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(4*S*)-Benzyl 4-benzyl-5-oxo-1,3-oxazolidine-3-carboxylate

Xing-Hua Zhang, Kan Lin and Yu-Fen Zhao*

Key Laboratory for Chemical Biology of Fujian Province, Department of Chemistry, Xiamen University, Xiamen 361005, People's Republic of China
Correspondence e-mail: t12g21@xmu.edu.cn

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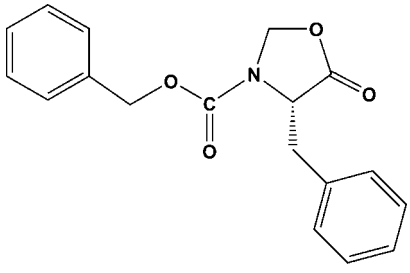
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.031; wR factor = 0.072; data-to-parameter ratio = 8.2.

In the title compound, $C_{18}H_{17}NO_4$, the carboxylate group is approximately coplanar with the oxazolidine ring, the largest deviation from the least-squares plane being 0.144 (2) Å at the nitrogen atom. The two benzyl rings are located on the same side of this plane, and make dihedral angles with it of 75.23 (4) and 56.97 (7)°.

Related literature

For related literature, see: Vidyasagar Reddy *et al.* (2000); Lin *et al.* (2007); Dorow & Gingrich (1999).



Experimental

Crystal data

$C_{18}H_{17}NO_4$
M_r = 311.33
Monoclinic, $P2_1$
a = 11.4729 (11) Å
b = 5.9019 (4) Å
c = 12.5093 (10) Å
β = 107.810 (10)°
V = 806.44 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.80 × 0.37 × 0.23 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.934, T_{max} = 0.981
5708 measured reflections
1705 independent reflections
1240 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.072
S = 0.95
1705 reflections
208 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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: *ORTEPIII* (Burnett & Johnson, 1996); *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2262).

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supplementary materials

Acta Cryst. (2007). E**63**, o4767 [**doi:10.1107/S1600536807055365**]

(4*S*)-Benzyl 4-benzyl-5-oxo-1,3-oxazolidine-3-carboxylate

X.-H. Zhang, K. Lin and Y.-F. Zhao

Comment

The title compound (I), has been used to synthesize dipeptides and *N*-methyl-α-amino acid (Dorow & Gingrich, 1999). The absolute configuration of the stereocentre remains unchanged during the synthetic procedure.

The title compound, $C_{18}H_{17}N_1O_4$, is built up from an oxazolidine-3-carboxylate moiety attached to two benzyl rings. The carboxylate and the oxazolidine are roughly coplanar with the largest deviation from the least-square plane being −0.144 (2)Å at the nitrogen atom. The two benzyl rings are located on the same side of this plane and make dihedral angle with it of 75.23 (4)° and 56.97 (7)° respectively (Fig. 1). The bond lengths and angles in (I) are in agreement with the values reported in the literature (Lin *et al.*, 2007).

Experimental

The *N*-Benzoxycarbonyl-*L*-phenylalanine (2.99 g, 1 mmol), paraformaldehyde (0.6 g, 2 mmol) and 4-methylbenzenesulfonic acid (0.15 g, 0.6 mmol) were dissolved in benzene (150 ml). The solution was refluxed for 1 h with a Dean-Stark trap. After cooling to room temperature, the mixture was washed with 0.3 *M* aqueous K_2CO_3 solution, water, and saturated aqueous NaCl solution, then dried over Mg_2SO_4 (Vidyasagar *et al.*, 2000). We got white soild after evaporation of the solvent. Single crystals were obtained by recrystallization from ethanol.

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic), 0.97 Å (methylene) or 0.98Å (methine) with $U_{iso}(H) = 1.2U_{eq}(C)$.

In the absence of significant anomalous scattering, the absolute configuration could not be reliably determined and then the Friedel pairs were merged and any references to the Flack parameter were removed. The absolute configuration was deduced from the synthetic procedure.

Figures

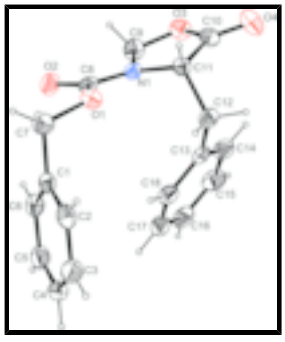


Fig. 1. Molecular view of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

(4*S*)-Benzyl 4-benzyl-5-oxo-1,3-oxazolidine-3-carboxylate

Crystal data

$C_{18}H_{17}NO_4$	F_{000} = 328
M_r = 311.33	D_x = 1.282 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 2497 reflections
a = 11.4729 (11) Å	θ = 4.2–32.7º
b = 5.9019 (4) Å	μ = 0.09 mm^{-1}
c = 12.5093 (10) Å	T = 173 (2) K
β = 107.810 (10)º	Block, colorless
V = 806.44 (11) Å^3	0.80 × 0.37 × 0.23 mm
Z = 2	

Data collection

Bruker APEX area-detector diffractometer	1705 independent reflections
Radiation source: fine-focus sealed tube	1240 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.027
T = 173(2) K	θ_{max} = 26.0º
φ and ω scan	θ_{min} = 4.2º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	h = −14→13
T_{min} = 0.934, T_{max} = 0.981	k = −7→7
5708 measured reflections	l = −15→13

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.072$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.95$	$\Delta\rho_{max} = 0.11$ e Å^{-3}
1705 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}
208 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.42377 (15)	0.7609 (3)	0.84565 (14)	0.0362 (4)
C8	0.36781 (19)	0.8526 (4)	0.91468 (16)	0.0400 (5)
O1	0.28778 (13)	0.7072 (2)	0.93711 (12)	0.0457 (4)
O3	0.58323 (14)	0.7171 (3)	0.77604 (14)	0.0522 (4)
C11	0.41290 (18)	0.5305 (4)	0.80224 (17)	0.0380 (5)
H11	0.4233	0.4231	0.8642	0.046*
O2	0.38833 (15)	1.0438 (3)	0.95256 (13)	0.0540 (4)
C1	0.08937 (19)	0.8764 (4)	0.91329 (16)	0.0391 (5)
O4	0.55782 (18)	0.3612 (3)	0.71837 (16)	0.0665 (5)
C7	0.2070 (2)	0.8026 (5)	0.99621 (18)	0.0520 (6)
H7A	0.2465	0.9312	1.0411	0.062*
H7B	0.1908	0.6897	1.0462	0.062*
C13	0.25038 (19)	0.6702 (4)	0.62687 (17)	0.0412 (5)
C6	0.0774 (2)	1.0868 (4)	0.86208 (19)	0.0470 (6)
H6	0.1430	1.1873	0.8806	0.056*
C18	0.1527 (2)	0.8044 (4)	0.63085 (17)	0.0473 (6)
H18	0.1126	0.7714	0.6834	0.057*
C12	0.2927 (2)	0.4786 (4)	0.70912 (18)	0.0443 (6)
H12A	0.3042	0.3443	0.6687	0.053*
H12B	0.2294	0.4455	0.7434	0.053*
C10	0.5233 (2)	0.5193 (4)	0.7604 (2)	0.0464 (6)
C2	−0.0100 (2)	0.7305 (4)	0.88517 (19)	0.0478 (6)
H2	−0.0031	0.5892	0.9195	0.057*

C9	0.5257 (2)	0.8793 (4)	0.8266 (2)	0.0531 (6)
H9A	0.4975	1.0078	0.7771	0.064*
H9B	0.5818	0.9328	0.8969	0.064*
C3	−0.1181 (2)	0.7913 (5)	0.8076 (2)	0.0592 (7)
H3	−0.1839	0.6913	0.7892	0.071*
C15	0.2702 (3)	0.9083 (5)	0.4770 (2)	0.0657 (8)
H15	0.3108	0.9443	0.4253	0.079*
C17	0.1129 (2)	0.9859 (4)	0.5590 (2)	0.0549 (7)
H17	0.0462	1.0717	0.5624	0.066*
C5	−0.0318 (3)	1.1480 (5)	0.7835 (2)	0.0578 (7)
H5	−0.0392	1.2887	0.7485	0.069*
C14	0.3083 (2)	0.7238 (5)	0.54794 (18)	0.0553 (6)
H14	0.3734	0.6355	0.5424	0.066*
C16	0.1727 (3)	1.0383 (5)	0.48261 (19)	0.0614 (7)
H16	0.1475	1.1614	0.4347	0.074*
C4	−0.1294 (3)	1.0006 (5)	0.7571 (2)	0.0638 (8)
H4	−0.2031	1.0425	0.7049	0.077*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0350 (9)	0.0346 (10)	0.0357 (9)	−0.0039 (8)	0.0061 (8)	−0.0009 (8)
C8	0.0378 (12)	0.0417 (13)	0.0317 (10)	0.0002 (11)	−0.0022 (9)	0.0031 (11)
O1	0.0428 (9)	0.0458 (9)	0.0495 (9)	0.0026 (8)	0.0155 (7)	0.0040 (7)
O3	0.0434 (8)	0.0498 (9)	0.0658 (10)	−0.0059 (9)	0.0204 (8)	0.0041 (8)
C11	0.0405 (12)	0.0315 (10)	0.0411 (11)	−0.0006 (10)	0.0112 (9)	0.0035 (10)
O2	0.0617 (10)	0.0457 (10)	0.0511 (9)	−0.0065 (9)	0.0121 (8)	−0.0122 (8)
C1	0.0453 (13)	0.0426 (12)	0.0345 (11)	0.0021 (11)	0.0196 (10)	−0.0027 (10)
O4	0.0763 (12)	0.0482 (10)	0.0890 (13)	0.0123 (10)	0.0463 (10)	0.0088 (10)
C7	0.0531 (14)	0.0663 (16)	0.0383 (12)	0.0082 (13)	0.0166 (11)	0.0061 (11)
C13	0.0399 (12)	0.0472 (13)	0.0312 (11)	−0.0092 (11)	0.0031 (9)	−0.0082 (9)
C6	0.0587 (16)	0.0400 (14)	0.0451 (13)	−0.0027 (12)	0.0203 (12)	−0.0026 (10)
C18	0.0464 (13)	0.0572 (14)	0.0349 (12)	−0.0048 (12)	0.0074 (10)	−0.0004 (11)
C12	0.0445 (12)	0.0399 (12)	0.0446 (13)	−0.0095 (10)	0.0079 (11)	−0.0039 (10)
C10	0.0467 (13)	0.0422 (14)	0.0495 (13)	0.0045 (12)	0.0135 (11)	0.0095 (12)
C2	0.0564 (14)	0.0467 (13)	0.0487 (13)	−0.0005 (13)	0.0285 (12)	−0.0025 (11)
C9	0.0518 (14)	0.0463 (13)	0.0630 (15)	−0.0135 (12)	0.0201 (12)	−0.0061 (12)
C3	0.0484 (15)	0.0724 (18)	0.0593 (15)	−0.0075 (13)	0.0202 (13)	−0.0162 (14)
C15	0.0684 (18)	0.092 (2)	0.0355 (13)	−0.0063 (17)	0.0144 (13)	0.0131 (14)
C17	0.0622 (16)	0.0579 (16)	0.0403 (13)	0.0052 (13)	0.0094 (12)	−0.0033 (12)
C5	0.0779 (18)	0.0519 (14)	0.0428 (13)	0.0208 (15)	0.0174 (14)	0.0012 (11)
C14	0.0496 (13)	0.0779 (17)	0.0374 (13)	0.0003 (14)	0.0117 (11)	−0.0026 (13)
C16	0.0737 (18)	0.0630 (16)	0.0371 (13)	−0.0034 (16)	0.0017 (13)	0.0046 (12)
C4	0.0502 (16)	0.088 (2)	0.0465 (14)	0.0222 (17)	0.0043 (12)	−0.0156 (16)

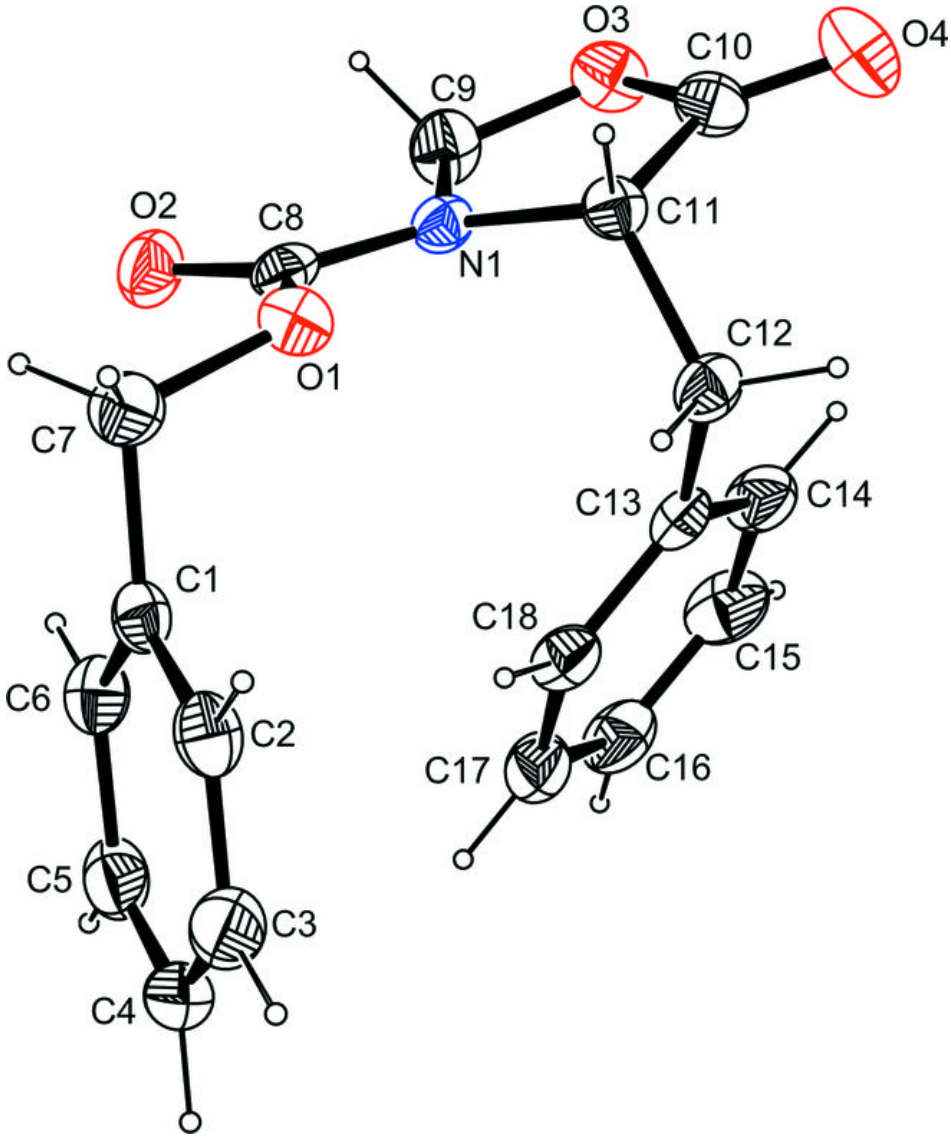
Geometric parameters (Å, °)

N1—C8	1.338 (3)	C6—H6	0.9300
N1—C9	1.444 (3)	C18—C17	1.383 (3)

N1—C11	1.455 (3)	C18—H18	0.9300
C8—O2	1.218 (3)	C12—H12A	0.9700
C8—O1	1.348 (3)	C12—H12B	0.9700
O1—C7	1.464 (3)	C2—C3	1.368 (4)
O3—C10	1.339 (3)	C2—H2	0.9300
O3—C9	1.416 (3)	C9—H9A	0.9700
C11—C10	1.512 (3)	C9—H9B	0.9700
C11—C12	1.539 (3)	C3—C4	1.376 (4)
C11—H11	0.9800	C3—H3	0.9300
C1—C6	1.385 (3)	C15—C16	1.375 (4)
C1—C2	1.386 (3)	C15—C14	1.388 (4)
C1—C7	1.493 (3)	C15—H15	0.9300
O4—C10	1.197 (3)	C17—C16	1.371 (4)
C7—H7A	0.9700	C17—H17	0.9300
C7—H7B	0.9700	C5—C4	1.376 (4)
C13—C14	1.385 (3)	C5—H5	0.9300
C13—C18	1.386 (3)	C14—H14	0.9300
C13—C12	1.506 (3)	C16—H16	0.9300
C6—C5	1.383 (4)	C4—H4	0.9300
C8—N1—C9	118.93 (18)	C11—C12—H12B	108.9
C8—N1—C11	128.01 (17)	H12A—C12—H12B	107.7
C9—N1—C11	111.83 (18)	O4—C10—O3	121.3 (2)
O2—C8—N1	123.5 (2)	O4—C10—C11	127.7 (2)
O2—C8—O1	124.9 (2)	O3—C10—C11	111.0 (2)
N1—C8—O1	111.62 (18)	C3—C2—C1	120.9 (2)
C8—O1—C7	115.91 (17)	C3—C2—H2	119.5
C10—O3—C9	111.44 (18)	C1—C2—H2	119.5
N1—C11—C10	100.64 (17)	O3—C9—N1	104.96 (18)
N1—C11—C12	115.04 (18)	O3—C9—H9A	110.8
C10—C11—C12	112.36 (18)	N1—C9—H9A	110.8
N1—C11—H11	109.5	O3—C9—H9B	110.8
C10—C11—H11	109.5	N1—C9—H9B	110.8
C12—C11—H11	109.5	H9A—C9—H9B	108.8
C6—C1—C2	118.8 (2)	C2—C3—C4	119.9 (3)
C6—C1—C7	121.6 (2)	C2—C3—H3	120.1
C2—C1—C7	119.6 (2)	C4—C3—H3	120.1
O1—C7—C1	109.80 (16)	C16—C15—C14	120.4 (2)
O1—C7—H7A	109.7	C16—C15—H15	119.8
C1—C7—H7A	109.7	C14—C15—H15	119.8
O1—C7—H7B	109.7	C16—C17—C18	119.5 (3)
C1—C7—H7B	109.7	C16—C17—H17	120.3
H7A—C7—H7B	108.2	C18—C17—H17	120.3
C14—C13—C18	117.7 (2)	C4—C5—C6	119.9 (3)
C14—C13—C12	122.3 (2)	C4—C5—H5	120.0
C18—C13—C12	120.0 (2)	C6—C5—H5	120.0
C5—C6—C1	120.2 (2)	C13—C14—C15	120.6 (2)
C5—C6—H6	119.9	C13—C14—H14	119.7
C1—C6—H6	119.9	C15—C14—H14	119.7
C17—C18—C13	121.9 (2)	C17—C16—C15	119.9 (2)

C17—C18—H18	119.0	C17—C16—H16	120.1
C13—C18—H18	119.0	C15—C16—H16	120.1
C13—C12—C11	113.25 (17)	C3—C4—C5	120.2 (2)
C13—C12—H12A	108.9	C3—C4—H4	119.9
C11—C12—H12A	108.9	C5—C4—H4	119.9
C13—C12—H12B	108.9		

Fig. 1


O3
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C9
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C6
C2
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C16
C5
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C4