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Potential β-Blockers. I. 2-*tert*-Butyl-1,2,3,4-tetrahydro-4,6,8-trihydroxyisoquinolinium Sulfate Dihydrate

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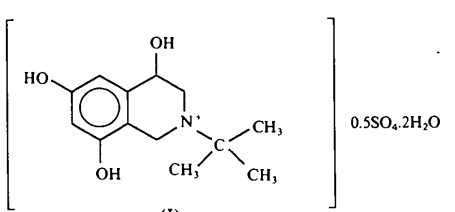
Abstract

The title molecule, $C_{13}H_{20}NO_3^+.0.5SO_4^{2-}.2H_2O$, consists of two six-membered condensed rings with the *tert*-butyl group in position 2. The aliphatic ring is in an envelope conformation. The five-atom planar part of the ring and the plane of the aromatic ring make an angle of 171.5 (1)° with respect to each other. The *tert*-butyl group is equatorial in relation to the ring. The sulfate ion lies on the twofold axis. The structure contains a three dimensional net of hydrogen bonds.

Comment

This paper commences reports on the structures of new derivatives of 1,2,3,4-tetrahydroisoquinoline-4,6,8-triol as potential β-blockers. The title compound (I) was synthesized in the Institute of Chemistry and Technology of Drugs, University of Medicine at Łódź by the simple Pictet–Spengler reaction of terbutaline, a well known β-adrenergic agonist, with formaldehyde (Brzezińska, 1994).



(I)

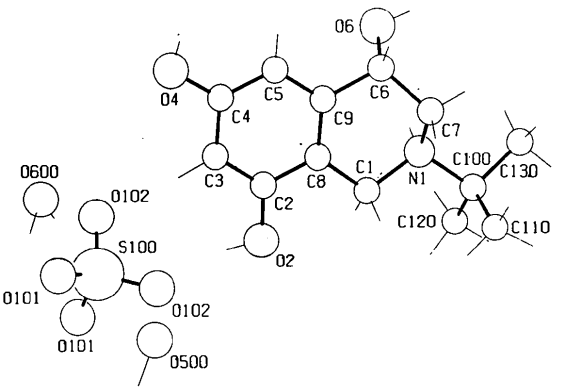


Fig. 1. View of the title compound with the atom-numbering scheme.

Experimental

Crystal data

$C_{13}H_{20}NO_3^+.0.5SO_4^{2-}.2H_2O$
M_r = 322.36
Orthorhombic
Pnca
a = 15.570 (3) Å
b = 16.579 (1) Å
c = 11.897 (1) Å
V = 3071.0 (7) Å^3
Z = 8
D_x = 1.3901 (3) Mg m^{-3}
D_m = 1.428 Mg m^{-3}
D_m measured by flotation
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 15–35°
μ = 1.508 mm^{-1}
Room temperature
Thick tabular
0.3 × 0.2 × 0.1 mm
Colourless
Crystal source: slow evaporation of water at room temperature

Data collection

Kuma K4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5706 measured reflections
2810 independent reflections
1871 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.028
θ_{max} = 80°
h = 0 → 19
k = −20 → 0
l = −15 → 13
2 standard reflections
frequency: 100 min
intensity variation: none

Refinement

Refinement on F
R = 0.0434
wR = 0.042
S = 1.08
1871 reflections
281 parameters
Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.079
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Atomic scattering factors from *CRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1986)

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

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

	x	y	z	U_{eq}
S100	3/4	1/2	0.0701 (1)	0.0459 (3)
O101	0.6917 (1)	0.5463 (1)	−0.0014 (2)	0.0669 (8)
O102	0.7000 (2)	0.4453 (1)	0.1406 (2)	0.0744 (8)

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N1	0.6543 (2)	0.3874 (2)	−0.2993 (2)	0.0444 (8)
C100	0.6588 (2)	0.3000 (2)	−0.2530 (2)	0.0516 (11)
C110	0.5755 (3)	0.2562 (3)	−0.2744 (5)	0.084 (2)
C120	0.7339 (3)	0.2580 (3)	−0.3105 (3)	0.0694 (15)
C130	0.6777 (3)	0.3049 (3)	−0.1273 (3)	0.0695 (16)
C1	0.6460 (3)	0.3912 (2)	−0.4253 (2)	0.0523 (11)
C8	0.6311 (2)	0.4749 (2)	−0.4681 (2)	0.0446 (10)
C9	0.6090 (2)	0.5393 (2)	−0.4007 (2)	0.0467 (19)
C6	0.5996 (2)	0.5263 (2)	−0.2757 (2)	0.0519 (11)
O6	0.6763 (2)	0.5575 (2)	−0.2251 (2)	0.0645 (19)
C7	0.5858 (2)	0.4392 (2)	−0.2473 (3)	0.0532 (12)
C2	0.6406 (2)	0.4879 (2)	−0.5834 (2)	0.0492 (10)
O2	0.6582 (2)	0.4215 (2)	−0.6469 (2)	0.0622 (19)
C3	0.6320 (2)	0.5635 (3)	−0.6280 (3)	0.0594 (13)
C4	0.6130 (2)	0.6278 (2)	−0.5596 (3)	0.0609 (12)
O4	0.6082 (2)	0.7020 (2)	−0.6080 (3)	0.0929 (13)
C5	0.5988 (2)	0.6158 (2)	−0.4454 (3)	0.0571 (12)
O500	0.5301 (2)	0.4330 (2)	0.0586 (2)	0.0822 (10)
O600	0.4121 (2)	0.1760 (2)	0.4543 (4)	0.1315 (17)

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

S100—O101	1.462 (2)	C100—C130	1.526 (4)
S100—O102	1.460 (3)	O500—H501	1.081 (4)
N1—C1	1.506 (3)	O6—O101	2.679 (3)
N1—C7	1.503 (5)	H600—O101	1.86 (4)
N1—C100	1.552 (5)	O500—H501	1.081 (4)
C1—C8	1.496 (5)	O500—O102	2.827 (4)
C2—C3	1.368 (6)	H501—O102	1.755 (4)
C2—C8	1.396 (3)	N1—H100	0.86 (3)
C2—O2	1.363 (4)	N1—O6^{i}	2.928 (4)
C3—C4	1.373 (6)	H100—O6^{i}	2.08 (3)
C4—C5	1.391 (5)	O4—H400	0.78 (4)
C4—O4	1.360 (5)	O4—O600^{ii}	2.745 (5)
C5—C9	1.384 (5)	H400—O600^{ii}	2.00 (4)
C6—C7	1.499 (5)	O2—H200	0.84 (5)
C6—C9	1.510 (3)	O2—O102^{iii}	2.640 (3)
C6—O6	1.434 (4)	H200—O102^{iii}	1.83 (5)
C8—C9	1.379 (4)	O600—H602	1.081 (5)
C100—C110	1.508 (6)	O600—O101^{iv}	2.748 (4)
C100—C120	1.523 (6)	H200—O102^{v}	3.32 (5)
O101—S100—O102	109.2 (1)	C6—C7—N1	110.9 (3)
O101—S100—O101^{i}	108.8 (1)	C1—C8—C2	117.5 (3)
O102—S100—O102^{i}	109.9 (1)	C1—C8—C9	124.0 (2)
O101—S100—O102^{i}	109.8 (1)	C2—C8—C9	118.6 (3)
O102—S100—O101^{i}	109.9 (1)	C5—C9—C6	119.9 (3)
C1—N1—C7	109.0 (3)	C5—C9—C8	121.0 (3)
C1—N1—C100	113.4 (3)	C6—C9—C8	119.1 (3)
C7—N1—C100	114.8 (3)	N1—C100—C110	110.6 (3)
N1—C1—C8	113.0 (2)	N1—C100—C120	107.6 (3)
C3—C2—C8	120.8 (3)	N1—C100—C130	107.9 (3)
C3—C2—O2	123.0 (3)	C110—C100—C120	111.4 (3)
C8—C2—O2	116.2 (3)	C110—C100—C130	110.9 (3)
C2—C3—C4	120.2 (3)	C120—C100—C130	108.4 (3)
C3—C4—C5	120.1 (3)	O6—H600—O101	165 (4)
C3—C4—O4	117.6 (3)	O500—H501—O102	170.8 (3)
C5—C4—O4	122.3 (3)	N1—H100—O6^{i}	168 (3)
C4—C5—C9	119.2 (3)	O4—H400—O600^{ii}	161 (4)
C7—C6—C9	111.9 (2)	O2—H200—O102^{iii}	163 (5)
C7—C6—O6	111.9 (2)	O600—H602—O101^{iv}	152.9 (4)
C9—C6—O6	106.3 (3)		
C1—C8—C2—O2	3.4 (4)	C100—N1—C7—C6	166.3 (3)
C1—C8—C2—C3	−176.9 (3)	O6—C6—C7—N1	−66.6 (3)
C1—C8—C9—C6	0.5 (5)	C7—N1—C1—C8	43.9 (4)
C1—C8—C9—C5	177.9 (3)	C7—N1—C100—C110	60.9 (4)
C8—C9—C6—C7	−20.1 (4)	C7—N1—C100—C120	−177.3 (3)
C8—C9—C6—O6	102.3 (3)	C7—N1—C100—C130	−60.5 (4)
C9—C6—C7—N1	52.7 (3)	N1—C1—C8—C9	−13.1 (5)
C1—N1—C7—C6	−65.4 (3)	N1—C1—C8—C2	166.7 (3)

Symmetry codes: (i) $1-x, 1-y, z$; (ii) $1-x, 1-y, -z$; (iii) $x, y, z-1$; (iv) $1-x, y, -z$; (v) $\frac{3}{2}-x, 1-y, z-1$.

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). The *E* map revealed the positions of all the non-H atoms. Refinement was by least squares using *SHELX76* (Sheldrick, 1976) of the *CRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1986) with isotropic and then anisotropic temperature factors. H atoms were located on a difference Fourier map. The H atoms of the water molecules (H501, H502, H601, H602) were refined as components of a rigid body.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71437 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1042]

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2*H*-1-Benzopyrans. I. (*E*)-4-Chloro-2-[dimethoxyphosphoryl(dimethoxyphosphoryloxy)methylene]-2*H*-1-benzopyran

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Abstract

The title molecule, $C_{14}H_{17}ClO_8P_2$, consists of two six-membered condensed rings, with a Cl atom at position 4 and a dimethoxyphosphoryl(dimethoxyphosphoryloxy)methylene group at position 2. Both rings are aromatic. The best planes of the two rings

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