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(3*S*)-3-Benzyloxymethyl-1,4-dioxane-2,5-dione

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
R factor = 0.043
wR factor = 0.102
Data-to-parameter ratio = 7.1

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

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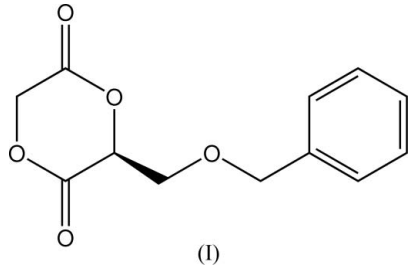
The lactide ring in the title compound, $C_{12}H_{12}O_5$, adopts a screw-boat conformation. C—H···O interactions link the molecules into a chain in the [100] direction.

Comment

The structure of the title compound, (I), was determined in the course of our investigations towards a better understanding of the regioselectivity observed in the ring-opening polymerization of various substituted (3*S*)-3-benzyloxymethyl-1,4-dioxane-2,5-dione derivatives (Leemhuis *et al.*, 2005). Earlier, we reported the crystal structures of the 6(*R*)-methyl (Kooijman *et al.*, 2005*a*) and the 6(*S*)-methyl derivatives (Kooijman *et al.*, 2005*b*). The molecular structure of (I) is displayed in Fig. 1 and selected geometric parameters are given in Table 1.



(I)

The lactide ring has taken a somewhat deformed screw-boat conformation. The asymmetry parameter (Duax & Norton, 1975) ΔC_2(C2—O3) = 6.4 (5)°; all other asymmetry parameters have values of 18° or higher. The Cremer & Pople puckering parameters (Cremer & Pople, 1975) are θ =

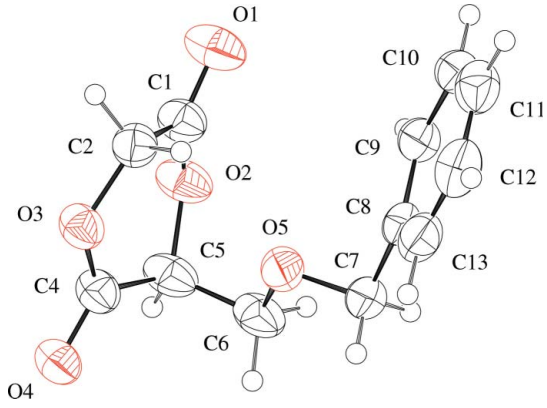


Figure 1
Atomic displacement plot (Spek, 2003) of the title compound, showing the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

77.1 (6)° and φ = 320.3 (6)°; the ideal values for the observed screw-boat conformation are θ = 67.5° and φ = 330°. The benzyloxymethyl substituent of the lactide ring occupies the axial position, as illustrated by the angle between the least-squares plane through the non-planar lactide ring and the C5—C6 bond, which amounts to 77.9 (3)°. In the 6(*R*)-methyl derivative, the benzyloxymethyl group also occupies the axial position [plane–bond angle = 67.20 (13)°]. The 6(*S*)-methyl derivative, however, has the benzyloxymethyl group in the equatorial position [plane–bond angle is 13.13 (13)°], most likely due to steric hindrance between the substituents of the lactide ring. The link between the two ring systems is not in an all-*trans* conformation, the torsion angles C4—C5—C6—O4 and O5—C7—C8—C9 having the *−gauche* conformation.

The packing displays short C—H···O contacts, geometric details of which are given in Table 2. These contacts link the molecules into an infinite chain in the [100] direction (see Fig. 2).

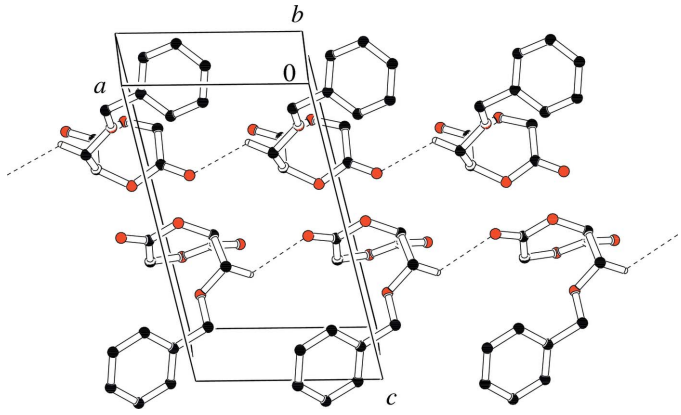


Figure 2
Short contacts C6—H6*A*···O1(x − 1, y, z) link the molecules into an infinite chain in the [100] direction.

Experimental

The synthesis of the title compound is described elsewhere (Leemhuis *et al.*, 2003). Crystals were grown from a solution in methyl *tert*-butyl ether.

Crystal data

$C_{12}H_{12}O_5$	D_x = 1.413 Mg m^{-3}
M_r = 236.22	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 219 reflections
a = 6.925 (4) Å	θ = 2.0–25.0°
b = 7.025 (4) Å	μ = 0.11 mm^{-1}
c = 11.733 (8) Å	T = 150 K
β = 103.44 (3)°	Prism, colourless
V = 555.2 (6) Å^3	0.15 × 0.05 × 0.05 mm
Z = 2	

Data collection

Nonius KappaCCD area-detector diffractometer	899 reflections with $I > 2\sigma(I)$
φ scans and ω scans with κ offsets	R_{int} = 0.087
Absorption correction: none	θ_{max} = 25.3°
12280 measured reflections	$h = -8 \rightarrow 8$
1098 independent reflections	$k = -8 \rightarrow 8$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.1P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.043	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.102	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
S = 1.11	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
1098 reflections	
154 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O2—C1	1.339 (4)	O3—C2	1.437 (5)
O2—C5	1.446 (4)	O3—C4	1.333 (4)
C1—O2—C5	118.3 (3)	C2—O3—C4	120.7 (3)
C7—O5—C6—C5	−179.6 (3)	C4—C5—C6—O5	−61.9 (4)
C6—O5—C7—C8	158.0 (3)	O5—C7—C8—C9	−59.7 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6*A*···O1^{i}	0.99	2.58	3.274 (5)	127

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

In the absence of significant anomalous scatterers, Friedel's law still holds. Friedel pairs were therefore averaged. The absolute configuration of C5 was chosen in accordance with the enenatiopure starting material. H atoms were introduced in calculated positions, with C—H = 0.95–1.00 Å, and refined as riding on their carrier atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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