observed reflections $R = 0·035$, $wR = 0·033$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974), $(\Delta/\sigma)_{max} = 0·3$, $(\Delta/\sigma)_{mean} = 0·1$; $S = 6·03$, final $\Delta\rho$ map had all peaks less than 0·3 e Å^{-3}. All calculations using the NRC-PDP-8e system of programs (Larson & Gabe, 1978) adapted for VAX computer. Positional parameters and B parameters are given in Table 1. The packing of the molecules down the *a* axis is shown in Fig. 1 and the bond lengths and bond angles are in Fig. 2.*

Related literature. The average C—N single bond can be compared with the 1·494 (7) Å observed in 1,2-dimethyl-4-hydroxy-4-phenylpiperidine hydrochloride (Codding & James, 1974) but is larger than those found in piperidine alcohols (De Camp & Ahmed, 1972). The intramolecular N···O(1) distance of 4·178 (3) Å is less than the suggested value (5·5 Å) between the positively charged amino nitrogen and the negatively charged carbonyl oxygen for local anaesthetic activity (Korolkovas, 1970). The torsion angles of the alkyl chain linking the piperidine ring and the phenyl group are comparable to the values observed in other local anaesthetics (Sinha, Subramanian, Vasantha Pattabhi & Trotter, 1984; Kashino, Ikeda & Haisa, 1982; Hanson & Röhrl, 1972). The phenyl ring is significantly non-planar ($\chi^2 = 126·3$).

The authors thank the University Grants Commission of India for financial assistance.

* 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 43365 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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