C(1)—N(1)—C(2)	118.31 (21)	C(3)—C(4)—C(5)	118.4 (3)
C(3)—N(2)—C(7)	116.87 (25)	C(4)—C(5)—C(6)	118.9 (3)
C(8)—N(3)—C(12)	116.50 (24)	C(5)—C(6)—C(7)	118.9 (3)
N(1)—C(1)—C(2*a*)	121.20 (21)	N(2)—C(7)—C(6)	123.3 (3)
N(1)—C(1)—C(8)	114.97 (21)	N(3)—C(8)—C(1)	116.63 (22)
C(2*a*)—C(1)—C(8)	123.80 (22)	N(3)—C(8)—C(9)	123.35 (24)
N(1)—C(2)—C(1*a*)	120.49 (22)	C(1)—C(8)—C(9)	119.97 (23)
N(1)—C(2)—C(3)	115.99 (21)	C(8)—C(9)—C(10)	118.3 (3)
C(1*a*)—C(2)—C(3)	123.51 (21)	C(9)—C(10)—C(11)	119.5 (3)
N(2)—C(3)—C(2)	115.35 (22)	C(10)—C(11)—C(12)	118.0 (3)
N(2)—C(3)—C(4)	123.64 (25)	N(3)—C(12)—C(11)	124.3 (3)
C(2)—C(3)—C(4)	121.00 (24)		

Table 3. *A comparison of various dihedral angles* (°) *and short intramolecular distances* (Å) *in* TPPZ-I *and* BPQ-I, *and* TPPZ-II, BPQ-II *and* BPPZ

	TPPZ-I	BPQ-I	TPPZ-II	BPQ-II	BPPZ
Angles					
∠*AB*	60.4	58.9	62.4	60.6	54.1
∠*AC*	59.0	54.0	48.9	31.7	42.2
∠*BC*	46.4	45.2	51.7	46.4	42.2
Distances					
N(1)···N(2)	2.907	2.803			
N(1)···C(9)	2.912	2.910			
N(3)···C(4a^{i})	3.297	3.180			
N(1)···C(4)			2.939	2.854	2.859
N(1)···C(9)			2.958	2.941	2.859
N(2)···N(3a^{ii})			3.237	3.017	2.962

Symmetry operations: (i) 1.5 - *x*, 0.5 - *y*, 1.5 - *z*; (ii) 2 -*x*, -*y*, -*z*.

The sample of TPPZ used in the synthesis of the zinc(II) complex was originally synthesized according to Goodwin & Lions (1959) and recrystallized from CH_2Cl_2. The structure was solved by direct methods using the *NRCVAX* system (Gabe, Le Page, Charland, Lee & White, 1989) which was used for all further calculations. The H atoms were located from difference maps.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55512 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1025]

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Structure of Eltoprazine

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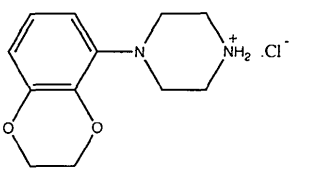
Abstract

The piperazine ring of 1-(2,3-dihydro-1,4-benzodioxin-5-yl)piperazine hydrochloride is oriented in such a way that the C atoms of one of the C—C bonds are almost coplanar with the plane of the phenyl ring. The piperazine ring adopts an almost perfect chair conformation and the dihydro-1,4-dioxine ring a half-chair conformation. The protonated secondary amino group donates two strong hydrogen bonds to symmetry-related Cl^- anions.

Comment

The psychoactive drug eltoprazine (1) reduces aggressive behaviour (Mos & Olivier, 1988; Olivier, Mos, van der Heyden & Hartog, 1989) and enhances social interest and exploration in rats (Olivier, Mos, van der Heyden & Hartog, 1989). This compound has recently been shown to bind at serotonergic (5-HT_1) receptor sites in the rat brain (Sijbesma, Schipper & de Kloet, 1990) and its conformation might therefore be very useful in developing pharmacophores for serotonergic receptors.



(1)

The X-ray results show that the C6—C5—N1—C9 torsional angle, the main source of conformational freedom, is 11(1)°. This means that one of the C—C bonds of the piperazine ring is almost coplanar with the plane of the phenyl ring. The piperazine ring appears to be in an almost perfect chair conformation and the dihydro-1,4-dioxine ring adopts a half-chair conformation. The terminal secondary amino group is protonated and donates two strong hydrogen bonds to Cl^- at *x*, 1+*y*, *z* and Cl^- at 2 −*x*, $\frac{1}{2}$+*y*, $\frac{1}{2}$ − *z* with geometries N···Cl^-, N—H, H···Cl^- and N—H···Cl^- of 3.172(8), 1.08(1), 2.125(8) Å and 162.7(8)° and 3.045(8), 1.08(1), 1.980(8) Å and 168.3(8)° respectively.

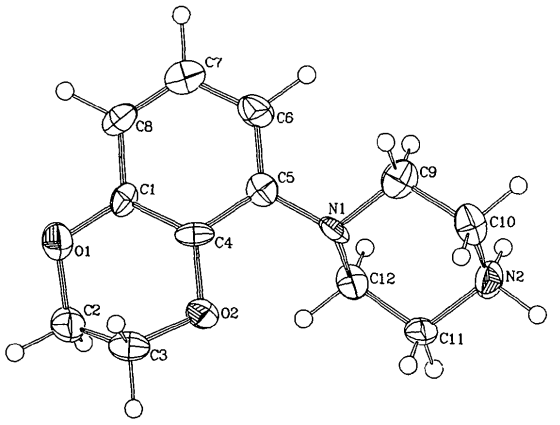


Fig. 1. View of the $C_{12}H_{17}N_2O_2$ part of the title compound showing the labelling of the non-H atoms. Thermal ellipsoids are shown at a 50% probability level; H atoms are drawn as small circles of arbitrary radius.

Experimental

Crystal data

$C_{12}H_{17}N_2O_2^+.Cl^-$
M_r = 256.73
Orthorhombic
$P2_12_12_1$
a = 7.315 (1) Å
b = 7.941 (2) Å
c = 21.393 (4) Å
V = 1242.7 (4) Å^3
Z = 4
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 15.67–18.01°
μ = 2.97 cm^{-1}
T = 295 K
Plate
0.33 × 0.25 × 0.05 mm
Colourless
Crystal source: grown from ethanol solution

Data collection

Enraf–Nonius CAD-4 diffractometer
Background-peak-background from $\omega/2\theta$ scans
Absorption correction: none
2998 measured reflections
2639 independent reflections
850 observed reflections [$I>2.5\sigma(I)$]
R_{int} = 0.120
θ_{max} = 27.50°
h = −9 → 9
k = 0 → 11
l = 0 → 27
3 standard reflections
frequency: 60 min
intensity variation: 1.2%

Refinement

Refinement on F
Final R = 0.069
wR = 0.063
S = 1.41
850 reflections
171 parameters
No hydrogen parameters were refined except for an overall displacement parameter
$w = 1/[\sigma^2(F)+0.0004F^2]$
$(\Delta/\sigma)_{max}$ = 0.091
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1990*a*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *EUCLID* (Spek, 1982). Software used to prepare material for publication: *PLATON*92 (Spek, 1990*b*).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.140 (1)	0.4225 (8)	0.0017 (3)	0.047 (3)
O2	0.421 (1)	0.4142 (7)	0.0945 (3)	0.035 (2)
N1	0.498 (1)	0.6923 (9)	0.1637 (3)	0.029 (3)
N2	0.830 (1)	0.783 (1)	0.2272 (4)	0.040 (3)
C1	0.176 (1)	0.558 (1)	0.0392 (4)	0.027 (3)
C2	0.275 (1)	0.289 (1)	0.0058 (5)	0.041 (4)
C3	0.329 (2)	0.264 (1)	0.0716 (4)	0.037 (4)
C4	0.312 (1)	0.556 (1)	0.0840 (4)	0.029 (3)
C5	0.347 (1)	0.700 (1)	0.1203 (4)	0.029 (3)
C6	0.236 (1)	0.837 (1)	0.1130 (5)	0.040 (4)
C7	0.095 (1)	0.834 (1)	0.0701 (4)	0.037 (4)
C8	0.065 (1)	0.701 (1)	0.0331 (5)	0.036 (4)
C9	0.502 (2)	0.831 (1)	0.2085 (4)	0.041 (4)
C10	0.647 (1)	0.805 (1)	0.2568 (4)	0.042 (4)
C11	0.824 (1)	0.642 (1)	0.1808 (5)	0.034 (3)
C12	0.678 (1)	0.677 (1)	0.1325 (4)	0.035 (3)
Cl1	0.8903 (4)	0.1561 (3)	0.1785 (1)	0.049 (1)

Table 2. *Geometric parameters* (Å, °)

O1—C1	1.37 (1)	C1—C4	1.38 (1)
O1—C2	1.45 (1)	C1—C8	1.40 (1)
O2—C3	1.45 (1)	C2—C3	1.48 (1)
O2—C4	1.40 (1)	C4—C5	1.41 (1)
N1—C5	1.44 (1)	C5—C6	1.37 (1)
N1—C9	1.46 (1)	C6—C7	1.38 (1)
N1—C12	1.48 (1)	C7—C8	1.34 (1)
N2—C10	1.49 (1)	C9—C10	1.50 (1)
N2—C11	1.50 (1)	C11—C12	1.51 (1)
C1—O1—C2	114.1 (7)	O2—C4—C5	117.6 (7)
C3—O2—C4	110.0 (7)	C1—C4—C5	120.3 (7)
C5—N1—C9	113.9 (8)	N1—C5—C4	117.4 (7)
C5—N1—C12	113.2 (6)	N1—C5—C6	124.2 (8)
C9—N1—C12	109.9 (8)	C4—C5—C6	118.4 (7)
C10—N2—C11	110.1 (6)	C5—C6—C7	120.4 (8)
O1—C1—C4	122.4 (7)	C6—C7—C8	122.0 (7)
O1—C1—C8	118.1 (7)	C1—C8—C7	119.3 (8)
C4—C1—C8	119.4 (8)	N1—C9—C10	111.3 (8)
O1—C2—C3	109.8 (8)	N2—C10—C9	111.1 (8)
O2—C3—C2	109.6 (7)	N2—C11—C12	109.7 (6)
O2—C4—C1	122.1 (7)	N1—C12—C11	109.6 (7)

Because of the poor crystallization habits of the title compound, the crystals investigated were of inferior quality. This accounts for the relatively large proportion of weak reflections (75%) and therefore is the source of the relatively high value of R_{int}. The absolute structure shown is arbitrary, as parallel refinement of the enantiomorphs resulted in insignificant differences in the R values.

We would like to thank Organon International bv for supplying the compound.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55463 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1011]

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(1*R*,2*R*)-1,2-Diphenyl-1,2-ethanediamine Monohydrobromide

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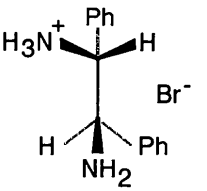
Abstract

The cation in (1*R*,2*R*)-1,2-diphenyl-1,2-ethanediamine monohydrobromide has approximate twofold symmetry and is involved in a three-dimensional hydrogen-bond network with the bromide anion [H···Br 2.51(4)-2.82(3), N···Br 3.279(2)-3.560(3) Å]. Principal bond lengths include Csp^3—Csp^3 1.535(4), Csp^3—NH_3^+ 1.489(3), Csp^3—NH_2 1.469(4), and Csp^3—C_{ar} 1.517(3) and 1.528(3) Å. The main torsion angles defining the conformation are NH_3^+—Csp^3—Csp^3—NH_2 −44.3(2) and C_{ar}—Csp^3—Csp^3—C_{ar} 64.3(2)°. The absolute stereochemistry (known on chemical grounds) was confirmed by the analysis.

Comment

(1*R*,2*R*)-1,2-Diphenyl-1,2-ethanediamine monohydrobromide is a versatile chiral auxiliary (Corey, Imwinkelreid, Pikul & Xiang, 1989) that has also been investigated as a chiral solvating agent in NMR spectroscopy (Fullwood & Parker, 1992). It forms 2:1 complexes with a wide range of chiral carboxylic acids (e.g. α-arylpropionic, α-halo- and α-deuteriocarboxylic acids) in which sufficient ^{1}H NMR chemical shift non-equivalence is observed to allow the direct measurement of the enantiomeric purity of the acid.

The structure of the monohydrobromide salt (I) was undertaken to establish the geometry of the quaternary ammonium cation and investigate the three-dimensional hydrogen-bonding network in the lattice. Suitable crystals were grown by slow evaporation of an isopropyl alcohol–isopropyl ether solution.



(I)

The absolute configuration of the cation was established by the analysis (see *Experimental*) and is in agreement with that deduced chemically. The cation (see scheme above and Fig. 1) has approximate twofold symmetry through the midpoint of the C(1)—C(2) bond and the conformation about the central C—C bond has the C—H H atoms fully staggered, with torsion angles N(1)—C(1)—C(2)—N(2) -44.3(2), C(11)—C(1)—C(2)—C(21) 64.3(2) and H(1)—C(1)—C(2)—H(2) −173(2)°.

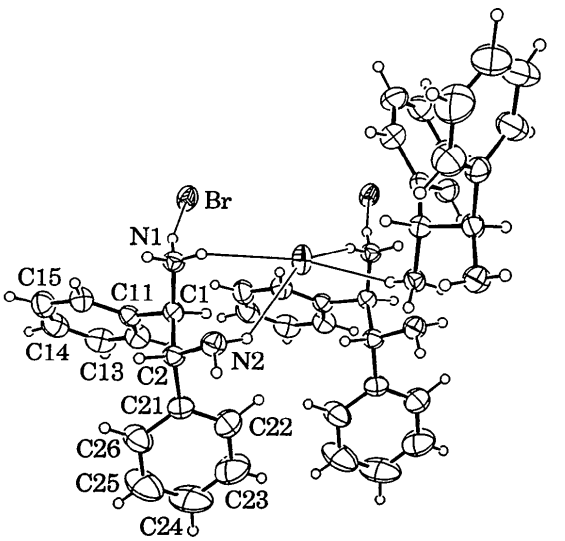


Fig. 1. A view of three formula units showing the general conformation, the intermolecular hydrogen bonding and the numbering scheme. The non-H atoms are shown with ellipsoids drawn at the 50% probability level. For clarity the H atoms are drawn as small spheres of an arbitrary size.

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