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Clemizole Hydrochloride

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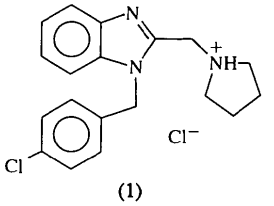
Abstract

The crystal structure of the title compound, 1-[1-(*p*-chlorobenzyl)-2-benzimidazolylmethyl]pyrrolidinium chloride, $C_{19}H_{21}ClN_3^+.Cl^-$, a potent anti-allergic agent, has been determined. The crystals are composed of independent cations which are hydrogen bonded to chloride ions, with an N···Cl distance of 3.021 (6) Å and an N—H···Cl angle of 173°.

Comment

As a continuation of studies on the anti-allergic drugs [5-methoxy-3-(1-methylethoxy)-1-phenyl-*N*-(1*H*-tetrazol-5-yl)-1*H*-indole-2-carboxamide–diethylamine (Parvez, Unangst, Connor & Mullican, 1991*a*) and 3-(1-methylethoxy)-7-phenyl-*N*-(1*H*-tetrazol-5-yl)-2-benzofurancarboxamide (Parvez, Unangst, Connor & Mullican, 1991*b*)] effective on H1 receptors, the crystal structure of clemizole hydrochloride, (1), has been determined and is described herein.



(1)

An *ORTEP* drawing (Johnson, 1976) of (1) with the atomic numbering scheme is shown in Fig. 1. The molecular dimensions in the cation are unexceptional, with mean bond distances Cl—C_{sp^2} 1.722 (9), N—C_{sp^3} 1.50 (3), N—C_{sp^2} 1.37 (1), C_{sp^3}—C_{sp^3} (in the pyrrolidinyl ring) 1.49 (3), C_{sp^3}—C_{sp^2} 1.50 (1), C—$C_{aromatic}$ 1.38 (1) and C=N 1.325 (8) Å, and normal angles. The benzimidazole moiety and the six-membered phenyl ring are essentially planar, with maximum deviations from the respective least-squares planes of 0.006 (9) and 0.016 (9) Å; the planes are inclined at almost right angles to one another [82.8 (9)°]. The pyrrolidinyl ring has an N3-envelope conformation, with the N3 atom 0.537 (6) Å out of the plane of the remaining four C atoms of the ring [maximum deviation of 0.04 (1) Å for the C17 atom].

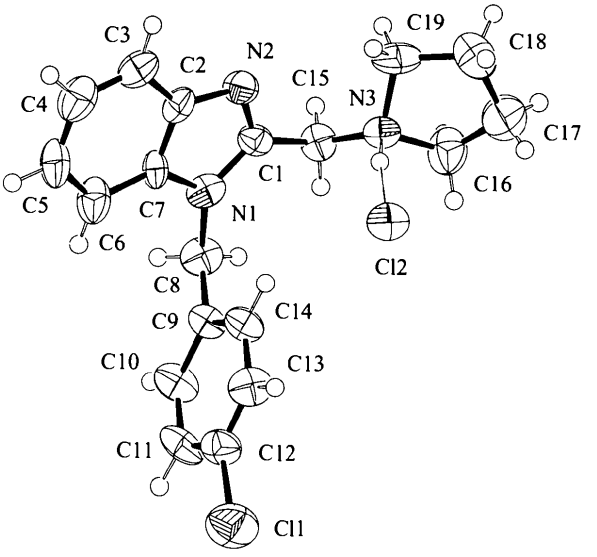


Fig. 1. An *ORTEP*II drawing (Johnson, 1976) of the title compound with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms have been assigned arbitrary radii.

The clemizole cation is hydrogen bonded to the chloride ion *via* normal N—H···Cl interactions (Table 2).

Experimental

Colourless prismatic crystals of the title compound (Sigma Inc.) were grown from a mixture of CH_3OH/CH_3CN (1:1) by slow evaporation at room temperature.

Crystal data

$C_{19}H_{21}ClN_3^+.Cl^-$
M_r = 362.30
Monoclinic
$P2_1/n$
a = 5.525 (2) Å
b = 10.968 (3) Å
c = 30.003 (5) Å
β = 91.01 (3)°
V = 1817.8 (7) Å^3
Z = 4
D_x = 1.324 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 17 reflections
θ = 18.4–24.4°
μ = 0.362 mm^{-1}
T = 296 K
Needle
0.50 × 0.40 × 0.30 mm
Colourless

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
1488 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.044

Absorption correction:	$\theta_{max} = 27.56°$
ψ scans (North, Phillips & Mathews, 1968)	$h = 0 \rightarrow 7$
$T_{min} = 0.922$, $T_{max} = 1.000$	$k = 0 \rightarrow 14$
4873 measured reflections	$l = -39 \rightarrow 39$
4424 independent reflections	3 standard reflections monitored every 200 reflections
	intensity decay: 0.5%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.03$
$R = 0.0663$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
$wR = 0.0647$	$\Delta\rho_{min} = -0.40$ e Å^{-3}
$S = 2.967$	Extinction correction: none
1488 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
217 parameters	
H atoms riding with C—H and N—H 0.95 Å	
$w = 1/[\sigma^2(F_o) + 0.013(F_o^2)]$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Cl1	0.2812 (5)	−0.0013 (2)	0.04817 (9)	0.093 (1)
Cl2	0.6271 (3)	−0.3527 (2)	0.21221 (7)	0.0534 (7)
N1	0.1854 (10)	−0.5783 (6)	0.1281 (2)	0.045 (2)
N2	0.4311 (12)	−0.6965 (6)	0.1686 (2)	0.049 (2)
N3	0.2775 (10)	−0.5423 (5)	0.2456 (2)	0.041 (2)
C1	0.2441 (13)	−0.6210 (7)	0.1694 (3)	0.042 (3)
C2	0.4955 (13)	−0.7036 (7)	0.1248 (3)	0.044 (3)
C3	0.6789 (14)	−0.7705 (7)	0.1042 (3)	0.057 (3)
C4	0.7048 (15)	−0.7603 (9)	0.0593 (3)	0.066 (3)
C5	0.5535 (16)	−0.6864 (9)	0.0337 (3)	0.064 (3)
C6	0.3694 (14)	−0.6199 (8)	0.0527 (3)	0.061 (3)
C7	0.3438 (12)	−0.6304 (7)	0.0984 (2)	0.039 (2)
C8	−0.0122 (12)	−0.4952 (8)	0.1143 (3)	0.054 (3)
C9	0.0700 (12)	−0.3753 (7)	0.0967 (2)	0.042 (2)
C10	−0.0741 (13)	−0.3125 (8)	0.0669 (3)	0.056 (3)
C11	−0.0141 (15)	−0.1988 (9)	0.0507 (3)	0.061 (3)
C12	0.2004 (15)	−0.1447 (8)	0.0660 (3)	0.055 (3)
C13	0.3494 (14)	−0.2078 (8)	0.0947 (3)	0.060 (3)
C14	0.2856 (14)	−0.3212 (8)	0.1105 (3)	0.051 (3)
C15	0.1109 (13)	−0.5880 (7)	0.2111 (3)	0.048 (3)
C16	0.1440 (15)	−0.4816 (9)	0.2838 (3)	0.070 (3)
C17	0.3169 (19)	−0.4781 (10)	0.3205 (3)	0.102 (5)
C18	0.4935 (15)	−0.5806 (10)	0.3140 (3)	0.073 (4)
C19	0.4305 (13)	−0.6369 (8)	0.2694 (3)	0.062 (3)

Table 2. *Selected geometric parameters (Å, °)*

Cl1—C12	1.722 (9)	N3—C16	1.526 (10)
N1—C1	1.357 (8)	N3—C19	1.509 (9)
N1—C7	1.384 (8)	C1—C15	1.507 (10)
N1—C8	1.475 (9)	C8—C9	1.491 (10)
N2—C1	1.325 (8)	C16—C17	1.45 (1)
N2—C2	1.371 (8)	C17—C18	1.50 (1)
N3—C15	1.462 (9)	C18—C19	1.51 (1)
C1—N1—C7	107.5 (6)	N2—C2—C3	130.8 (8)
C1—N1—C8	129.3 (7)	N2—C2—C7	110.4 (6)
C7—N1—C8	123.2 (7)	N1—C7—C2	104.6 (6)
C1—N2—C2	105.5 (6)	N1—C7—C6	132.8 (8)
C15—N3—C16	112.0 (6)	N1—C8—C9	114.5 (6)
C15—N3—C19	116.0 (6)	N3—C15—C1	111.1 (6)
C16—N3—C19	102.7 (6)	N3—C16—C17	105.1 (7)
N1—C1—N2	112.0 (7)	C16—C17—C18	107.6 (9)
N1—C1—C15	124.2 (7)	C17—C18—C19	106.3 (7)
N2—C1—C15	123.8 (7)	N3—C19—C18	104.8 (7)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1N3···Cl2	0.95	2.08	3.021 (6)	173

The space group, $P2_1/n$, was uniquely determined from the systematic absences: $h0l$, $h + l = 2n + 1$ and $0k0$, $k = 2n + 1$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,3-Bis(ethylsulfonyl)benzo[*b*]thiophene

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Abstract

The structure of the title compound, diethyl benzo[*b*]-thiophene-2,3-disulfinate, $C_{12}H_{14}O_4S_3$, is composed of an essentially planar benzothiophene moiety contain-