$w = 1/[\sigma^2(F_o^2) + (0.0891P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

N3A—C2A—C6A—C7A	142.6 (4)	N3B—C2B—C6B—C7B	137.2 (4)
O1A—C2A—C6A—C7A	−99.8 (5)	O1B—C2B—C6B—C7B	−105.0 (4)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N10A—H10A···O1A^{i}	0.86	2.36	3.051 (4)	138
N10B—H10B···O1B^{ii}	0.86	2.34	3.004 (4)	134
C15A—H15A···O1B^{iii}	0.93	2.70	3.599 (5)	163
C15B—H15B···O1A^{iv}	0.93	2.73	3.632 (5)	164

Symmetry codes: (i) $x-1, y, z$; (ii) $1+x, y, z$; (iii) $x, 1+y, 1+z$; (iv) $x, y, z-1$.

H atoms were treated as riding (N—H 0.86 and C—H 0.93–0.98 Å). At an intermediate stage in the analysis, the site occupancies of the atom pairs N10*A*/C7*A* and N10*B*/C7*B* were allowed to vary in order to check for possible N/C disorder; the occupancy factors obtained did not differ significantly from unity and therefore, in the final refinement cycles, no N/C disorder was allowed for. The anomalous dispersion terms for O, N, C are small and the absolute structure was not determined by our X-ray analysis. However, it can be inferred from the known absolute configuration of the (1*R*,2*S*)-(−)-ephedrine starting material used in the synthesis and the structure of a related thiazole derivative (Fitzsimons & Gallagher, 1999).

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4 and *CELDIM* in *CAD-4-PC Software*. Data reduction: *DATRD*2 in *NRCVAX*96 (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *NRCVAX*96 and *SHELXL*97 (Sheldrick, 1997*a*). Molecular graphics: *NRCVAX*96, *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 1998). Software used to prepare material for publication: *NRCVAX*96, *SHELXL*97 and *PREP*8 (Ferguson, 1998).

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X-ray investigations of potential β-blockers. IV

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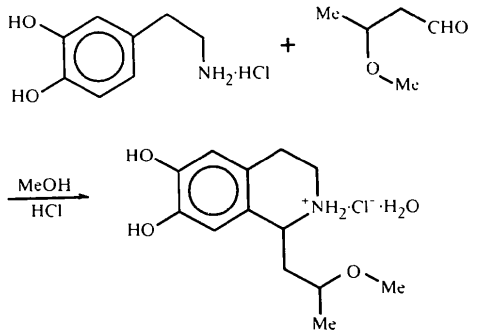
Abstract

X-ray studies of 6,7-dihydroxy-1-(2-methoxypropyl)-1,2,3,4-tetrahydroisoquinolinium chloride hydrate, $C_{13}H_{20}NO_3^+\cdot Cl^-\cdot H_2O$, show that the saturated part of the rings has a deformed half-chair conformation, with an axially attached 2-methoxypropyl group. The structure is ionic with a net of hydrogen bonds.

Comment

This paper is a continuation of the structural work on saturated isoquinoline derivatives (Olszak *et al.*, 1994, 1996; Olszak, 1998). Tetrahydroisoquinolines are known to exhibit β-adrenomimetic activity (Brzezińska, 1994). The group of 6,7-dihydroxy-1,2,3,4-tetrahydroisoquinolines substituted in position 1 has been synthesized in order to determine the role of the hydrophobic group at C1 on the activity of these derivatives as β-adrenoreceptors (Brzezińska *et al.*, 1996). The structure of one of these compounds, 6,7-dihydroxy-1-(2-methoxypropyl)-1,2,3,4-tetrahydroisoquinolinum chloride hydrate, (I), is presented here.



(I)

A perspective view of (I) showing the atomic numbering scheme is given in Fig. 1. The numbering scheme corresponds to those of previously published structures (Olszak, 1998). Bond lengths and angles, and details of the hydrogen-bonding geometry, are listed in Tables 1 and 2.

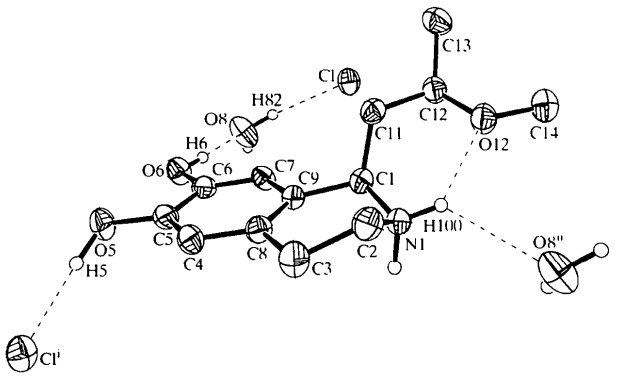


Fig. 1. The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as spheres of an arbitrary radius. Symmetry codes are as in Table 2.

The saturated part of (I) has a half-chair conformation. The puckering parameters (Cremer & Pople, 1975) are Q_T = 0.479 (3) Å, φ_2 = −29.5 (5)° and θ_2 = 46.5 (3)°, corresponding to the N1–C1–C9–C8–C3–C2 atom sequence, with a twofold pseudo-axis passing through the midpoints of the N1—C2 and C8—C9 bonds [asymmetry parameter (Nardelli, 1983*a*) Δ_2 = 0.004 (1)]. The aromatic part of the saturated isoquinoline moiety is planar. Saturation of the isoquinoline moiety does not influence the aromatic part of the molecule. Atoms C1 and C12 are chiral: C1 has an *R* configuration, while C12 has an *S* configuration. The Flack parameter (Flack, 1983) is 0.10 (2).

The 2-methoxypropyl moiety is attached axially, with torsion angles C2—N1—C1—C11 76.0 (3) and C11—C1—C9—C8 −105.0 (3)°. The unexpected axial configuration compared with previous studies (Olszak *et al.*, 1994, 1996; Olszak, 1998) seems to be stabilized by a strong intramolecular hydrogen bond, N1—H100···O12 (Table 2), which closes the six-membered ring in a deformed half-chair conformation with total puckering parameters Q_T = 0.515 (3) Å, φ_2 = 100 (1)° and θ_2 = 41 (2)°, corresponding to the N1–C1–C11–C12–O12–H100 atom sequence.

The structure is found to be ionic, with the proton of the hydrochloric acid attached to the N atom of the isoquinoline moiety. The molecules are linked by an intermolecular net of hydrogen bonds.

Apart from the axially attached moiety at position 1, previously studied structures (Olszak *et al.*, 1994, 1996; Olszak, 1998) do not show any significant differences from the title compound.

Experimental

The title compound was obtained by the Picket–Spengler synthesis of 2-(3,4-dihydroxyphenyl)-1-ethylamine hydrochloride with 3-methoxybutanal (Brzezińska *et al.*, 1996), and the structural formula was confirmed from ^{1}H NMR spectra.

Crystal data

$C_{13}H_{20}NO_3^+ \cdot Cl^- \cdot H_2O$	Cu $K\alpha$ radiation
M_r = 291.77	λ = 1.54178 Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	θ = 27.52–36.24°
a = 9.8412 (6) Å	μ = 2.436 mm^{-1}
b = 15.6366 (7) Å	T = 293 (2) K
c = 9.4044 (5) Å	Prism
V = 1447.2 (1) Å^3	0.20 × 0.15 × 0.10 mm
Z = 4	Colourless
D_x = 1.339 Mg m^{-3}	
D_m not measured	

Data collection

Rigaku AFC-5*S* diffractometer	1880 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.023
Absorption correction: analytical (de Meulenaer & Tompa, 1965)	θ_{max} = 60.05°
T_{min} = 0.646, T_{max} = 0.805	h = −11 → 11
	k = −17 → 17
	l = −10 → 10

8586 measured reflections
1263 independent reflections (plus 892 Friedel-related reflections)
3 standard reflections every 150 reflections
intensity decay: < 2.6%, corrected

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.080$
$S = 1.040$
2155 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.042$
$\Delta\rho_{max} = 0.193$ e Å^{-3}
$\Delta\rho_{min} = -0.171$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0004 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.10 (2)

Table 1. *Selected geometric parameters (Å, °)*

C9—C8	1.393 (3)	C1—C11	1.521 (3)
C9—C1	1.515 (3)	C11—C12	1.508 (4)
C8—C3	1.509 (3)	C12—O12	1.418 (3)
C3—C2	1.519 (4)	C12—C13	1.515 (4)
C2—N1	1.488 (3)	O12—C14	1.429 (3)
N1—C1	1.512 (3)		
C8—C9—C1	123.0 (2)	C9—C1—C11	111.9 (2)
C9—C8—C3	121.4 (2)	C12—C11—C1	116.1 (2)
C8—C3—C2	112.6 (2)	O12—C12—C11	107.3 (2)
N1—C2—C3	110.0 (2)	O12—C12—C13	113.1 (2)
C2—N1—C1	113.6 (2)	C11—C12—C13	109.4 (2)
N1—C1—C9	109.8 (2)	C12—O12—C14	113.0 (2)
N1—C1—C11	110.6 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···O8	0.78 (3)	1.88 (3)	2.647 (3)	168 (3)
N1—H100···O12	0.92 (3)	2.06 (3)	2.813 (3)	138 (2)
O8—H82···Cl	0.93 (3)	2.29 (3)	3.211 (2)	172 (3)
O5—H5···Cl^{i}	1.01 (4)	1.98 (4)	2.989 (2)	174 (3)
N1—H100···O8^{ii}	0.92 (3)	2.61 (3)	3.268 (3)	129 (2)

Symmetry codes: (i) $x-1, y, z$; (ii) $x, y, 1+z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEX* (McArdle, 1995). Software used to prepare material for publication: *PARST*97 (Nardelli, 1983*b*).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1043). Services for accessing these data are described at the back of the journal.

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(2*S*,3*S*)- and (2*R*,3*S*)-2-[2-(benzyloxy)ethyl]-3-(6-chloro-9*H*-purin-9-yl)oxolane†

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Abstract

The title diastereomeric compounds, $C_{18}H_{19}ClN_4O_2$, are the products formed when the (2*S*,3*S*)- and (2*R*,3*S*)-3-(5-amino-6-chloropyrimidinyl)amino-2-(2-benzyloxy)ethyloxolanes are treated with triethyl orthoformate in the presence of 4-toluenesulfonic acid. The crystal structure determination unambiguously shows the *cis* and *trans* orientations, respectively, of the 2-benzyloxyethyl and the 6-chloropurinyl substituents of the oxolanyl ring.

Comment

Lithium aluminium hydride reduction of (2*S*,3*S*)- and (2*R*,3*S*)-2-methoxycarbonyl-3-(tritylamino)oxolanes (Papaioannou *et al.*, 1991) followed by *O*-benzylation ($PhCH_2Br$/NaH) and detritylation with 4-toluenesulfonic acid, produced unexceptionally the corresponding (2*S*,3*S*)- and (2*R*,3*S*)-3-amino-2-(2-benzyloxy)ethyloxolanes (Papaioannou, 1998). Treatment of these amines with 4,6-dichloro-5-nitropyrimidine, followed by

† Alternative names: 9-{(2*S*,3*S*)- and (2*R*,3*S*)-2-[(2-benzyloxy)ethyl]-oxolan-3-yl}-6-chloro-9*H*-purine.