Fig. 2. Packing of the molecules in the unit cell viewed down **b**.

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Structure of 6-(2,6-Dichlorophenyl)-2,3,6,7-tetrahydro-5H-pyrrolo[1,2-a]imidazole Hydrochloride Monohydrate (ICI-101187), $C_{12}H_{13}Cl_2N_2^+.Cl^-.H_2O$

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Abstract. $M_r = 309.6$, triclinic, $P\bar{1}$, $a = 8.320$ (1), $b = 8.326$ (1), $c = 11.166$ (2) Å, $\alpha = 102.96$ (1), $\beta = 104.26$ (1), $\gamma = 86.32$ (1)°, $V = 730.6$ (4) Å^3, $Z = 2$, $D_x = 1.41$ Mg m^{-3}, Cu $K\bar{\alpha}$, $\bar{\lambda} = 1.54178$ Å, $\mu = 11.17$ mm^{-1}, $F(000) = 320$, room temperature, final $R = 0.065$ for 1715 reflections. The title compound is an α_2-adrenoceptor agonist and antihypertensive agent. The two moieties of the molecule are almost perpendicular. N–H···Cl hydrogen bonds contribute to the crystalline cohesion.

Introduction. The class of 6-aryl-2,3,6,7-tetrahydro-5H-pyrrolo[1,2-a]imidazoles, including recently synthesized ICI-101187, were shown to be α-agonists. ICI-101187 is as potent as clonidine in lowering blood pressure while it is only one-tenth as active as a sedative (Clough, Hatton, Pettinger, Samuels Gillian & Shaw, 1978).

This work has been undertaken in order to compare the conformation of ICI-101187 with those of clonidine (Byre, Mostad & Rømming, 1976; Carpy, Hickel & Léger, 1979) and related analogues (Carpy, Léger, Leclerc, Decker, Rouot & Wermuth, 1982, and references therein).

Experimental. White elongated prism (from 2-propanol), 0.25 × 0.20 × 0.10 mm, D_m not measured, Enraf–Nonius CAD-4 diffractometer, 25 reflections used for measuring lattice parameters ($6 < \theta < 30°$), $2760 \pm h \pm kl$ independent reflections with $\theta < 70°$, 1715 with $I \geq 3\sigma(I)$, Lp correction, absorption ignored; two check reflections ($2\bar{1}3, 0\bar{4}0$) measured every 5400s: no variation. Direct methods (*MULTAN*, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), anisotropic diagonal-matrix refinement on F; weighting scheme: $w = 1$ if $|F_o| < P, P = (F_o^2$ max./10)$^{1/2}$, $w = (P/F_o)^2$ if $|F_o| > P$; H_2O and H from ΔF synthesis, H isotropic, final $R = 0.065$, $wR = 0.045$, $S = 0.980$; final maximum shift to error 0.295, max. and min. heights in final difference Fourier map ± 0.5 e Å^{-3}; f_i of

non-hydrogen atoms (C,O,N,Cl and Cl⁻) from *International Tables for X-ray Crystallography* (1974), f_i of H atoms from Stewart, Davidson & Simpson (1965); IRIS 80, CII, computer of the 'Centre Interuniversitaire de Calcul' (Talence).

Discussion. Atomic parameters for the non-H atoms are given in Table 1, and bond distances and angles in Table 2.* Fig. 1 shows the numbering scheme and Fig. 2 the packing. The two moieties of the molecules are almost perpendicular [87 (1)°] as was previously found

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38624 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters*

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

	x	y	z	$B_{eq}(\text{Å}^2)$
C(1)	12689 (6)	7373 (5)	4576 (4)	5·2 (2)
C(2)	14415 (7)	7490 (7)	5036 (6)	7·0 (3)
C(3)	15204 (9)	7510 (9)	6266 (7)	9·1 (3)
C(4)	14251 (10)	7402 (8)	7091 (6)	9·7 (4)
C(5)	12547 (9)	7272 (7)	6705 (5)	7·8 (3)
C(6)	11823 (7)	7251 (6)	5468 (5)	5·6 (2)
C(7)	11891 (6)	7399 (6)	3223 (4)	5·5 (2)
C(8)	10577 (7)	8819 (6)	3047 (5)	6·2 (2)
N(9)	9172 (5)	7959 (5)	2144 (4)	5·7 (2)
C(10)	7507 (8)	8481 (7)	1566 (6)	7·3 (3)
C(11)	6737 (8)	6868 (8)	735 (6)	7·9 (3)
N(12)	8044 (6)	5618 (5)	1064 (4)	6·6 (2)
C(13)	9352 (6)	6358 (5)	1838 (4)	5·0 (2)
C(14)	10995 (7)	5770 (6)	2464 (5)	6·0 (2)
Cl(15)	15636 (2)	7658 (4)	4016 (2)	12·5 (1)
Cl(16)	9669 (2)	7092 (2)	5025 (1)	6·7 (1)
Cl(17)	2447 (2)	8061 (2)	251 (1)	6·8 (1)
O(18)	1336 (5)	1767 (5)	1291 (4)	8·2 (2)

Table 2. *Bond distances* (Å) *and angles* (°)

C(1)–C(2)	1·403 (9)	C(7)–C(8)	1·570 (8)
C(1)–C(6)	1·389 (8)	C(7)–C(14)	1·555 (8)
C(1)–C(7)	1·497 (8)	C(8)–N(9)	1·449 (8)
C(2)–C(3)	1·37 (1)	N(9)–C(10)	1·460 (9)
C(2)–Cl(15)	1·734 (6)	N(9)–C(13)	1·308 (7)
C(3)–C(4)	1·38 (1)	C(10)–C(11)	1·53 (1)
C(4)–C(5)	1·38 (1)	C(11)–N(12)	1·493 (9)
C(5)–C(6)	1·361 (9)	N(12)–C(13)	1·302 (8)
C(6)–Cl(16)	1·744 (6)	C(13)–C(14)	1·481 (8)
C(2)–C(1)–C(6)	114·8 (5)	C(1)–C(7)–C(14)	113·6 (5)
C(2)–C(1)–C(7)	121·0 (5)	C(8)–C(7)–C(14)	106·2 (5)
C(6)–C(1)–C(7)	124·2 (5)	C(7)–C(8)–N(9)	102·7 (5)
C(1)–C(2)–C(3)	123·4 (7)	C(8)–N(9)–C(10)	133·4 (5)
C(1)–C(2)–Cl(15)	119·3 (5)	C(8)–N(9)–C(13)	115·0 (5)
C(3)–C(2)–Cl(15)	117·3 (6)	C(10)–N(9)–C(13)	111·4 (5)
C(2)–C(3)–C(4)	118·0 (8)	N(9)–C(10)–C(11)	102·5 (5)
C(3)–C(4)–C(5)	121·8 (8)	C(10)–C(11)–C(12)	102·9 (6)
C(4)–C(5)–C(6)	117·8 (7)	C(11)–N(12)–C(13)	109·6 (5)
C(1)–C(6)–C(5)	124·1 (6)	N(9)–C(13)–N(12)	113·4 (5)
C(1)–C(6)–Cl(16)	119·7 (4)	N(9)–C(13)–C(14)	113·0 (5)
C(5)–C(6)–Cl(16)	116·1 (5)	N(12)–C(13)–C(14)	133·6 (5)
C(1)–C(7)–C(8)	114·1 (5)	C(7)–C(14)–C(13)	102·8 (5)

in the solid-state conformations of clonidine hydrochloride (Carpy, Hickel & Léger, 1979) and related drugs such as xylazine phosphate (Carpy, Gadret & Léger, 1979).

The protonation of the molecule occurs on the nitrogen N(12). In fact, the bond lengths N(9)–C(13) and N(12)–C(13) are very similar, 1·308 (7) and 1·302 (8) Å respectively; they are shorter than usual C–N bonds, *e.g.* C(8)–N(9) or C(10)–N(9). This indicates a delocalization of the positive charge; the main part of it should be located on C(13) as was previously shown for some imidazoline α_2-stimulants (Carpy *et al.*, 1982).

Among the centers which have commonly been implicated in the interaction of α-ligands with their receptor are the quaternary N and the phenyl ring (Pullman, Coubeils, Courrière & Gervois, 1972). We have calculated the distances between the two N atoms and the center of the Ph ring: $N(9)-\pi = 4\cdot85$ (2) and $N(12)-\pi = 6\cdot06$ (2) Å and between the two N atoms and the plane of the ring: 0·611 (5) and $-1\cdot229$ (5) Å respectively. The first pair of values is in agreement with our model of adrenoceptor drugs (Carpy *et al.*, 1982).

The crystalline cohesion is strengthened by the hydrogen bond $N(12)\cdots Cl(17)(1-x, \quad 1-y, \quad \bar{z}) = 3\cdot09$ (6), $N(12)-H(120) = 0\cdot95$ (6), $H(120)\cdots Cl(17)$
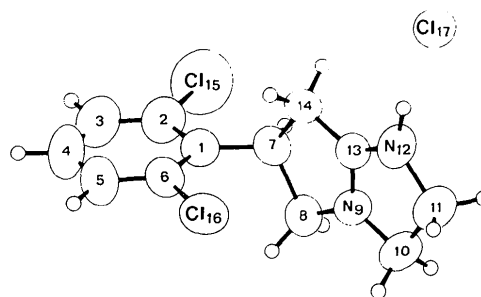
Fig. 1. Perspective view of the molecule showing the numbering of the atoms.
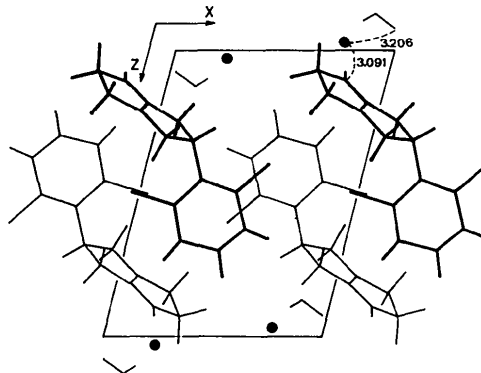
Fig. 2. Packing of the molecules projected on (010), projection axis Oy. (Distances in Å.)

$= 2 \cdot 15\ (6)$ Å,   N—H$\cdots$Cl $= 171\ (6)°$. The water molecule is hydrogen bounded with the Cl⁻ anion: Cl(17)$\cdots$O(18)$(x,\ 1 + y,\ z) = 3 \cdot 206\ (5)$, O(18)—H(180) $= 1 \cdot 01\ (6)$,   Cl(17)$\cdots$H(180) $= 2 \cdot 20\ (6)$ Å, Cl$\cdots$H—O $= 173\ (5)°$; O(18)$\cdots$Cl(17)$(x,\ 1-y,\ z) = 3 \cdot 206\ (5)$, O(18)—H(181) $= 1 \cdot 03\ (7)$, H(181)$\cdots$Cl(17) $= 2 \cdot 20\ (7)$ Å, O—H$\cdots$Cl $= 167\ (6)°$.

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Structure of 5,6-Dimethoxy-2-methyl-3-[2-(4-phenyl-1-piperazinyl)ethyl]-1*H*-indole (Oxypertine) Dihydrate, $C_{23}H_{29}N_3O_2.2H_2O$

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Abstract. $M_r = 415 \cdot 5$, monoclinic, $P2_1/n$, $a = 10 \cdot 936\ (1)$, $b = 13 \cdot 640\ (1)$, $c = 15 \cdot 502\ (2)$ Å, $\beta = 92 \cdot 66\ (1)°$, $V = 2309 \cdot 9\ (3)$ Å³, $Z = 4$, $D_x = 1 \cdot 20$ Mg m⁻³, Cu $K\bar{\alpha}$, $\lambda = 1 \cdot 54178$ Å, $\mu = 2 \cdot 69$ mm⁻¹, $F(000) = 896$, room temperature, final $R = 0 \cdot 044$ for 2112 reflections. The title compound is a tranquilizer and α-adrenoceptor antagonist. The solid-state conformation differs somewhat from that of AR-C 239, a well known specific α_1-antagonist.

Introduction. Among a series of (indolylalky)(phenyl)-piperazines synthesized some twenty years ago in Sterling–Winthrop Research Institute, New York (Archer, Wylie, Harris, Lewis, Schulenberg, Bell, Kullnig & Arnold, 1962), oxypertine has been found to possess a potent central depressant activity. This compound is used in therapeutics as a tranquilizer.

Like other groups of potent tranquilizers, oxypertine exhibits some adrenergic blocking activity (Wylie & Archer, 1962; Campbell, 1981). For this reason, we included oxypertine in a research program on the conformational requirements of α-adrenoceptor antagonists.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters*

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

	x	y	z	B_{eq}(Å²)
C(1)	7284 (3)	7384 (2)	9369 (2)	3·2 (1)
C(2)	8435 (3)	7698 (2)	9134 (2)	3·8 (1)
C(3)	9073 (3)	8395 (3)	9608 (2)	4·4 (2)
C(4)	8589 (3)	8803 (3)	10332 (2)	4·7 (2)
C(5)	7454 (3)	8503 (2)	10566 (2)	4·5 (2)
C(6)	6800 (3)	7804 (2)	10097 (2)	3·9 (1)
N(7)	6647 (2)	6675 (2)	8854 (2)	3·4 (1)
C(8)	5453 (3)	6354 (2)	9146 (2)	4·3 (2)
C(9)	4780 (3)	5727 (2)	8470 (2)	4·3 (2)
N(10)	5503 (2)	4865 (2)	8247 (2)	3·3 (1)
C(11)	6680 (3)	5206 (2)	7950 (2)	4·1 (2)
C(12)	7358 (3)	5808 (2)	8630 (2)	4·2 (2)
C(13)	4894 (3)	4273 (2)	7567 (2)	3·7 (1)
C(14)	3714 (3)	3768 (2)	7809 (2)	3·8 (1)
C(15)	3383 (3)	2982 (2)	7163 (2)	3·4 (1)
C(16)	2645 (3)	3065 (2)	6431 (2)	3·7 (1)
N(17)	2673 (2)	2204 (2)	5968 (2)	3·7 (1)
C(18)	3440 (3)	1553 (2)	6405 (2)	3·1 (1)
C(19)	3897 (3)	2011 (2)	7156 (2)	3·1 (1)
C(20)	4702 (3)	1495 (2)	7723 (2)	3·1 (1)
C(21)	5002 (3)	546 (2)	7528 (2)	3·2 (1)
C(22)	4533 (3)	101 (2)	6756 (2)	3·4 (1)
C(23)	3747 (3)	592 (2)	6194 (2)	3·5 (1)
O(24)	5768 (2)	−51 (2)	8022 (1)	4·1 (1)
C(25)	6226 (4)	331 (3)	8826 (2)	5·2 (2)
O(26)	4942 (2)	−839 (2)	6630 (2)	4·9 (1)
C(27)	4503 (4)	−1330 (3)	5869 (3)	6·4 (2)
C(28)	1872 (4)	3884 (3)	6108 (3)	5·7 (2)
O(29)	1581 (2)	1838 (2)	4232 (1)	5·4 (1)
O(30)	3199 (2)	2191 (2)	2843 (2)	5·6 (1)