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Methyl *N*-(benzyloxycarbonyl)oxamate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.098
Data-to-parameter ratio = 19.3

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

The title compound, $C_{11}H_{11}NO_5$, is almost planar. The molecules pack as hydrogen-bonded dimers.

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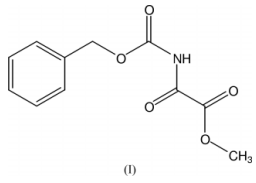
Comment

N- and C-protected ethyl oxamates are excellent nitrogen nucleophiles in Gabriel reactions (Berrée *et al.*, 1999). They are available by oxidation of protected serine or threonine (Stachulski, 1982). For our synthesis of methyl *N*-(benzyloxycarbonyl)oxamate, (I), we chose oxidation with RuO_2/$NaIO_4$ (Garner & Park, 1990), which leads to better yields.



(I)

The title compound is almost planar (r.m.s. deviation for all non-H atoms is 0.06 Å). All torsion angles excluding H atoms are close to either 0 or 180°. The biggest deviations from planarity are −2.75 (18)° for O2—C3—N4—C5 and 175.02 (10)° for C11—C1—O2—C3. An interesting feature of (I) is the rather long Csp^3—Csp^3 bond between atoms C5 and C6. The molecules pack as hydrogen-bonded dimers.

Only one similar compound, (II) (Wyss & Brisse, 1984), has been found in the Cambridge Structural Database (CSD, Version 5.24 of April 2003; Allen, 2002) which differs from (I) in the terminal groups: the benzyl group in (I) is replaced by a *tert*-butyl group in (II) and the methyl group in (I) is replaced by a benzyl group in (II). Although this leads to a completely

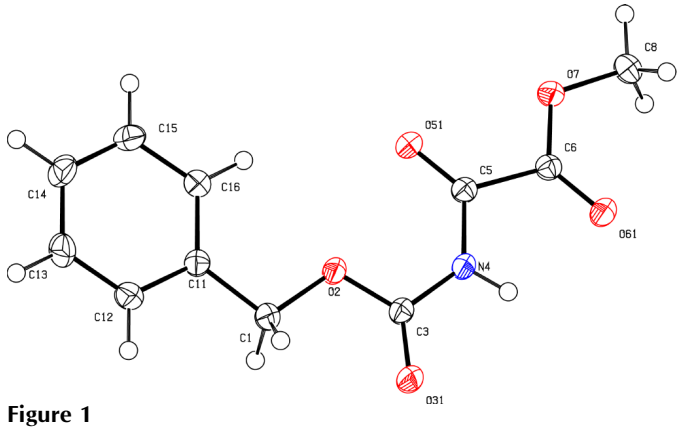


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

different conformation of (II), the latter also packs as hydrogen-bonded dimers. The rather long Csp^3-Csp^3 (1.533 Å) bond is also observed in (II).

Experimental

N-Benzyloxycarbonyl-L-serine methyl ester (2 g) was dissolved in 200 ml of a 3:2 mixture of acetone and water. With stirring, 10 equivalents of sodium periodate and 0.08 equivalents of ruthenium dioxide were added. The suspension was stirred for 24 h at room temperature. 10 ml of 2-propanol were added and the mixture stirred for a further 90 min to consume excess oxidant. The suspension was separated by centrifugation, yielding a clear yellow solution which was then concentrated *in vacuo*. HPLC purification yielded 1.44 g of pure *N*-benzyloxycarbonyl methyl oxamate (77%). The crystals were obtained directly from the HPLC fractions.

Crystal data

$C_{11}H_{11}NO_5$
M_r = 237.21
Monoclinic, $P2_1/c$
a = 8.0139 (8) Å
b = 5.4828 (5) Å
c = 24.701 (3) Å
β = 95.446 (2)°
V = 1080.4 (2) Å^3
Z = 4
D_x = 1.458 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 512 reflections
θ = 3.2–20.7°
μ = 0.12 mm^{-1}
T = 173 (2) K
Plate, colourless
0.52 × 0.28 × 0.12 mm

Data collection

Siemens 1K CCD three-circle diffractometer
ω scans
Absorption correction: none
17271 measured reflections
3064 independent reflections
2248 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 30.6°
$h = -11 \rightarrow 11$
$k = -7 \rightarrow 7$
$l = -33 \rightarrow 34$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.098
S = 1.02
3064 reflections
159 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 0.2657P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0114 (17)

Table 1
Selected geometric parameters (Å).

C1—O2	1.4529 (15)	C5—O51	1.2057 (14)
O2—C3	1.3243 (14)	C5—C6	1.5430 (17)
C3—O31	1.2088 (15)	C6—O61	1.1980 (14)
C3—N4	1.3868 (16)	C6—O7	1.3221 (15)
N4—C5	1.3737 (16)	O7—C8	1.4570 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O31^{i}	0.863 (17)	2.156 (17)	2.9778 (14)	158.9 (14)

Symmetry code: (i) $-x, -y, 1-z$.

H atoms bonded to C atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C_{methyl})] using a riding model with C—H = 0.99 Å or methyl C—H = 0.98 Å. The H atom bonded to N was refined freely.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97.

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