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

Huub Kooijman,[a]* Mark Leemhuis,[b] Cornelus F. van Nostrum,[b] Wim E. Hennink[b] and Anthony L. Spek[a]

[a]Bijvoet Centre for Biomolecular Research, Department of Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands, and [b]Department of Pharmaceutics, Utrecht Institute for Pharmaceutical Sciences (UIPS), Faculty of Pharmaceutical Sciences, Utrecht University, PO Box 80082, 3508 TB Utrecht, The Netherlands

Correspondence e-mail: h.kooijman@chem.uu.nl

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.095
Data-to-parameter ratio = 9.7

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

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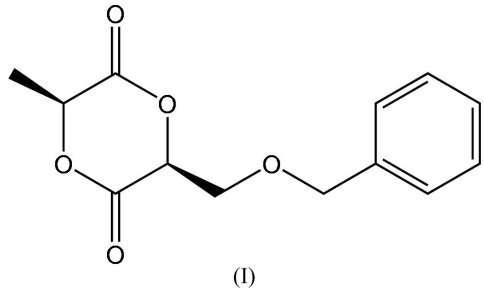
The chiral centres in the dilactone moiety of the title compound, $C_{13}H_{14}O_5$, are in the configuration 3*S*,6*S*. The ring itself has a boat conformation. C—H···O interactions link the molecules into a chain in the [010] direction.

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).



(I)

The sample from which the crystals were grown was synthesized from enantiopure (2*S*)-3-benzyloxy-2-hydroxy propanoic acid and (±)-α-bromopropionylbromide. The stereoisomers of this reaction were separated by column chromatography. The absolute configuration of the chiral centres in the lactide ring was chosen in accordance with the enantiopure starting material. Both chiral atoms, C2 and C5, are in the *S* configuration. The structure of the *R*,*S* stereoisomer has also been determined and is published in a separate report (Kooijman *et al.*, 2005). The lactide ring has a boat conformation, as is common for 3*S*,6S-substituted lactides (*e.g.* Bolte *et al.*, 1994). All ring substituents are in the equatorial position. The lowest asymmetry parameters (Duax & Norton, 1975) are ΔC_s(C2) = 9.74 (16)° and ΔC_s(O2—C1)=7.1 (2)°; all other asymmetry parameters have values 27° or higher. The Cremer & Pople (1975) puckering parameters are θ = 90.59 (18)° and φ = 125.54 (18)°; the ideal values for the observed boat conformation are $\theta = 90°$ and $\varphi = 120°$. The link between the two ring systems has an all-*trans* conformation, with the exception of the C—Ph bond.

The packing displays some relatively short C—H···O contacts, geometric details of which are given in Table 2. Both axial H atoms of the lactide ring, H2 and H5, have a short contact with keto atom O1($-x, -\frac{1}{2}+y, 1-z$). Atom H5 is also involved in a contact with another O1 atom, at equivalent position ($x, y-1, z$). The sum of the intermolecular angles involving atom H5 is 359°, indicating a bifurcated character.

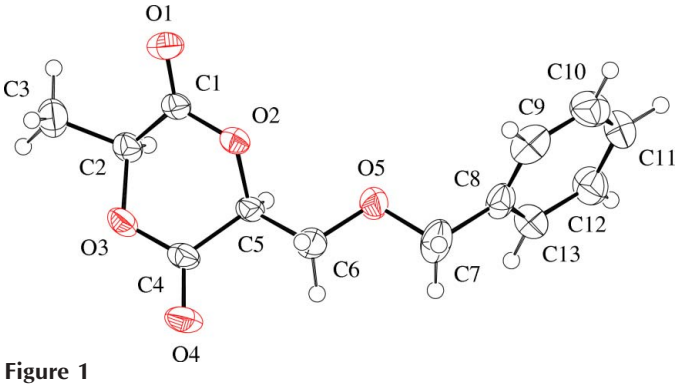


Figure 1
Atomic displacement plot (Spek, 2003) of the title compound, showing the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

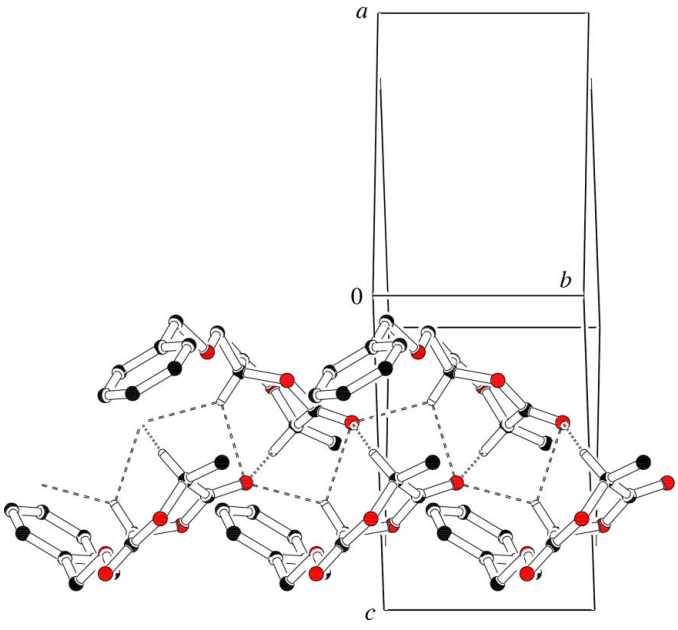


Figure 2
Short contacts [C2—H2···O1($-x, \frac{1}{2}-y, 1-z$), C5—H5···O1($-x, \frac{1}{2}-y, 1-z$) and C5—H5···O1($x, y-1, z$), dashed lines] link the molecules into an infinite chain in the [010] direction.

The C—H···O contacts join the molecules into an infinite chain in the [010] direction (Fig. 2).

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 to which some hexane was added. This solution was placed in a refrigerator and after 1 h colourless crystals were formed.

Crystal data

$C_{13}H_{14}O_5$
$M_r = 250.24$
Monoclinic, $P2_1$
$a = 8.944\ (2)$ Å
$b = 5.9400\ (10)$ Å
$c = 12.559\ (3)$ Å
$\beta = 107.905\ (12)^\circ$
$V = 634.9\ (2)$ Å^3
$Z = 2$
$D_x = 1.309$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 171 reflections
$\theta = 2.0$–25.0°
$\mu = 0.10$ mm^{-1}
$T = 150$ K
Plate, colourless
0.35 × 0.15 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: none
16 869 measured reflections
1590 independent reflections
1430 reflections with $I > 2\sigma(I)$
$R_{int} = 0.093$
$\theta_{max} = 27.5^\circ$
$h = -11 \rightarrow 11$
$k = -7 \rightarrow 7$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.095$
$S = 1.08$
1590 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 0.05P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: none

Table 1
Selected geometric parameters (Å, °).

O2—C1	1.337 (2)	O3—C2	1.457 (2)
O2—C5	1.446 (2)	O3—C4	1.340 (2)
C1—O2—C5	116.91 (14)	C2—O3—C4	117.76 (15)
C7—O5—C6—C5	−165.8 (2)	C4—C5—C6—O5	166.08 (16)
C6—O5—C7—C8	172.4 (2)	O5—C7—C8—C9	84.7 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1^{i}	1.00	2.57	3.247 (3)	125
C5—H5···O1^{ii}	1.00	2.32	3.134 (3)	137
C5—H5···O1^{i}	1.00	2.54	3.272 (3)	130

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

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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References

Bolte, M., Beck, H., Nieger, M. & Egert, E. (1994). *Acta Cryst.* C**50**, 1717–1721.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structure*, Vol. 1. New York: IFI/Plenum.

Kooijman, H., Leemhuis, M., van Nostrum, C. F., Hennink, W. E. & Spek, A. L. (2005). *Acta Cryst.* E**61**, o898–o900.
Leemhuis, M., van Nostrum, C. F. & Hennink, W. E. (2005). In preparation.
Leemhuis, M., van Steenis, J. H., van Uxem, M. J., van Nostrum, C. F., Hennink, W. E. (2003). *Eur. J. Org. Chem.* pp. 3344–3349.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1986). *SHELXS86*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.