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5-Chloro-1-[3-(dimethylamino)propyl]-1,3-dihydro-3-phenyl-2*H*-benzimidazol-2-one Monohydrochloride Monohydrate (Clodazone)

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Abstract. $C_{18}H_{20}ClN_3O.H_2O.HCl$, $M_r = 384{\cdot}31$, monoclinic, $P2_1/c$, $a = 5{\cdot}898$ (6), $b = 31{\cdot}911$ (10), $c = 10{\cdot}817$ (6) Å, $\beta = 103{\cdot}52$ (5)°, $V = 1989{\cdot}3$ Å^3, $D_c = 1{\cdot}29$, $D_m = 1{\cdot}29$ g cm^{-3}, $Z = 4$; $R = 0{\cdot}030$ for 1833 reflexions with $I \geq 2{\cdot}5\sigma$; R_w $\{=[\sum w(|F_o| - |F_c|)^2/\sum wF_o^2]^{1/2}\} = 0{\cdot}038$. The conformation of the molecule corresponds to one of the five minima calculated for the isolated molecule.

Introduction. The title compound seems to act as a competitive inhibitor of noradrenaline and its structure was solved as part of a study on the structure–activity relationship of antidepressant drugs.

Samples were selected after recrystallization from 2-propanol. A colourless crystal was studied by photographic and diffractometric techniques. The cell dimensions and intensities were measured on a Nonius CAD-4 automatic diffractometer. The experimental conditions are given in Table 1.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) and refined by full-matrix least squares (Frenz & Okaya, 1975), with scattering

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Table 1. *Experimental conditions*

Crystal dimensions: 0·30 × 0·21 × 0·18 mm	
Absences: $h0l$, $l \neq 2n$; $0k0$, $k \neq 2n$; space group $P2_1/c$	
Source: graphite-monochromatized Cu $K\bar{\alpha}$ radiation; $\lambda = 1{\cdot}54178$ Å	
Scan: ω–2θ	
Scan length: $\Delta\theta = 0{\cdot}6 + 0{\cdot}3$ tg θ (°)	
Apertures: horizontal = 2·5 + 0·5 tg θ (mm); vertical = 4 mm	
$\theta_{min} = 2{\cdot}0°$; $\theta_{max} = 70{\cdot}0°$	
Confidence level: $2{\cdot}5\sigma$ with $\sigma^2(I) = S + B + (0{\cdot}03S)^2$, S being the total peak count and B the background count	
Number of independent measured reflexions: 3864	
Number of accepted reflexions ($\geq 2{\cdot}5\sigma$): 1833	
$\mu = 30{\cdot}799$ cm^{-1}; no corrections applied	
Weighting scheme: $w = 1/\sigma(F_o)^2$; $\sigma(F_o^2) = [\sigma^2(I) + (0{\cdot}02\,F_o^2)^2]^{1/2}$/Lp	

Table 2. *Fractional atomic coordinates* ($\times 10^4$, *for* H $\times 10^3$)

	x	*y*	*z*
Cl(1)	7537 (2)	4688 (1)	3011 (1)
C(2)	10014 (6)	4369 (1)	3260 (3)
C(3)	11649 (6)	4404 (1)	4399 (3)
C(4)	13560 (6)	4143 (1)	4556 (3)
C(5)	13843 (6)	3365 (1)	3610 (3)
C(6)	12212 (7)	3838 (1)	2479 (3)
C(7)	10262 (6)	4095 (1)	2313 (3)
N(8)	15526 (5)	4105 (1)	5551 (2)
C(9)	17033 (6)	3805 (1)	5237 (3)
O(10)	18901 (4)	3695 (1)	5904 (2)
N(11)	15995 (5)	3667 (1)	4050 (3)
C(12)	16918 (6)	3318 (1)	3452 (3)
C(13)	16495 (6)	2901 (1)	4014 (3)
C(14)	17252 (6)	2548 (1)	3267 (3)
N(15)	17516 (4)	2139 (1)	3943 (2)
C(16)	15366 (7)	1997 (1)	4306 (4)
C(17)	18339 (7)	1813 (1)	3156 (4)
C(18)	15821 (6)	4275 (1)	6794 (3)
C(19)	14233 (7)	4181 (1)	7499 (3)
C(20)	14582 (7)	4342 (1)	8718 (4)
C(21)	16480 (8)	4589 (1)	9212 (4)
C(22)	18025 (8)	4680 (2)	8492 (4)
C(23)	17706 (7)	4526 (1)	7283 (4)
Cl(24)	11417 (2)	2769 (1)	1290 (1)
O(25)	12907 (5)	3184 (1)	5977 (3)
H(31)	1149 (5)	460 (1)	504 (3)
H(61)	1233 (6)	363 (1)	184 (3)
H(71)	905 (6)	409 (1)	153 (3)
H(121)	1611 (6)	332 (1)	252 (3)
H(122)	1863 (5)	336 (1)	354 (3)
H(131)	1496 (5)	287 (1)	403 (3)
H(132)	1730 (5)	288 (1)	485 (3)
H(141)	1616 (5)	251 (1)	248 (3)
H(142)	1881 (5)	262 (1)	314 (3)
H(151)	1877 (6)	217 (1)	478 (3)
H(161)	1489 (6)	223 (1)	490 (3)
H(162)	1417 (6)	195 (1)	356 (3)
H(163)	1571 (6)	174 (1)	474 (3)
H(171)	1716 (6)	178 (1)	238 (3)
H(172)	1865 (6)	156 (1)	368 (3)
H(173)	1982 (6)	188 (1)	305 (3)
H(191)	1291 (6)	401 (1)	713 (3)
H(201)	1350 (6)	427 (1)	914 (3)

Table 2 (*cont.*)

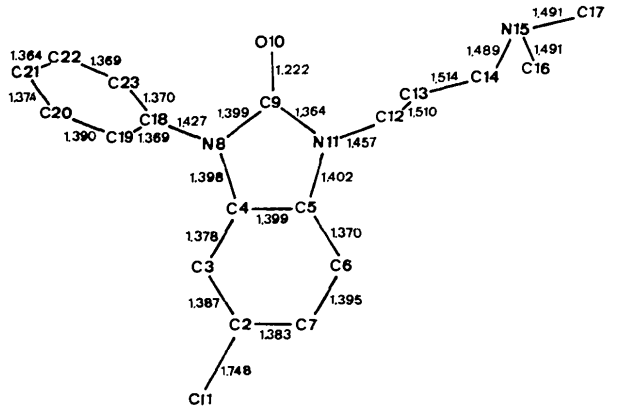
	x	*y*	*z*
H(211)	1661 (6)	471 (1)	1007 (3)
H(221)	1936 (7)	485 (1)	887 (4)
H(231)	1884 (6)	455 (1)	683 (3)
H(251)	1236 (6)	292 (1)	597 (3)
H(252)	1177 (7)	331 (1)	585 (3)

factors and anomalous-dispersion coefficients from Cromer & Waber (1974). Sixteen H atoms found in a ΔF map were included in the last cycle of refinement with the calculated positions of the remaining seven H atoms. For the 1833 accepted reflexions, the final R was 0·030 and R_w = 0·038. The final atomic parameters are given in Table 2.*

Discussion. The atomic numbering and the bond distances and angles are given in Fig. 1. Table 3 gives the bond distances and angles involving H atoms (numbered according to the atoms to which they are bonded). Fig. 2 shows the conformation of the molecule; Fig. 3 shows the packing. The atoms of the benzimidazolinone group are coplanar within 2°; the dihedral angle between this moiety and the phenyl substituent is 64°.

Potential-energy calculations, allowing rotation about C(12)–C(13) and C(13)–C(14), show that the

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33483 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.



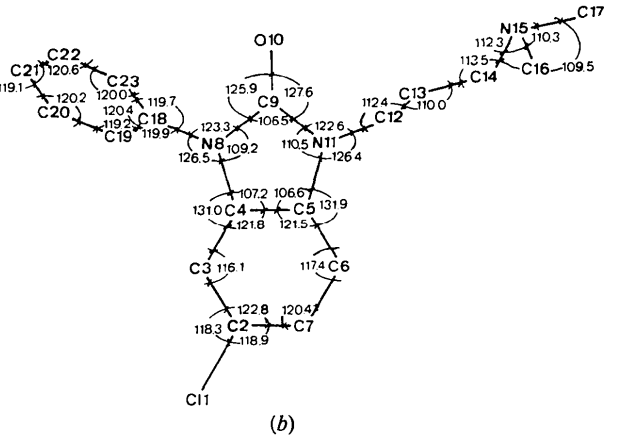


Fig. 1. (*a*) Bond distances (Å) and (*b*) bond angles (°). The e.s.d.'s for the bond lengths are in the range 0·002–0·004 Å and for the angles 0·2–0·3°.

Table 3. *Bond lengths* (Å) *and angles* (°) *involving* H *atoms*

The e.s.d.'s are in parentheses.

C(3)–H(31)	0·95 (2)	C(13)–H(132)	0·92 (2)	C(16)–H(163)	0·93 (3)	C(21)–H(211)	0·99 (3)
C(6)–H(61)	0·97 (2)	C(14)–H(141)	0·95 (2)	C(17)–H(171)	0·96 (3)	C(22)–H(221)	0·97 (3)
C(7)–H(7)	0·98 (2)	C(14)–H(142)	0·99 (2)	C(17)–H(172)	0·98 (3)	C(23)–H(231)	0·92 (2)
C(12)–H(121)	1·02 (2)	N(15)–H(151)	1·04 (2)	C(17)–H(173)	0·93 (3)	O(25)–H(251)	0·89 (3)
C(12)–H(122)	1·00 (2)	C(16)–H(161)	1·06 (3)	C(19)–H(191)	0·95 (2)	O(25)–H(252)	0·77 (3)
C(13)–H(131)	0·91 (2)	C(16)–H(162)	0·95 (2)	C(20)–H(201)	0·90 (2)		

C(2)–C(3)–H(31)	123·0 (13)	C(13)–C(14)–H(141)	110·2 (12)	N(15)–C(17)–H(173)	110·1 (16)
C(4)–C(3)–H(31)	120·9 (13)	C(13)–C(14)–H(142)	107·3 (11)	H(171)–C(17)–H(172)	114·4 (22)
C(5)–C(6)–H(61)	122·1 (14)	N(15)–C(14)–H(141)	108·1 (12)	H(171)–C(17)–H(173)	115·0 (23)
C(7)–C(6)–H(61)	120·2 (13)	N(15)–C(14)–H(142)	106·5 (11)	H(172)–C(17)–H(173)	102·5 (22)
C(2)–C(7)–H(71)	118·8 (14)	H(141)–C(14)–H(142)	111·2 (16)	C(18)–C(19)–H(191)	119·0 (14)
C(6)–C(7)–H(71)	120·9 (14)	C(14)–N(15)–H(151)	108·5 (12)	C(20)–C(19)–H(191)	122·2 (15)
N(11)–C(12)–H(121)	108·0 (13)	C(16)–N(15)–H(151)	106·1 (12)	C(19)–C(20)–H(201)	114·9 (17)
N(11)–C(12)–H(122)	108·8 (13)	C(17)–N(15)–H(151)	108·0 (12)	C(21)–C(20)–H(201)	124·3 (17)
C(13)–C(12)–H(121)	108·6 (13)	N(15)–C(16)–H(161)	108·7 (13)	C(20)–C(21)–H(211)	117·7 (15)
C(13)–C(12)–H(122)	110·0 (13)	N(15)–C(16)–H(162)	108·9 (15)	C(22)–C(21)–H(211)	122·7 (15)
H(121)–C(12)–H(122)	109·1 (18)	N(15)–C(16)–H(163)	107·5 (16)	C(21)–C(22)–H(221)	117·5 (17)
C(12)–C(13)–H(131)	111·0 (14)	H(161)–C(16)–H(162)	111·9 (20)	C(23)–C(22)–H(221)	121·7 (18)
C(12)–C(13)–H(132)	111·5 (14)	H(161)–C(16)–H(163)	110·1 (19)	C(18)–C(23)–H(231)	117·7 (17)
C(14)–C(13)–H(131)	110·4 (14)	H(162)–C(16)–H(163)	109·8 (21)	C(22)–C(23)–H(231)	121·8 (17)
C(14)–C(13)–H(132)	108·9 (14)	N(15)–C(17)–H(171)	108·4 (16)	H(251)–O(25)–H(252)	101·0 (30)
H(131)–C(13)–H(132)	104·9 (19)	N(15)–C(17)–H(172)	106·1 (16)		

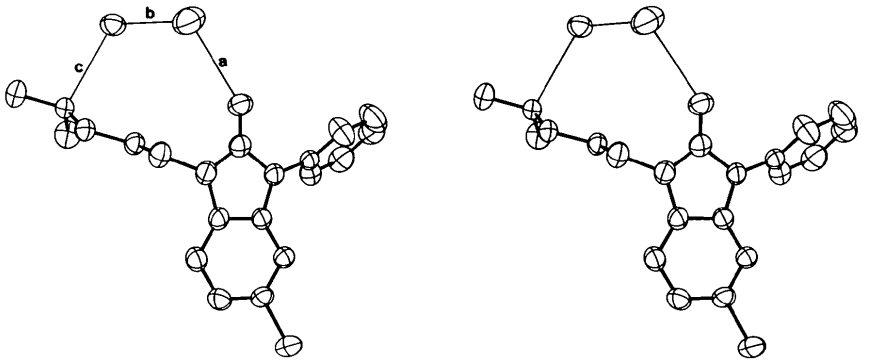


Fig. 2. Stereoscopic view of the molecule with 50% probability thermal ellipsoids (Johnson, 1965).

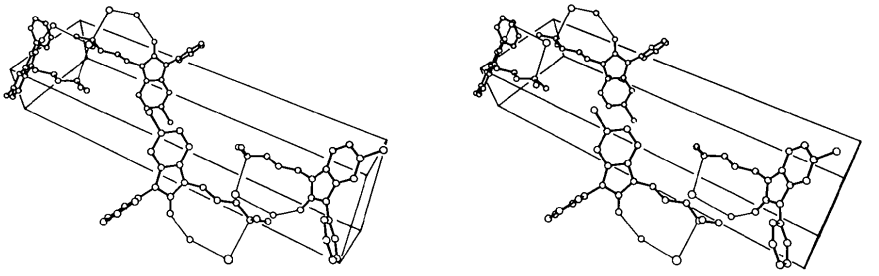


Fig. 3. Stereoscopic view of the molecular packing.

Table 4. *Hydrogen-bond distances* (Å)

O(25)···O(10)	2·858 (2)	N(15)···Cl(24)	3·025 (2)
H(252)···O(10)	2·08 (3)	H(151)···Cl(24)	2·26 (3)
O(25)···Cl(24)	3·204 (2)		
H(251)···Cl(24)	2·33 (3)		

crystal structure corresponds to one of the five energy minima of the isolated molecule.

Three H atoms are involved in hydrogen bonding (Table 4): water O(25)–H(252)···O(10) ($x-1, y, z$); water O(25)–H(251)···Cl(24) ($x, \frac{1}{2}-y, \frac{1}{2}+z$); and Cl(24)···H(151)–N(15) ($x-1, \frac{1}{2}-y, z-\frac{1}{2}$).

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