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(*R*)-(+)-(1-Hydroxy-3-phenylpropan-2-yl)-trimethylammonium bromide sesquihydrate

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Key indicators

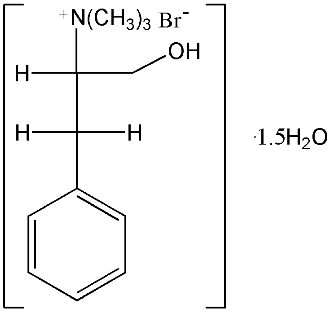
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.008 Å
Disorder in solvent or counterion
R factor = 0.040
wR factor = 0.102
Data-to-parameter ratio = 15.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{12}H_{20}NO^+\cdot Br^-\cdot 1.5H_2O$, two crystallographically independent quaternary ammonium cations are associated by three very short C—H⋯π interactions with distances between the H atoms and the centroids of the benzene rings of 2.787 (8), 2.878 (7) and 2.932 (8) Å. A one-dimensional hydrogen-bonded spiral is formed along the *b* axis by O—H⋯Br hydrogen bonds. Neighboring spirals are interrelated by translation and held together mainly by hydrogen bonds and O—H⋯O interactions. C—H⋯O and C—H⋯Br hydrogen bonds and C—H⋯π interactions play an important role in stabilizing the structure.

Comment

Nishio and co-workers first pointed out the nature of C—H⋯π interactions (Kodama *et al.*, 1976). Over the last two decades, the role of this interaction in crystal packing and molecular recognition has been constantly investigated by X-ray diffraction (Kodama *et al.*, 1976; Vyas *et al.*, 1988; Quicho *et al.*, 1989; Tsuzuki *et al.*, 1999; Kim *et al.*, 2000; Novoa & Mota, 2000; Tsuzuki *et al.*, 2000). We have synthesized the title compound, (I), which contains three short C—H⋯π interactions, and report the structure here.



(I)

The asymmetric unit of (I) consists of two (*R*)-(+)-(1-hydroxy-3-phenylpropan-2-yl)trimethylammonium cations, two bromide anions and three water molecules (Fig. 1). The benzene rings of the cations make a dihedral angle of 86.9 (3)°. There are three C—H⋯π interactions involving the cations (Table 2).

Water molecule O3 links O4 and O5 *via* O3—H3*A*⋯O4 and O3—H3*B*⋯O5 hydrogen bonds, and they, in turn, link with the anions *via* O5—H5*A*⋯Br2 and O4—H4″*A*⋯Br1^{ii} (symmetry code as in Table 2), forming a hydrogen-bonded spiral (Fig. 2 and Table 2). The screw-pitch of the spiral is equal to the length of the *b* axis. Neighboring spirals are connected mainly by hydrogen bonds of types O4—H4*B*⋯O5^{i} and O5—H5*B*⋯O4^{iii} (symmetry codes as in

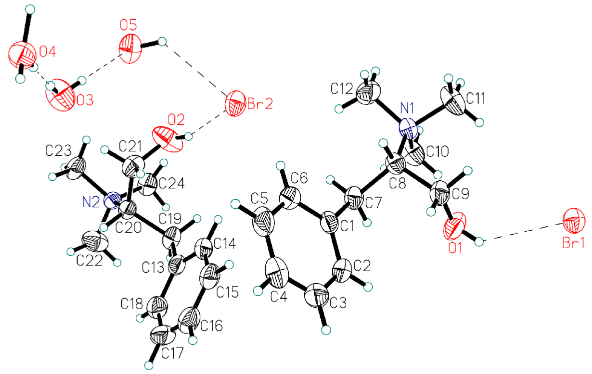


Figure 1
The asymmetric unit of (I) with the atom labels, showing 40% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

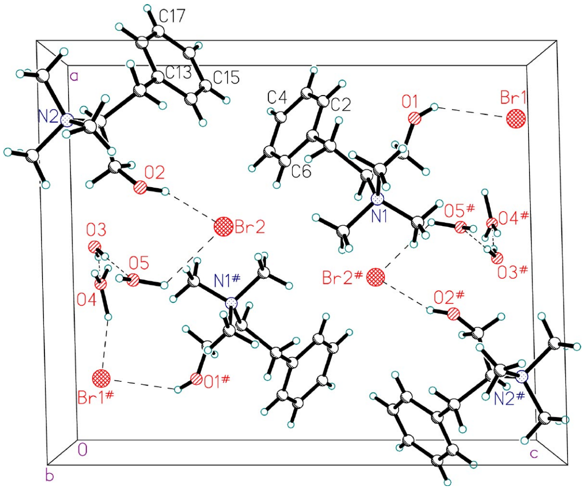


Figure 2
Two hydrogen-bonded spirals viewed along the *b* axis. Hydrogen bonds are shown as dashed lines, and a hash (#) indicates symmetry code $(1-x, y+\frac{1}{2}, 1-z)$.

Table 2) to establish the whole crystal structure. C—H···O and C—H···Br hydrogen bonds (Table 2) and C—H···π interactions play an important role in stabilizing the structure. C24—H24*C*···O2 is the shortest of its type.

Experimental

Treatment of l-phenylalaninol, prepared by $LiAlH_4$ reduction of *N*-ethyl-l-phenylalanine in 80% yield, with formaldehyde and formic acid, according to a procedure reported by Tseng *et al.* (1977), gave *N*,*N*-dimethyl-L-phenylalaninol in 88% yield. *N*,*N*-Dimethyl-L-phenylalaninol (10 mmol, 1.67 g) in acetonitrile (100 ml) with MeBr (15 mmol, 1.42 g) gave (I) (7.7 mmol, 2.02 g) in 77% yield. Recrystallization of the synthesized product from water, at 293 K, gave single crystals of (I) (m.p. 469–471 K); $[\alpha]_D^{20}$ = +8.80 (*c* = 1.0, CH_3OH); ^1H NMR (300 MHz, D_2O, p.p.m.): 2.86 (*dd*, *J* = 8.5, 13.4 Hz, 1H, 3H), 3.02 (*dd*, *J* = 5.3, 13.4 Hz, 1H, 3H), 3.20 [*s*, 9H, $N(CH_3)_3$], 3.25–3.33 (*m*, 1H, 2H), 3.62 (*dd*, *J* = 7.2, −10.6 Hz, 1H, 1H), 3.98 (*dd*, *J* = 4.0, −10.6 Hz, 1H, 1H), 7.30–7.39 (*m*, 5H, PhH); IR (KBr, cm^{-1}): 3320, 3021, 2942, 2890, 1643, 1483, 1255, 1091, 1049, 959, 754, 703.

Crystal data

$C_{12}H_{20}NO^+\cdot Br^-\cdot 1.5H_2O$
M_r = 301.225
Monoclinic, $P2_1$
a = 13.2146 (17) Å
b = 6.9739 (9) Å
c = 16.210 (2) Å
β = 91.490 (2)°
V = 1493.3 (3) Å^3
Z = 4

D_x = 1.340 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 1820 reflections
θ = 5.2–51.7°
μ = 2.75 mm^{-1}
T = 273 (2) K
Rod, colorless
0.29 × 0.26 × 0.22 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.471, T_{max} = 0.547
7855 measured reflections

4850 independent reflections
3769 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.2°
$h = -15 \rightarrow 15$
$k = -8 \rightarrow 8$
$l = -19 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.102
S = 0.99
4850 reflections
305 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.07P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack (1983), 1925 Friedel pairs
Flack parameter = 0.018 (12)

Table 1
Selected bond lengths (Å).

O1—C9	1.399 (6)	C1—C7	1.495 (7)
N1—C11	1.483 (6)	C3—C4	1.358 (8)
N1—C10	1.494 (7)	C4—C5	1.382 (8)
N1—C12	1.507 (6)	C5—C6	1.374 (9)
N1—C8	1.542 (7)	C7—C8	1.532 (7)
C1—C2	1.376 (7)	C8—C9	1.516 (7)
C1—C6	1.385 (6)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Br1	0.82	2.65	3.226 (4)	128
O2—H2···Br2	0.82	2.44	3.231 (5)	162
O3—H3*A*···O4	0.93	1.91	2.829 (6)	172
O3—H3*B*···O5	0.86	1.91	2.770 (7)	172
O4—H4*B*···O5^i	0.94	2.15	2.830 (6)	129
O4—H4″*A*···Br1^{ii}	1.18	2.13	3.270 (4)	161
O5—H5*A*···Br2	0.98	2.72	3.343 (4)	122
O5—H5*B*···O4^{iii}	0.92	1.94	2.830 (6)	161
C7—H7*B*···O1	0.97	2.48	2.868 (6)	104
C10—H10*A*···O1	0.96	2.47	3.083 (8)	122
C12—H12*C*···Br2	0.96	2.94	3.779 (6)	146
C12—H12*A*···Br2^{iv}	0.96	2.91	3.840 (7)	163
C16—H16···O1^v	0.93	2.56	3.313 (8)	138
C19—H19*B*···O2	0.97	2.57	2.970 (7)	105
C22—H22*A*···Br1^{vi}	0.96	2.93	3.844 (7)	160
C24—H24*C*···O2	0.96	2.32	3.037 (8)	131

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

Water H atoms were located by difference Fourier methods and refined with isotropic displacement parameters 1.2 times U_{eq} of the parent atoms. Other H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.82–0.97 Å, with isotropic displacement parameters 1.2–1.5 times U_{eq} of the parent atoms. The disordered H atom attached to O4 was split over two sites (H4′*A* and H4″*A*) with occupancies of 0.5. The other H atom of the water molecules was ordered and was treated normally.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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