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α-[(Diphenylmethoxy)methyl]-2,5-dimethyl-1-pyrrolidineethanol

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.116
Data-to-parameter ratio = 12.6

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

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The conformation of the pyrrolidine ring in the title compound, $C_{22}H_{29}NO_2$, is close to an envelope. The phenyl rings are almost perpendicular to one another. The molecules are connected into centrosymmetric dimers by O—H···N hydrogen bonds.

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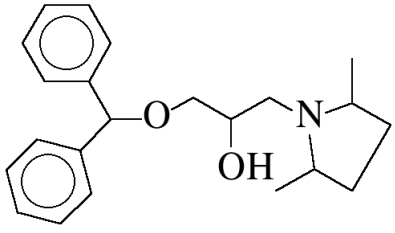
Comment

The title compound, (I), was tested as a potential anti-arrythmic agent (Hoefle *et al.*, 1991*b*). Its activity was only medium, and due to a number of side effects it was not studied further. Better activity was provided by piperidine derivatives, especially by 2,6-dimethyl α,α-diaryl-1-piperidinebutanols (Hoefle *et al.*, 1991*a*).



(I)

The conformation of the pyrrolidine ring is close to an envelope; four atoms (C2′, C3′, C4′ and C5′) are approximately coplanar [maximum deviation from the least-squares plane is 0.028 (2) Å] and N1′ is 0.611 (2) Å out of this plane. The asymmetry parameter ΔC_s (Duax & Norton, 1975) is relatively large, 6.1°. The conformation of the O1—C1—C3—O3 group is *anti* [the torsion angle is 178.07 (9)°]. The same conformation was observed in α-{[(2-methylphenyl)phenylmethoxy]methyl}-2,6-dimethyl-1-piperidineethanol, while in the molecule with four methyl substituents, α-[(bis(2,6-dimethylphenyl)methoxy)methyl}-2,6-dimethyl-1-piperidineethanol, the mutual disposition of both O atoms was *gauche* [O—C—C—O torsion angle is 64.8 (4)°; Kubicki & Codding, 2001]. The phenyl rings are almost perpendicular to one another; the torsion angle beteween their least-squares planes is 82.85 (4)°. The disposition of the phenyl rings with respect to the C3—O3—C4 plane follows the trends found in similar compounds without significant steric stress: one ring is almost parallel to the C—O—C plane [dihedral angle is 7.53 (13)°], and the other is almost perpendicular [89.63 (7)°]. In a bis(2,6-dimethyl)phenyl derivative both rings are almost perpendicular to the C—O—C plane (Kubicki & Codding, 2001).

The molecules are connected into centrosymmetric dimers by means of the O—H···N hydrogen bonds (Fig. 2), and weak

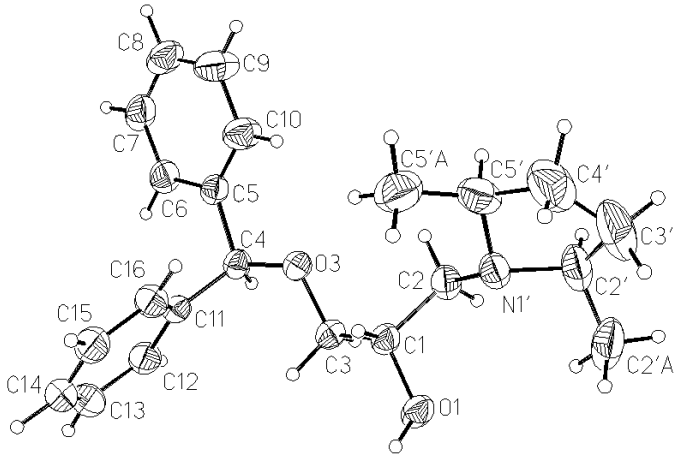


Figure 1
A perspective view of the title compound together with the atom-numbering scheme (Siemens, 1989). Displacement ellipsoids are drawn at the 50% probabilty level and H atoms are shown as spheres with arbitrary radii.

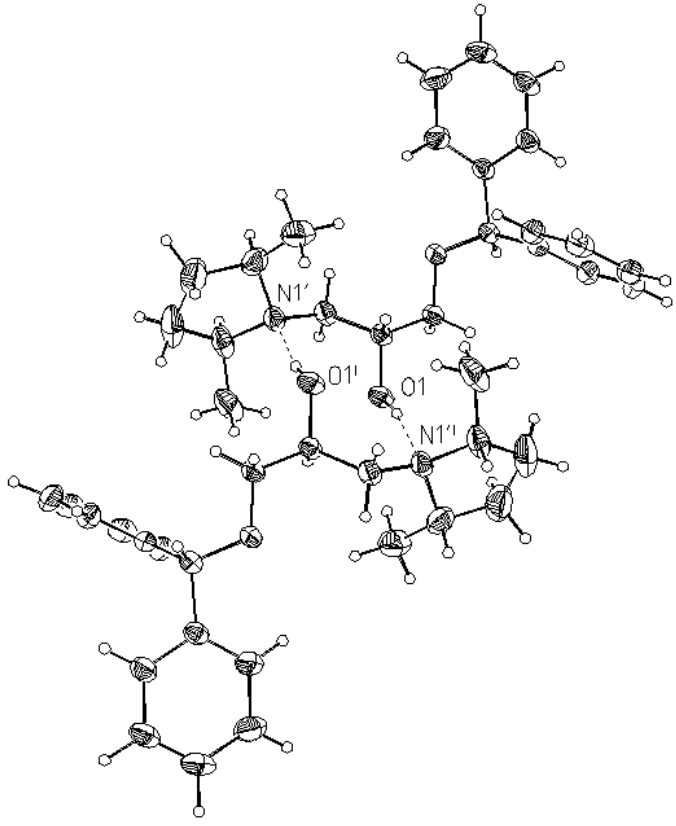


Figure 2
The centrosymmetric hydrogen-bonded dimer (Siemens, 1989). Hydrogen bonds are drawn as dashed lines. [Symmetry code: (i) $-x$, $1-y$, $1-z$.]

C—H···O bonds connect these dimers into infinite chains along the [100] direction.

Experimental

The sample was provided by Parke—Davis Pharmaceutical Research Division, Warner–Lambert Company, Ann Arbor, MI, USA. Colourless crystals were grown from the methanol solution by slow evaporation.

Crystal data

$C_{22}H_{29}NO_2$	$Z = 2$
$M_r = 339.46$	$D_x = 1.102$ Mg m^{-3}
Triclinic, $P\overline{1}$	Cu $K\alpha$ radiation
$a = 8.3588$ (4) Å	Cell parameters from 25 reflections
$b = 10.6774$ (4) Å	$\theta = 15$–$38°$
$c = 12.5709$ (5) Å	$\mu = 0.54$ mm^{-1}
$\alpha = 99.033$ (3)°	$T = 293$ (2) K
$\beta = 108.701$ (4)°	Block, colourless
$\gamma = 99.057$ (3)°	0.25 × 0.20 × 0.10 mm
$V = 1023.33$ (7) Å^3	

Data collection

CAD-4*F* four-circle diffractometer	$h = -10 \rightarrow 9$
$\omega/2\theta$ scans	$k = -13 \rightarrow 13$
4207 measured reflections	$l = 0 \rightarrow 15$
4207 independent reflections	2 standard reflections frequency: 33 min intensity decay: 2%
3569 reflections with $I > 2\sigma(I)$	
$\theta_{max} = 74.9°$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 0.1008P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.043$	
$wR(F^2) = 0.116$	
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.001$
4207 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
333 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
H atoms: see below	Extinction correction: *SHELXL*97 Extinction coefficient: 0.0034 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1′^{i}	0.98 (2)	1.91 (2)	2.870 (1)	167 (2)
C15—H15···O1^{ii}	0.95 (2)	2.49 (2)	3.400 (2)	161 (2)

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

Positional parameters of all H atoms were refined. The isotropic displacement parameters of the H atoms connected to the C2′, C3′, C4′ and C5′ atoms were set at 1.3 times U_{iso} of the appropriate carrier atoms, and those of the remaining H atoms were refined freely.

Data collection and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989); data reduction: *ENPROC* (Rettig, 1978); structure solution: *SHELXS*97 (Sheldrick, 1990); structure refinement: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989).

References

Duax, W. L. & Norton, D. A. (1975). In *Atlas of Steroid Structure*, Vol. 1. Plenum: New York.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Entraf–Nonius, Delft, The Netherlands.
Hoefle, M., Blouin, L. T., Fleming, R. W., Hastings, S., Hinkley, J. M., Mertz, T. E., Steffe, T. J. & Stratton, C. S. (1991*a*). *J. Med. Chem.* **34**, 12–19.
Hoefle, M. L., Blouin, L. T., Fleming, R. W., Hastings, S., Hinkley, J. M, Mertz, T. E., Steffe, T. J., Stratton, C. S. & Werbel, L. M. (1991*b*). *J. Med. Chem.* **34**, 7–12.
Kubicki, M. & Codding, P. W. (2001). *Pol. J. Chem.* Submitted.
Rettig, S. (1978). *ENPROC. Data Reduction Program for Enraf–Nonius CAD-4F Diffractometer*. University of British Columbia, Vancouver, BC, Canada.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1989). *Stereochemical Workstation Operation Manual*. Release 3.4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.