Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-{[(2-Hydroxyphenyl)methyl]methylamino}-1-phenylpropan-1-ol

Fang Yuan,[a] Yi-Zhi Li,[b] Cheng-Jian Zhu,[a] Yi Pan[a]* and Lin-Lin Xu[a]

[a]Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China, and [b]Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

Correspondence e-mail: llyyjz@nju.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.074
Data-to-parameter ratio = 14.0

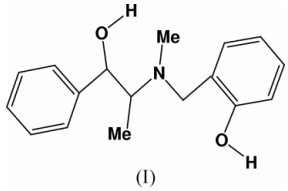
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 March 2003
Accepted 1 April 2003
Online 9 April 2003

In the title compound, $C_{17}H_{21}NO_2$, there are two chiral C atoms. There are two intramolecular hydrogen bonds and one intermolecular hydrogen bond. The latter intermolecular hydrogen bond links the molecules in a helical fashion along the *b*-axis direction.

Comment

Ephedrine and its derivatives have been studied for a long time and the preparation of *N*-substituted ephedrines is common (Neelakantan, 1971). They are inexpensive and readily available in enantiomerically pure form. Therefore, with the development of stereochemistry and organometallic chemistry, they have become more and more important as chiral ligands (Kuznetsov *et al.*, 1999). Recently, the focus of our group has been on organogallium complexes with chiral ligands, such as ephedrine and its derivatives. We report here the structure and relative stereochemistry of the title compound, (I), which resulted from the condensation of (−)-ephedrine with salicylaldehyde.



(I)

The molecule looks like a camber, with the two aromatic rings located at opposite ends of the C1—C2—N1—C10 camber chain. The dihedral angle between the mean planes through the aromatic rings is 39.42 (7)°. There are two chiral C atoms (C1 and C2) in the molecule which are of opposite hands (1*S*, 2*R* or 1*R*, 2*S*). Two intramolecular hydrogen bonds (O2—H2*A*···N1 and C17—H17*A*···O1) and one intermolecular hydrogen bond (O1—H1*A*···O2 at $-x+2$, $y-\frac{1}{2}$, $-z+\frac{1}{2}$) are present. The intermolecular hydrogen bond links the molecules into a one-dimensional helical chain (see Fig. 2).

Experimental

(1*R*,2*S*)-(−)-Ephedrine (2.5 g, 15 mmol) and salicylaldehyde (1.6 ml, 15 mmol) were heated under reflux in benzene (20 ml) for 1 h. The calculated amount of water was then removed. The excess of benzene was abstracted by a rotary evaporator and the residue recrystallized from alcohol to give the oxazolidine, a white needle-shaped crystal. To one molar equivalent of $LiAlH_4$ (0.5 g, 13.2 mmol) in boiling dry dioxane (20 ml), a dioxane (15 ml) solution of one mole of the oxazolidine (1.5 g, 5.54 mmol) was slowly added, and the mixture refluxed overnight. After cooling, ice and an excess of 10% sodium hydroxide solution were added, the resulting product extracted with benzene and the extract was washed with water till neutral, dried and

concentrated to obtain the title compound as an oil. Crystals of (I) were obtained by recrystallization from CH_2Cl_2/hexane.

Crystal data

$C_{17}H_{21}NO_2$
$M_r = 271.35$
Orthorhombic, $P2_12_12_1$
$a = 6.004\ (1)$ Å
$b = 10.451\ (1)$ Å
$c = 23.790\ (3)$ Å
$V = 1492.8\ (3)$ Å^3
$Z = 4$
$D_x = 1.207$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3564 reflections
$\theta = 2.6$–$24.3°$
$\mu = 0.08$ mm^{-1}
$T = 293\ (2)$ K
Block, colorless
$0.3 \times 0.2 \times 0.2$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*XPREP*; Bruker, 2000)
$T_{min} = 0.98$, $T_{max} = 0.99$
7631 measured reflections
2605 independent reflections
1908 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$
$\theta_{max} = 25.0°$
$h = -7 \rightarrow 7$
$k = -9 \rightarrow 12$
$l = -26 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.074$
$S = 1.08$
2605 reflections
186 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.003P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.034 (2)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.82	2.00	2.8165 (19)	176
O2—H2A···N1	0.82	1.84	2.581 (2)	150
C17—H17A···O1	0.96	2.55	3.156 (2)	121

Symmetry code: (i) $2 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

The positions of all H atoms were fixed geometrically and refined isotropically using a riding model. The bond lengths for C—H and O—H are in the range 0.82--0.98 Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); 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*.

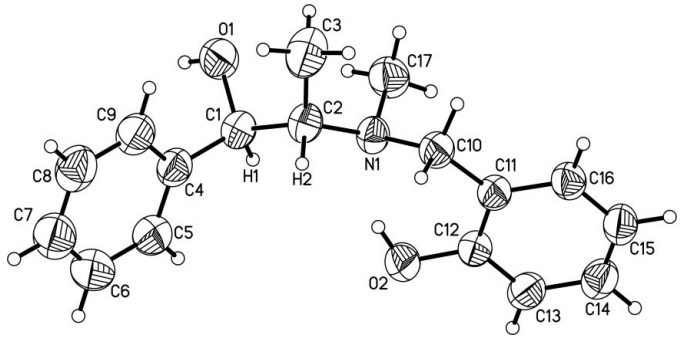


Figure 1
The title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

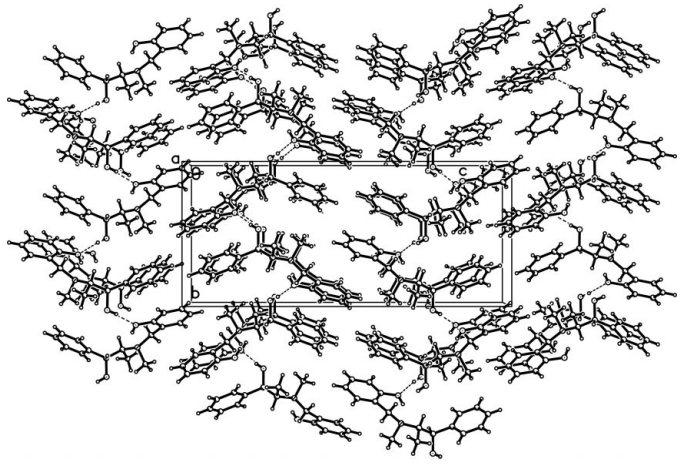


Figure 2
Crystal packing of the title compound, viewed down the *a* axis.

This work was supported by the Nanjing University Talent Development Foundation (No. 0205005122).

References

Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.22), *SHELXTL* (Version 6.10) and *XPREP*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kuznetsov, V. F., Facey, G.-A., Yap, G. P.-A. & Alper, H. (1999). *Organometallics*, **18**, 4706–4711.
Neelakantan, L. (1971). *J. Org. Chem.* **36**, 2256–2260.