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Key indicators

Single-crystal X-ray study
T = 150 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 8.9

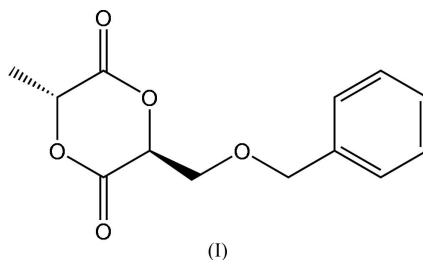
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

(3*S*,6*R*)-3-Benzoyloxymethyl-6-methyl-1,4-dioxane-2,5-dione

The chiral centres in the dilactone moiety of the title compound, $\text{C}_{13}\text{H}_{14}\text{O}_5$, are in the configuration 3*S*,6*R*. The ring itself has a somewhat flattened twist-boat conformation. C—H \cdots O interactions join the molecules into a two-dimensional network running parallel to the (101) plane.

Comment

The structure of the title compound was determined in the course of our investigations towards a better understanding of the regioselectivity observed in the ring-opening polymerization of the title compound, (I) (Leemhuis *et al.*, 2005).



The sample from which the crystals were grown was synthesized from enantiopure (2*S*)-3-benzyloxy-2-hydroxypropanoic acid and (\pm)- α -bromopropionyl bromide. The stereoisomers of this reaction were separated by column chromatography. The absolute configuration of the chiral centres in the dilactone moiety was chosen in accordance with the enantiopure starting material. The configuration of the chiral atom C2 is *R*; that of C5 is *S*. The structure of the *S,S* stereoisomer has also been determined and is published in a separate report (Kooijman *et al.*, 2005). The lactide ring has a somewhat flattened twist-boat conformation, as is common for 3*S*,6*R*-substituted lactides (*e.g.* Bolte *et al.*, 1994). The maximum deviation of the ring atoms from the least-squares plane through the lactide ring is 0.1402 (14) \AA for O3. The lowest asymmetry parameters (Duax & Norton, 1975) are $\Delta C_2(\text{C1}) = 3.24 (16)^\circ$ and $\Delta C_2(\text{C2}-\text{O3}) = 4.0 (2)^\circ$; all other asymmetry parameters have values above 10° . The Cremer & Pople (1975) puckering parameters are $\theta = 90.3 (3)$ and $\varphi = 330.9 (4)^\circ$; the ideal values for this particular twist-boat conformation are $\theta = 90^\circ$ and $\varphi = 330^\circ$. The link between the two ring systems is not in an all-*trans* conformation; the torsion angles O2—C5—C6—O5, C6—O5—C7—C8 and O5—C7—C8—C9 all take the +*gauche* conformation.

The packing displays short C—H \cdots O contacts, geometric details of which are given in Table 2. The C2—H2 \cdots O2(1 + *x*, *y*, *z*) contacts join the molecules into an infinite chain in the [100] direction, and the C10—H10 \cdots O4(*x* - 1, *y* + 1, *z*)

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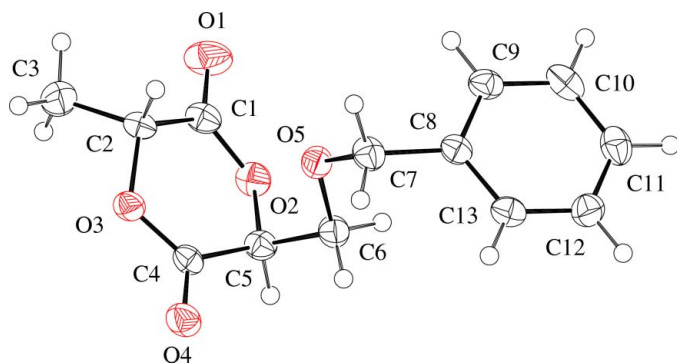


Figure 1

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

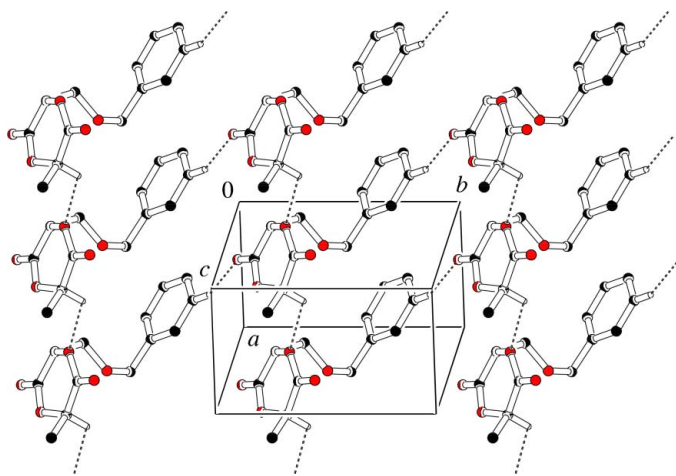


Figure 2

Short contacts [C2–H2...O2(1 + *x*, *y*, *z*) and C10–H10...O4(*x* – 1, *y* + 1, *z*), dashed lines] link the molecules into a two-dimensional network, parallel to the (101) plane.

contacts join the molecules into an infinite chain in the [110] direction. The combination of these chains generates a two-dimensional network, parallel to the (101) plane (Fig. 2).

Experimental

The synthesis of the title compound is described elsewhere (Leemhuis *et al.*, 2003). Crystals were grown from a sample obtained by vacuum distillation of the crude compound. To a solution of the refined product in methyl-*tert*-butyl ether some hexane was added. This solution was placed in a refrigerator and after a few hours colourless crystals were formed.

Crystal data

C₁₃H₁₄O₅
M_r = 250.24
 Monoclinic, *P*2₁
a = 5.289 (2) Å
b = 8.678 (2) Å
c = 13.178 (5) Å
 β = 96.748 (12)°
V = 600.7 (4) Å³
Z = 2

D_x = 1.383 Mg m^{–3}
 Mo *K*α radiation
 Cell parameters from 156 reflections
 θ = 2.0–25.0°
 μ = 0.11 mm^{–1}
T = 150 K
 Block, colourless
 0.25 × 0.10 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer
 φ scans, and ω scans with κ offsets
 Absorption correction: none
 16381 measured reflections
 1464 independent reflections

1308 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.056
 θ_{max} = 27.5°
h = –6 → 6
k = –11 → 11
l = –17 → 17

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.031
wR(*F*²) = 0.076
S = 1.07
 1464 reflections
 164 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.02P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.12 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O2–C1	1.336 (2)	O3–C2	1.450 (2)
O2–C5	1.440 (2)	O3–C4	1.326 (2)
C1–O2–C5	121.33 (13)	C2–O3–C4	121.39 (13)
C7–O5–C6–C5	163.22 (14)	O2–C5–C6–O5	66.08 (16)
C6–O5–C7–C8	57.98 (19)	O5–C7–C8–C9	66.3 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C2–H2...O2 ⁱ	1.00	2.60	3.152 (2)	115
C10–H10...O4 ⁱⁱ	0.95	2.57	3.465 (3)	158

Symmetry codes: (i) *x* + 1, *y*, *z*; (ii) *x* – 1, *y* + 1, *z*.

In the absence of significant anomalous scatterers, Friedel pairs were averaged. The methyl group was refined as a rigid group, allowing for rotation around the C–C bond. H atoms were treated as riding, with C–H distances of 0.95–1.00 Å and *U*_{iso}(H) values set to 1.5 or 1.2 times *U*_{eq} of the carrier atom for methyl and other H atoms, respectively.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1986); 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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