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(5*R*,6*R*)-Methyl 6-methyl-2-phenyl-5,6-dihydro-4*H*-1,3-oxazine-5-carboxylate

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The title compound, $C_{13}H_{15}NO_3$, contains a chiral *cis*-disubstituted oxazine linked to a phenyl ring at the C-2 atom. The racemic crystal structure is stabilized by both intra- and intermolecular C—H···O hydrogen bonds.

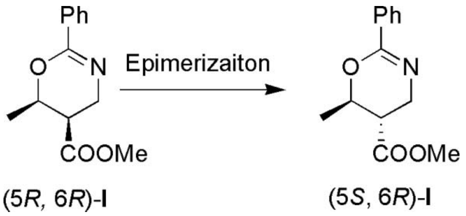
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.137
Data-to-parameter ratio = 15.0

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

Comment

This class of oxazine derivatives has been reported in the preparation of (3*R*,4*R*)-4-acetoxy-3-{(*R*)-1′-[(*t*-butyldimethylsilyl)oxy]ethyl}-2-azetidinone, which is a versatile intermediate for the synthesis of carbapenems (Fuganti *et al.*, 1994). The epimerization of *cis*-disubstituted oxazine provided an attractive methodology for changing the stereochemistry of a compound under mild conditions (Schneider *et al.*, 1990). Establishment of the three-dimensional structure could help us to further investigate the mechanism of this conversion. Here we report the crystal structure of the title compound, (I).



The molecular structure of (I) is shown in Fig. 1. Atoms O(1), C(2), N(3) and C(4) are almost coplanar with the phenyl ring, probably because of conjugation of the electron lone pair of the O atom and the π orbitals of the phenyl ring and imine double bond. The *cis*-disubstituted oxazine ring is slightly distorted and adopts a half-chair conformation, with the methoxycarbonyl group equatorial and the methyl group axial, with a torsion angle of 62.14 (18)° for C(13)—C(5)—C(6)—C(15). Although there is no –NH or –OH group available in the structure to form strong hydrogen bonds, the C atoms are involved in the formation of non-classical hydrogen bonds (Table 1).

Experimental

Compound (I) was synthesized by the treatment of (2*S*,3*S*)-2-acetylaminomethyl-3-hydroxybutyric acid methyl ester with thionyl chloride, as described by Schneider *et al.* (1990). The crude product was dissolved in hot methanol. Single crystals of (I) suitable for X-ray diffraction measurements were obtained by slow evaporation at room temperature (m.p. 360–361 K).

Crystal data

$C_{13}H_{15}NO_3$
$M_r = 233.26$
Monoclinic, $P2_1/c$
$a = 8.095\ (4)$ Å
$b = 6.318\ (3)$ Å
$c = 23.940\ (10)$ Å
$\beta = 101.501\ (13)°$

$V = 1199.9\ (9)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 293\ (2)$ K
$0.25 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.977$, $T_{\max} = 0.991$

5176 measured reflections
2337 independent reflections
1714 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.137$
$S = 1.04$
2337 reflections

156 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C(15)—H(15*B*)···O(2)	0.96	2.74	3.274 (3)	116
C(6)—H(6)···O(3)$^{\rm i}$	0.98	2.83	3.596 (2)	136
C(10)—H(10)···O(2)$^{\rm ii}$	0.93	2.91	3.513 (3)	124
C(14)—H(14*A*)···O(2)$^{\rm iii}$	0.96	2.76	3.477 (3)	132

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

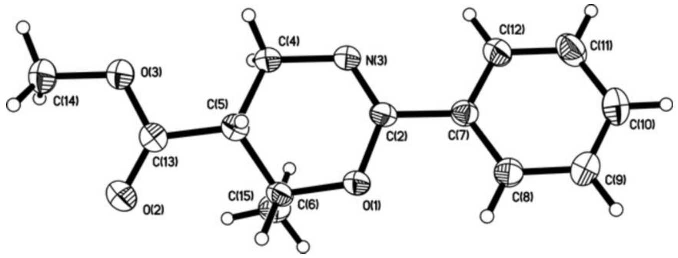


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å, and with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $1.2U_{\rm eq}({\rm C})$ for other H atoms. Methyl groups were allowed to rotate freely about the C—C bond.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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o2366 Dai *et al.* • $C_{13}H_{15}NO_3$ *Acta Cryst.* (2007). E**63**, o2365–o2366