

ring) is 5.67 (2) Å. The distances N(10)–plane III and N(10)–plane I are, respectively, 1.832 (3) and 0.736 (2) Å. Neither of these pairs of values agrees with our model of α -ligands (Carpy, Léger, Leclerc, Decker, Rouot & Wermuth, 1982).

The crystalline cohesion is strengthened by hydrogen bonds involving the two water molecules: N(17)···O(29) = 2.977 (4), N(17)–H(170) = 0.98 (3), H(170)···O(29) = 1.96 (3) Å, N–H···O = 173 (3)°; N(10)···O(29)($\frac{1}{2}+x$, $\frac{1}{2}-y$, $\frac{1}{2}+z$) = 2.992 (4), O(29)–H(290) = 1.00 (3), N(10)···H(290) = 2.04 (3) Å, N···H–O = 158 (3)°; N(7)···O(30)(1– x , 1– y , 1– z) = 3.063 (4), O(30)–H(301) = 0.93 (3), N(7)···H(301) = 2.16 (3) Å, N···H–O = 163 (3)°; O(26)···O(30)(1– x , \bar{y} , 1– z) = 2.837 (4), O(30)–H(300) = 0.96 (3), O(26)···H(300) = 1.92 (3) Å, O···H–O = 159 (3)°.

The sample was kindly provided by Winthrop, France.

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Structure of 3-{*N*–[(4,5-Dihydro-2-imidazolyl)methyl]-*p*-toluidino}phenol (Phentolamine) Hydrochloride, C₁₇H₂₀N₃O⁺.Cl[–]

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Abstract. α -Adrenoceptor antagonist. Antihypertensive drug. $M_r = 317.7$, monoclinic, $P2_1/c$, $a = 12.201$ (1), $b = 10.321$ (1), $c = 13.292$ (3) Å, $\beta = 97.89$ (1)°, $V = 1658.0$ (3) Å³, $Z = 4$, $D_x = 1.27$ g cm^{–3}, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 20.84$ cm^{–1}, $F(000) = 746$, room temperature. Final $R = 0.037$ for 1168 reflections. The imidazoline ring is perpendicular to the 3-hydroxyphenyl ring. Both nitrogens of the guanidine function are involved in hydrogen bonds with the Cl[–] ions.

Introduction. Phentolamine is a classical α -adrenoceptor antagonist, blocking both pre- (α_2) and postsynaptic (α_1) adrenoceptors (Langer, 1977; Starke, 1977; Westfall, 1977). It is used for its antihypertensive and vasodilating properties, specially for the diagnosis of the pheochromocytoma.

The crystal structure determination of this drug has been undertaken in order to compare its conformation with those of 2-imidazoline antihypertensive α_2 -

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adrenoceptor agonists related to clonidine (Carpy, Léger, Leclerc, Decker, Rouot & Wermuth, 1982, and references therein).

Experimental. Small white plates (from ethanol), 0.13 × 0.13 × 0.08 mm, Enraf–Nonius CAD-4 diffractometer, lattice parameters from 22 reflections ($3 < \theta < 36^\circ$), 1419 independent $\pm hkl$ with $\theta < 45^\circ$ ($-11 \leq h \leq 11$, $0 \leq k \leq 9$, $0 \leq l \leq 12$), 1168 with $I \geq 3\sigma(I)$, L_p correction, absorption ignored; direct methods (*MULTAN*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), anisotropic diagonal-matrix refinement on F , H from ΔF synthesis, isotropic, final $R = 0.037$, $wR = 0.032$, $S = 0.782$; $w = 1$ if $|F_o| < P$, $P = (F_o^2 \text{ max.}/10)^{1/2}$; $w = (P/F_o)^2$ if $|F_o| > P$; $(\Delta/\sigma)_{\text{max}} = 0.4$, in final difference map $\Delta\rho = \pm 0.3$ e Å^{–3}; f_i of non-hydrogen atoms (*International Tables for X-ray Crystallography*, 1974), f_i of H atoms (Stewart, Davidson & Simpson, 1965), IRIS 80, CII, computer of the ‘Centre Interuniversitaire de Calcul’ (Talence).

Discussion. Atomic parameters are in Table 1, bond distances and angles in Table 2.* Fig. 1 shows atomic numbering and Fig. 2 the molecular packing.

* Lists of structure factors, anisotropic thermal parameters, H coordinates and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38656 (13 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 isotropic temperature factors

$$B_{eq} = \frac{4}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
C(1)	12610 (3)	4314 (3)	3994 (3)	3.3 (2)
C(2)	11999 (3)	3259 (3)	4142 (3)	3.4 (2)
C(3)	12354 (3)	2261 (4)	4839 (3)	3.8 (2)
C(4)	13325 (3)	2285 (4)	5399 (4)	5.0 (2)
C(5)	13935 (3)	3322 (4)	5241 (4)	5.6 (2)
C(6)	13595 (3)	4341 (4)	4551 (4)	4.8 (2)
N(7)	12226 (2)	5340 (3)	3298 (2)	3.8 (1)
C(8)	12806 (3)	6503 (4)	3250 (3)	3.3 (2)
C(9)	12928 (3)	7325 (4)	4148 (3)	3.7 (2)
C(10)	13476 (3)	8463 (4)	4120 (3)	4.2 (2)
C(11)	13897 (3)	8815 (4)	3187 (3)	4.3 (2)
C(12)	13771 (3)	7980 (4)	2290 (3)	4.7 (2)
C(13)	13236 (3)	6838 (4)	2322 (3)	4.2 (2)
C(14)	11464 (3)	5036 (4)	2361 (3)	3.8 (2)
C(15)	10404 (3)	5030 (3)	2613 (3)	3.3 (2)
N(16)	10100 (2)	5667 (3)	3443 (2)	4.1 (1)
C(17)	9007 (3)	5522 (4)	3437 (3)	5.1 (2)
C(18)	8703 (3)	4636 (4)	2451 (3)	5.0 (2)
N(19)	9667 (2)	4424 (3)	2026 (2)	4.2 (1)
C(20)	14439 (4)	10073 (5)	3128 (4)	7.1 (3)
O(21)	11758 (2)	1224 (3)	5013 (2)	5.5 (1)
Cl(22)	9534 (1)	2168 (1)	4737 (1)	4.4 (0)

Table 2. Interatomic distances (\AA) and angles ($^\circ$)

C(1)–C(2)	1.385 (5)	C(9)–C(10)	1.385 (5)
C(1)–C(6)	1.390 (6)	C(10)–C(11)	1.384 (6)
C(1)–N(7)	1.409 (5)	C(11)–C(12)	1.385 (6)
C(2)–C(3)	1.377 (5)	C(11)–C(20)	1.492 (7)
C(3)–C(4)	1.376 (6)	C(12)–C(13)	1.380 (6)
C(3)–O(21)	1.366 (5)	C(14)–C(15)	1.482 (5)
C(4)–C(5)	1.372 (6)	C(15)–N(16)	1.316 (5)
C(5)–C(6)	1.383 (6)	C(15)–N(19)	1.294 (5)
N(7)–C(8)	1.432 (5)	N(16)–C(17)	1.459 (5)
N(7)–C(14)	1.454 (5)	C(17)–C(18)	1.521 (6)
C(8)–C(9)	1.378 (5)	C(18)–N(19)	1.463 (5)
C(8)–C(13)	1.381 (5)		
C(2)–C(1)–C(6)	118.8 (3)	C(8)–C(9)–C(10)	120.5 (4)
C(2)–C(1)–N(7)	119.9 (3)	C(9)–C(10)–C(11)	121.1 (4)
C(6)–C(1)–N(7)	121.2 (3)	C(10)–C(11)–C(12)	117.9 (4)
C(1)–C(2)–C(3)	120.7 (3)	C(10)–C(11)–C(20)	121.4 (4)
C(2)–C(3)–C(4)	120.8 (4)	C(12)–C(11)–C(20)	120.7 (4)
C(2)–C(3)–O(21)	121.7 (3)	C(11)–C(12)–C(13)	121.1 (4)
C(4)–C(3)–O(21)	117.5 (4)	C(8)–C(13)–C(12)	120.6 (4)
C(3)–C(4)–C(5)	118.4 (4)	N(7)–C(14)–C(15)	114.6 (3)
C(4)–C(5)–C(6)	121.9 (4)	C(14)–C(15)–N(16)	124.0 (3)
C(1)–C(6)–C(5)	119.3 (4)	C(14)–C(15)–N(19)	123.8 (3)
C(1)–N(7)–C(8)	120.2 (3)	N(16)–C(15)–N(19)	112.2 (3)
C(1)–N(7)–C(14)	117.8 (3)	C(15)–N(16)–C(17)	110.8 (3)
C(8)–N(7)–C(14)	118.0 (3)	N(16)–C(17)–C(18)	102.8 (3)
N(7)–C(8)–C(9)	119.3 (3)	C(17)–C(18)–N(19)	102.7 (3)
N(7)–C(8)–C(13)	122.0 (3)	C(15)–N(19)–C(18)	111.4 (3)
C(9)–C(8)–C(13)	118.8 (3)		

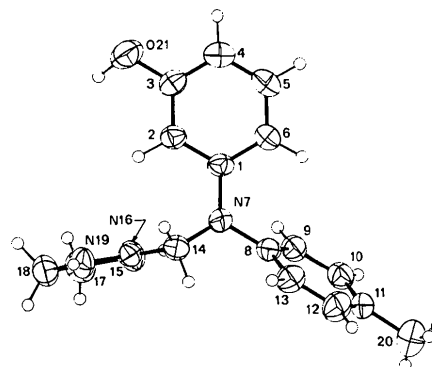


Fig. 1. Perspective view of the molecule showing the numbering of atoms. H atoms are numbered such that Hmn is the n th H on Xm ($X = O, N$ and C). Cl(22) is the chloride ion.

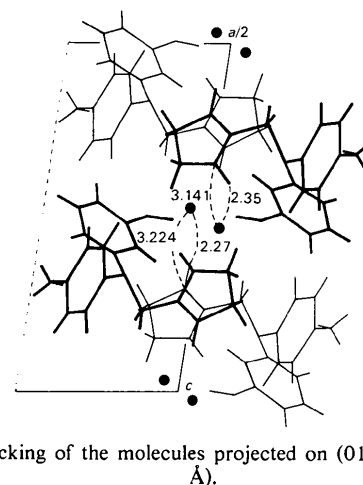


Fig. 2. Packing of the molecules projected on (010) (distances in \AA).

The respective positions of the three rings of the molecule are defined by the following angles: 3-hydroxyphenyl/*p*-toluidinomethyl = $73 (1)^\circ$; *p*-toluidinomethyl/imidazole = $48 (1)^\circ$; 3-hydroxyphenyl/imidazole = $88 (1)^\circ$. The last value has already been found in the solid-state conformations of clonidine phosphate (Carpy, Hickel & Léger, 1979) and related α_2 -agonists such as xylazine phosphate (Carpy, Gadret & Léger, 1979), lofexidine hydrochloride (Carpy, Hickel & Léger, 1980) or IPRO-4 hydrochloride (Carpy, Léger, Wermuth & Leclerc, 1981). One can also define the respective positions of the rings by the values of the torsion angles ($\pm 1^\circ$):

C(2)–C(1)–N(7)–C(8)	172	C(2)–C(1)–N(7)–C(14)	
C(9)–C(8)–N(7)–C(1)	–70	C(1)–N(7)–C(14)–C(15)	
C(9)–C(8)–N(7)–C(14)	133	N(7)–C(14)–C(15)–N(16)	
C(8)–N(7)–C(14)–C(15)	244		

Even though the C(15)–N(19) bond is shorter than C(15)–N(16), 1.294 (5) and 1.316 (5) \AA respectively, both nitrogens of the guanidine function are chemically equivalent. The C(15)–N bond lengths are shorter than N(16)–C(17) and C(18)–N(19); this shows a delocalization of the positive charge on the C(15) atom

and probably on the H atoms bonded to the nitrogens as already shown by CNDO/2 calculations on imidazolines (Carpy, Léger, Leclerc, Decker, Rouot & Wermuth, 1982).

Among the centres, those which have commonly been implicated in the interaction of α -ligands with their receptor are the quaternary N and the hydroxyphenyl ring (Pullman, Coubeils, Courrière & Gervois, 1972). We have calculated the distances D between the two nitrogens of the guanidine function and the centre π of the hydroxyphenyl ring: $N(16)-\pi = 4.40$ (1) and $N(19)-\pi = 5.12$ (1) Å and the distances H between the two nitrogens and the plane containing the ring: -1.575 (3) and 0.191 (3) Å respectively. The distance D related to N(19) is in good agreement with those found in imidazoline agonists (≈ 5 Å) but H is much smaller (~ 0.2 Å instead of ~ 1 Å). In contrast this H distance seems consistent with those found in various α -antagonists such as WB-4101 (Carpy, Colleter & Léger, 1981) and raubasine (Dubost, Léger, Goursole, Carpy & Colleter, 1983).

The crystalline cohesion is ensured by two hydrogen bonds involving the two nitrogens of the imidazoline ring and the Cl⁻ ions: $N(16)\cdots Cl(22)(2-x, 1-y, 1-z) = 3.141$ (3) Å, $H(160)\cdots Cl(22) = 2.35$ (4) Å, $N-H\cdots Cl = 139$ (3)°, $Cl(22)\cdots N(19)(x, \frac{1}{2}-y, \frac{1}{2}+z) = 3.224$ (3) Å, $Cl(22)\cdots H(190) = 2.27$ (4) Å, $Cl\cdots H-N = 167$ (3)°, by C-H \cdots O bonds and by van der Waals contacts.

The sample was kindly provided by CIBA-GEIGY Limited (Basle, Switzerland).

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Structure of (+)-2,2-Dimethoxy-2-(6-methoxy-2-naphthyl)-1-methylethyl 2-Oxo-10-bornanesulfonate, C₂₆H₃₄O₇S

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Abstract. $M_r = 490.6$, orthorhombic, $P2_12_12_1$, $a = 10.703$ (1), $b = 29.091$ (5), $c = 8.435$ (1) Å, $V = 2626.4$ (5) Å³, $Z = 4$, $D_x = 1.24$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 1.41$ mm⁻¹, $F(000) = 1048$, $T = 298$ (2) K. Final $R = 0.047$ for 1916 unique observed reflections. The absolute configuration around the asymmetric C atom has been established as R .

Introduction. New methods for the synthesis of α -arylalkanoic acids by the use of 1,2-rearrangement of the aryl group have been designed to obtain biologically important substances (Tsuchihashi, Kitajima & Mitamura, 1981). Crystals of the title compound (I), a starting material of the 1,2-rearrangement reaction, have been subjected to X-ray crystal-structure analysis