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(1*S*,4*R*)-*N*-[(*S*)-2-Hydroxy-1-phenylethyl]-7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-carboxamide

Yi-Min Cai, Hua Fang, Qing-Le Zeng* and Yu-Fen Zhao

The Key Laboratory for Chemical Biology of Fujian Province, Department of Chemistry, Xiamen University, Xiamen 361005, People's Republic of China

Correspondence e-mail: qlzeng@xmu.edu.cn

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.116
Data-to-parameter ratio = 8.3

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

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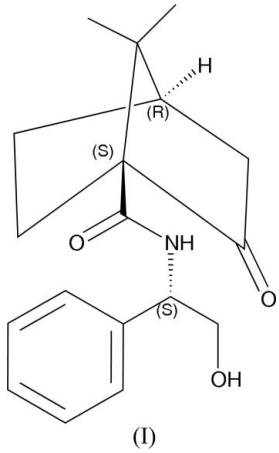
The title compound, $C_{18}H_{23}NO_3$, has been obtained by the reaction of (1*S*,4*R*)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-carbonyl chloride with (*S*)-2-amino-3-phenylpropan-1-ol. The carbonyl functionality of the carboxamide group and the hydroxy group are involved in O—H···O intermolecular hydrogen bonds.

Comment

During our continuing study of asymmetric synthesis, we have found a novel and useful chiral ligand, namely (1*R*,2*R*,4*R*)-7,7-dimethyl-1-[(*S*)-4-phenyl-4,5-dihydrooxazol-2-yl]bicyclo[2.2.1]heptan-2-ol, which may be derived from inexpensive D-camphor (Zeng, Liu, Cui *et al.*, 2002; Zeng, Liu, Mi *et al.*, 2002). The title compound, (I), is a key intermediate in the synthesis of this ligand, and has been obtained by the reaction of (1*S*,4*R*)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-carbonyl chloride and (*S*)-2-amino-2-phenylethanol (see *Experimental*). Bond lengths and angles in (I) (Fig. 1 and Table 1) are in agreement with values reported for a similar compound (Lalancette *et al.*, 1999). The carbonyl group C17═O3 and hydroxyl group O2—H2*D* are involved in intermolecular hydrogen bonds (Fig. 2 and Table 2).



(I)

Experimental

To a solution of (1*S*,4*R*)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-carbonyl chloride (0.4 g, 2 mmol) in dichloromethane (25 ml) cooled in an ice–water bath, was added, in small portions, a solution of (*S*)-2-amino-3-phenylpropan-1-ol (0.32 g, 2 mmol) and triethylamine (0.4 ml) in dichloromethane (10 ml) (Zeng, Liu, Cui *et al.*, 2002; Zeng, Liu, Mi *et al.*, 2002). The resulting solution was stirred at 298 K for 22 h. Water (10 ml) was then added to the mixture in order to quench

the reaction. The organic layer was separated and the aqueous layer was extracted with dichloromethane. The organic layers were combined, dried over anhydrous magnesium sulfate and filtered. The solvent was removed under reduced pressure, giving 0.51 g of a colourless liquid (yield: 81.0%). Compound (I) was crystallized from the crude product by slow evaporation of an ethyl acetate–dichloromethane (2:1) solution.

Crystal data

$C_{18}H_{23}NO_3$
M_r = 301.37
Orthorhombic, $P2_12_12_1$
a = 10.117 (5) Å
b = 10.401 (5) Å
c = 13.934 (7) Å
V = 1466.3 (12) Å^3
Z = 4
D_x = 1.365 Mg m^{-3}
Mo- $K\alpha$ radiation
Cell parameters from 3471 reflections
θ = 2.4–27.9°
μ = 0.09 mm^{-1}
T = 273 (2) K
Irregular fragment, colourless
0.20 × 0.20 × 0.12 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.982, T_{max} = 0.989
4877 measured reflections
1644 independent reflections
1491 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 26.0°
$h = -6 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.116
S = 1.23
1644 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.1311P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C17	1.300 (4)	O2—C6	1.375 (4)
N1—C11	1.430 (3)	O3—C17	1.202 (4)
O1—C13	1.181 (4)		
C17—N1—C11	122.2 (2)	O1—C13—C14	127.9 (3)
N1—C11—C18	111.8 (2)	O3—C17—N1	122.4 (3)
N1—C11—C6	109.3 (2)	O3—C17—C14	120.1 (3)
O1—C13—C5	126.0 (3)	N1—C17—C14	117.4 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2D···O3^{i}	0.82	2.03	2.847 (3)	173

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

H atoms were placed in idealized positions (C—H = 0.93, 0.98, 0.97 and 0.96 Å for phenyl, methine, methylene and methyl H atoms, respectively, O—H = 0.82 Å and N—H = 0.86 Å) and were included in the refinement in the riding-model approximation. Isotropic displacement parameters were set at 1.5U_{eq}(carrier atom) for methyl H atoms and 1.2 U_{eq}(carrier atom) for other H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration of (I) was assigned assuming that the absolute configurations of the starting materials were retained during the synthesis.

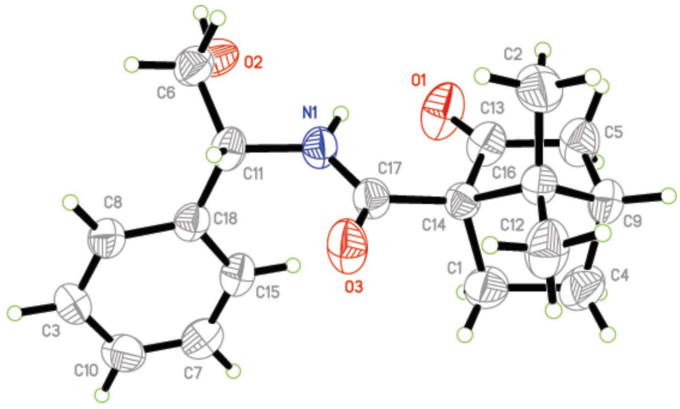


Figure 1
ORTEP3 (Farrugia, 1997) plot of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

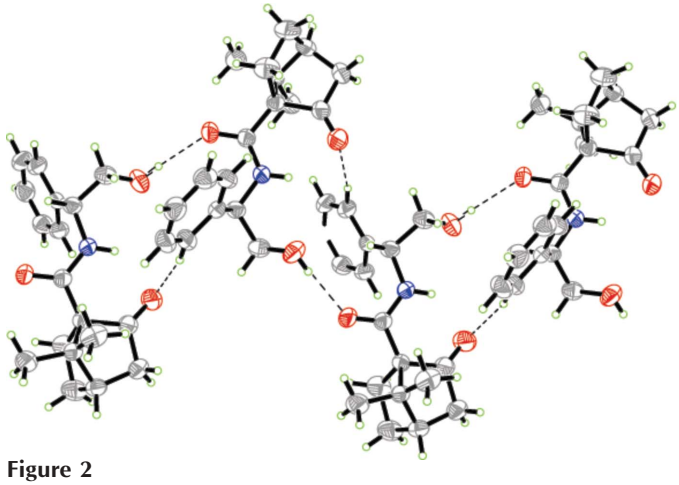


Figure 2
Part of the packing of (I). Intermolecular hydrogen bonds are represented by dashed lines.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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References

Bruker (2001). *SAINT* (Version 6.22), *SMART* (Version 5.625) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Lalancette, R. A., Coté, M. L., Smith, W. J. III, Thompson, H. W., Vanderhoff, P. A., Brunskill, A. P. J., Campana, C., Burshtein, I. & Rose, J. P. (1999). *Acta Cryst.* C**55**, 1600–1605.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zeng, Q., Liu, H., Cui, X., Mi, A., Jiang, Y., Li, X. S., Choi, M. C. K. & Chan, A. S. C. (2002). *Tetrahedron Asymmetry*, **13**, 115–117.
Zeng, Q., Liu, H., Mi, A., Jiang, Y., Li, X. S., Choi, M. C. K. & Chan, A. S. C. (2002). *Tetrahedron*, **58**, 8799–8803.