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2,3-*O*-Isopropylidene-1-*O*-*p*-tolylsulfonylglycerol

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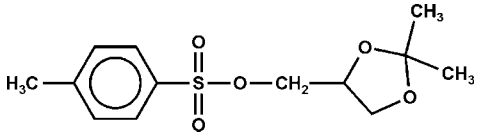
Key indicators: single-crystal X-ray study; T = 133 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.114; data-to-parameter ratio = 19.2.

In the title compound, $C_{13}H_{18}O_5S$, the five-membered ring has an envelope conformation. The packing involves four C—H···O interactions, three of which combine to form layers of molecules parallel to the *bc* plane.

Related literature

For related literature, see: Baer & Fischer (1948); Jones *et al.* (2003); Kazemi *et al.* (2007); Ouchi *et al.* (1990). The structure of a related derivative is presented in the following paper, see: Kuś *et al.* (2009).



Experimental

Crystal data

$C_{13}H_{18}O_5S$
M_r = 286.33
Monoclinic, $P2_1/c$
a = 15.143 (2) Å
b = 5.7297 (9) Å
c = 15.665 (2) Å
β = 90.385 (3)°
V = 1359.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 133 K
0.40 × 0.20 × 0.05 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: none
12509 measured reflections
3357 independent reflections
2339 reflections with $I > 2\sigma(I)$
R_{int} = 0.111

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.114
S = 1.05
3357 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16···O1^{i}	0.95	2.60	3.212 (2)	122
C17—H17C···O1^{ii}	0.98	2.65	3.369 (3)	131
C17—H17B···O1^{iii}	0.98	2.66	3.558 (3)	153
C5—H5A···O2^{iv}	0.98	2.64	3.509 (3)	148

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Siemens, 1994); 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: BT2935).

References

Baer, E. & Fischer, H. O. L. (1948). *J. Am. Chem. Soc.* **70**, 609–610.
Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jones, P. G., Kuś, P. & Celinski, R. (2003). *Acta Cryst.* E**59**, o117–o118.
Kazemi, F., Massah, A. R. & Javaherian, M. (2007). *Tetrahedron*, **63**, 5083–5087.
Kuś, P., Rojkiewicz, M., Zięba, G., Witoszek, M. & Jones, P. G. (2009). *Acta Cryst.* E**65**, o1192.
Ouchi, M., Inoue, Y., Liu, Y., Nagamune, S., Nakamura, S., Wada, K. & Hakushi, K. (1990). *Bull. Chem. Soc. Jpn*, 63, 1260–1262.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1994). *XP*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supporting information

Acta Cryst. (2009). E**65**, o1191 [doi:10.1107/S1600536809015037]

2,3-*O*-Isopropylidene-1-*O*-*p*-tolylsulfonylglycerol

Piotr Kuś, Marcin Rojkiewicz, Grzegorz Zięba and Peter G. Jones

S1. Comment

Isopropylidene and tosyl groups are often used as protecting or activating units in polyhydroxyalkyl compounds used for synthesis of sugar-like derivatives. Recently we described the crystal structure of *L*-arabitol tosylate protected by two isopropylidene groups (Jones *et al.*, 2003). *D*,*L*-Isopropylideneglycerol is an important substrate for the synthesis of many derivatives of glycerol; the isopropylidene protecting group is a convenient form of protection for the two vicinal hydroxyls in the molecule of glycerol. After the reaction at the third, free hydroxyl group, acid hydrolysis of the protecting group leads to 1-*O*-substituted glycerol derivatives. In our experiments we used commercial (Aldrich) solketal as precursor for the protecting group. Tosylation of this compound leads to compound 1 (Baer & Fischer, 1948; Ouchi *et al.* 1990; Kazemi *et al.* 2007).

The molecule is shown in Fig. 1. Bond lengths and angles may be regarded as normal. The five-membered ring displays an envelope conformation, with approximate local mirror symmetry about O5 and the midpoint of C2—O4. The chain of five atoms from S to C3 displays an extended conformation, with torsion angles close to ±180°.

There are four weak C—H···O interactions with H···O between 2.6 and 2.7 Å. Three of these combine to form layers of molecules parallel to the ***bc*** plane at $x \simeq 1/4$ (Fig. 2) and 3/4.

The structure of a related derivative is presented in the following paper (Kuś *et al.*, 2009).

S2. Experimental

The compound 1 was obtained according to method described by Kazemi *et al.* (2007). The analytical and spectroscopic data are consistent with the literature. Single crystals suitable for X-ray analysis were obtained by slow evaporation from petroleum ether.

NMR data: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.80 (d, 2H), 7.35 (d, 2H), 4.28 (q, 1H), 4.06–3.95 (m, 3H), 3.78–3.75 (dd, 1H), 2.45 (s, 3H), 1.34 (s, 3H), 1.31 (s, 3H). - ^{13}C NMR (100 MHz): δ 145.21, 132.81, 130.06, 128.15, 110.20, 73.05, 69.62, 66.35, 26.77, 25.28, 21.80.

IR data: —S(O_2)—O— vibrations at 1177 (*versus*) and 1348 cm^{-1} (*s*); 1,3-dioxalone ring at 971 cm^{-1}. There are no bands above 2990 cm^{-1}.

S3. Refinement

Methyl H atoms were identified in difference syntheses and refined as idealized rigid groups (C—H 0.98 Å, H—C—H 109.5°) allowed to rotate but not tip. Other H atoms were included at calculated positions and refined using a riding model, with fixed C—H bond lengths of 0.95 Å (CH, aromatic), 0.99 Å (CH_2) and 1.00 Å (CH, sp^3); U_{iso}(H) values were fixed at 1.2U_{eq} of the parent C atom (1.2U_{eq} for methyl H).

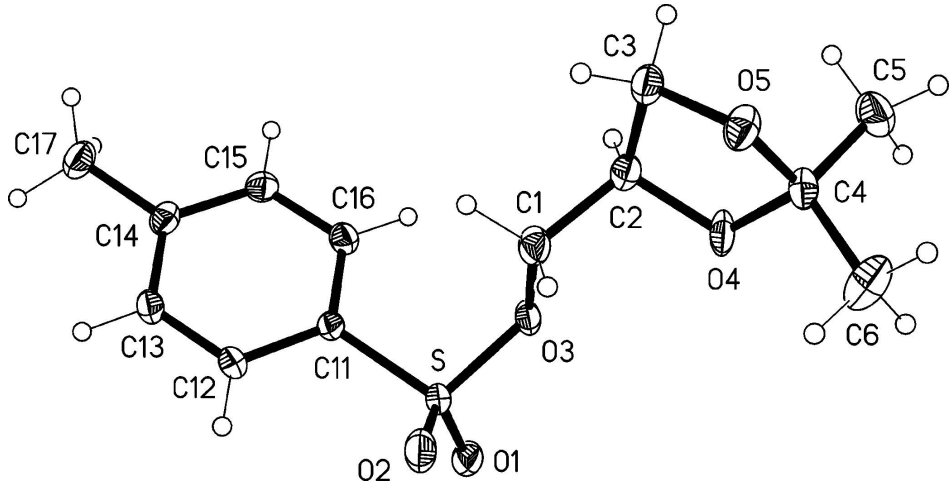

Figure 1

The title compound in the crystal structure. Displacement ellipsoids represent 50% probability levels.

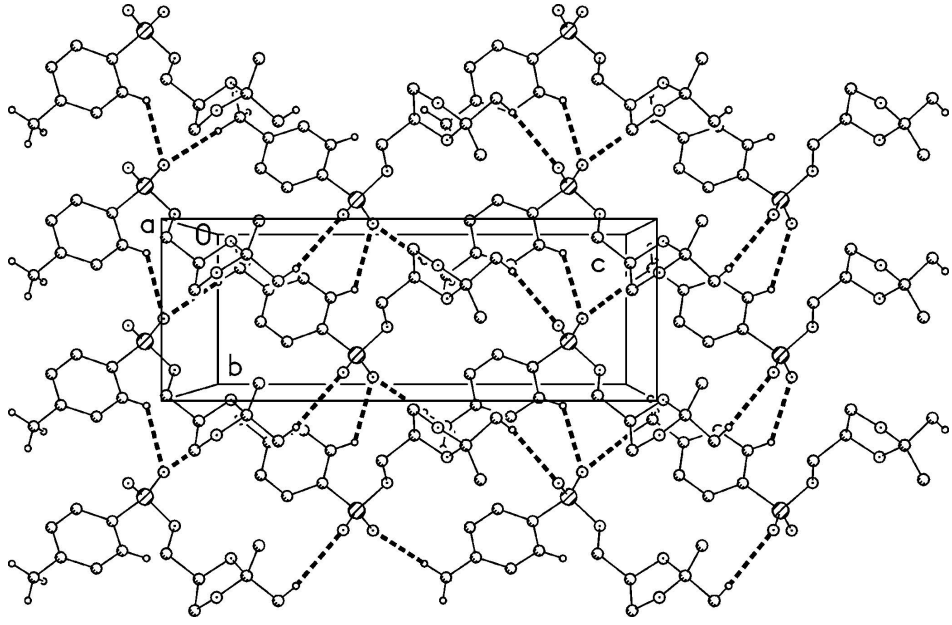

Figure 2

Packing diagram of the title compound in the region $x \simeq 1/4$. H atoms not involved in H bonding (thick dashed lines) are omitted for clarity.

2,3-*O*-Isopropylidene-1-*O*-*p*-tolylsulfonylglycerol

Crystal data

$C_{13}H_{18}O_5S$
$M_r = 286.33$
Monoclinic, $P2_1/c$
$a = 15.143$ (2) Å
$b = 5.7297$ (9) Å
$c = 15.665$ (2) Å
$\beta = 90.385$ (3)°
$V = 1359.2$ (4) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.399$ Mg m^{-3}
Melting point: 321 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5579 reflections
$\theta = 2.6$–29.6°
$\mu = 0.25$ mm^{-1}

T = 133 K
Lath, colourless

0.40 × 0.20 × 0.05 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.192 pixels mm^{-1}
ω scans
12509 measured reflections

3357 independent reflections
2339 reflections with $I > 2\sigma(I)$
R_{int} = 0.111
θ_{max} = 28.3°, θ_{min} = 1.3°
h = −19→20
k = −7→7
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.114
S = 1.05
3357 reflections
175 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.1472P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
S	0.20851 (4)	0.79423 (9)	0.35153 (3)	0.01847 (14)
O1	0.14179 (11)	0.9403 (3)	0.38666 (8)	0.0271 (4)
O2	0.28879 (11)	0.8986 (3)	0.32446 (9)	0.0287 (4)
O3	0.22741 (10)	0.6118 (2)	0.42410 (8)	0.0194 (3)
O4	0.31609 (11)	0.4304 (3)	0.56622 (8)	0.0278 (4)
O5	0.44258 (10)	0.2270 (3)	0.54736 (8)	0.0243 (4)
C1	0.30372 (15)	0.4563 (4)	0.41503 (12)	0.0229 (5)
H1A	0.3592	0.5478	0.4141	0.027*
H1B	0.2991	0.3661	0.3613	0.027*
C2	0.30289 (15)	0.2953 (4)	0.49053 (11)	0.0210 (4)
H2	0.2455	0.2093	0.4932	0.025*
C3	0.38036 (15)	0.1234 (4)	0.49008 (13)	0.0243 (5)
H3A	0.3618	−0.0325	0.5105	0.029*
H3B	0.4053	0.1077	0.4321	0.029*
C4	0.39249 (14)	0.3442 (4)	0.61038 (12)	0.0197 (5)

C5	0.36304 (18)	0.1785 (4)	0.67969 (13)	0.0322 (6)
H5A	0.3210	0.2579	0.7171	0.048*
H5B	0.4145	0.1280	0.7132	0.048*
H5C	0.3346	0.0420	0.6537	0.048*
C6	0.44515 (18)	0.5465 (5)	0.64354 (15)	0.0371 (6)
H6A	0.4604	0.6501	0.5961	0.056*
H6B	0.4994	0.4889	0.6708	0.056*
H6C	0.4102	0.6327	0.6854	0.056*
C11	0.16470 (14)	0.6269 (3)	0.26750 (11)	0.0162 (4)
C12	0.17492 (14)	0.7085 (4)	0.18417 (11)	0.0184 (4)
H12	0.2062	0.8489	0.1731	0.022*
C13	0.13833 (14)	0.5797 (4)	0.11806 (12)	0.0202 (5)
H13	0.1436	0.6356	0.0612	0.024*
C14	0.09418 (14)	0.3717 (4)	0.13239 (12)	0.0192 (4)
C15	0.08507 (14)	0.2955 (4)	0.21663 (12)	0.0201 (4)
H15	0.0547	0.1537	0.2277	0.024*
C16	0.11935 (14)	0.4224 (4)	0.28417 (12)	0.0188 (4)
H16	0.1119	0.3700	0.3412	0.023*
C17	0.05788 (16)	0.2279 (4)	0.06012 (13)	0.0259 (5)
H17A	0.0939	0.0872	0.0532	0.039*
H17B	0.0591	0.3195	0.0073	0.039*
H17C	−0.0031	0.1830	0.0727	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0273 (3)	0.0169 (2)	0.0112 (2)	−0.0003 (2)	−0.00206 (17)	0.0006 (2)
O1	0.0413 (11)	0.0222 (8)	0.0180 (7)	0.0097 (7)	−0.0027 (7)	−0.0032 (6)
O2	0.0373 (10)	0.0323 (9)	0.0166 (7)	−0.0122 (8)	−0.0038 (6)	0.0014 (6)
O3	0.0256 (9)	0.0215 (7)	0.0111 (6)	0.0052 (6)	−0.0013 (5)	0.0010 (5)
O4	0.0350 (10)	0.0365 (9)	0.0117 (6)	0.0169 (7)	−0.0068 (6)	−0.0064 (6)
O5	0.0205 (8)	0.0315 (9)	0.0207 (7)	0.0047 (7)	−0.0018 (6)	−0.0069 (6)
C1	0.0273 (13)	0.0260 (11)	0.0154 (9)	0.0079 (10)	0.0013 (8)	−0.0016 (8)
C2	0.0252 (12)	0.0222 (10)	0.0157 (9)	0.0014 (10)	−0.0013 (8)	−0.0029 (8)
C3	0.0284 (13)	0.0259 (11)	0.0185 (10)	0.0041 (10)	−0.0061 (8)	−0.0038 (8)
C4	0.0181 (11)	0.0271 (12)	0.0141 (9)	0.0026 (9)	−0.0020 (7)	−0.0006 (8)
C5	0.0432 (15)	0.0325 (13)	0.0209 (10)	0.0022 (12)	0.0017 (10)	0.0071 (10)
C6	0.0373 (16)	0.0429 (15)	0.0310 (12)	−0.0098 (12)	0.0041 (10)	−0.0151 (11)
C11	0.0203 (11)	0.0149 (9)	0.0132 (8)	0.0030 (8)	−0.0019 (7)	−0.0002 (7)
C12	0.0233 (11)	0.0179 (9)	0.0141 (8)	−0.0003 (9)	0.0012 (8)	0.0030 (8)
C13	0.0256 (12)	0.0214 (11)	0.0135 (9)	0.0027 (9)	−0.0014 (8)	0.0016 (8)
C14	0.0186 (11)	0.0213 (10)	0.0178 (9)	0.0034 (9)	−0.0011 (8)	−0.0040 (8)
C15	0.0218 (11)	0.0174 (9)	0.0211 (9)	−0.0032 (9)	0.0003 (8)	−0.0003 (9)
C16	0.0216 (12)	0.0191 (10)	0.0156 (9)	0.0004 (9)	0.0022 (8)	0.0020 (8)
C17	0.0303 (13)	0.0271 (12)	0.0203 (10)	−0.0024 (10)	−0.0022 (9)	−0.0069 (9)

Geometric parameters (Å, º)

S—O2	1.4220 (16)	C15—C16	1.382 (3)
S—O1	1.4253 (16)	C1—H1A	0.9900
S—O3	1.5694 (14)	C1—H1B	0.9900
S—C11	1.7552 (19)	C2—H2	1.0000
O3—C1	1.467 (2)	C3—H3A	0.9900
O4—C2	1.429 (2)	C3—H3B	0.9900
O4—C4	1.432 (2)	C5—H5A	0.9800
O5—C4	1.418 (2)	C5—H5B	0.9800
O5—C3	1.426 (2)	C5—H5C	0.9800
C1—C2	1.500 (3)	C6—H6A	0.9800
C2—C3	1.532 (3)	C6—H6B	0.9800
C4—C6	1.498 (3)	C6—H6C	0.9800
C4—C5	1.512 (3)	C12—H12	0.9500
C11—C16	1.384 (3)	C13—H13	0.9500
C11—C12	1.396 (2)	C15—H15	0.9500
C12—C13	1.384 (3)	C16—H16	0.9500
C13—C14	1.386 (3)	C17—H17A	0.9800
C14—C15	1.398 (3)	C17—H17B	0.9800
C14—C17	1.502 (3)	C17—H17C	0.9800
O2—S—O1	118.55 (10)	O4—C2—H2	110.4
O2—S—O3	110.12 (9)	C1—C2—H2	110.4
O1—S—O3	103.75 (8)	C3—C2—H2	110.4
O2—S—C11	109.01 (9)	O5—C3—H3A	111.1
O1—S—C11	110.18 (10)	C2—C3—H3A	111.1
O3—S—C11	104.21 (8)	O5—C3—H3B	111.1
C1—O3—S	118.31 (11)	C2—C3—H3B	111.1
C2—O4—C4	108.77 (15)	H3A—C3—H3B	109.0
C4—O5—C3	106.32 (16)	C4—C5—H5A	109.5
O3—C1—C2	106.63 (16)	C4—C5—H5B	109.5
O4—C2—C1	108.62 (17)	H5A—C5—H5B	109.5
O4—C2—C3	104.44 (16)	C4—C5—H5C	109.5
C1—C2—C3	112.42 (17)	H5A—C5—H5C	109.5
O5—C3—C2	103.40 (16)	H5B—C5—H5C	109.5
O5—C4—O4	105.15 (14)	C4—C6—H6A	109.5
O5—C4—C6	108.76 (18)	C4—C6—H6B	109.5
O4—C4—C6	109.08 (19)	H6A—C6—H6B	109.5
O5—C4—C5	111.38 (18)	C4—C6—H6C	109.5
O4—C4—C5	108.81 (19)	H6A—C6—H6C	109.5
C6—C4—C5	113.32 (18)	H6B—C6—H6C	109.5
C16—C11—C12	121.23 (18)	C13—C12—H12	120.8
C16—C11—S	120.43 (14)	C11—C12—H12	120.8
C12—C11—S	118.32 (16)	C12—C13—H13	119.1
C13—C12—C11	118.33 (19)	C14—C13—H13	119.1
C12—C13—C14	121.90 (18)	C16—C15—H15	119.3
C13—C14—C15	118.20 (18)	C14—C15—H15	119.3

C13—C14—C17	121.64 (18)	C15—C16—H16	120.5
C15—C14—C17	120.16 (19)	C11—C16—H16	120.5
C16—C15—C14	121.30 (19)	C14—C17—H17A	109.5
C15—C16—C11	119.02 (18)	C14—C17—H17B	109.5
O3—C1—H1A	110.4	H17A—C17—H17B	109.5
C2—C1—H1A	110.4	C14—C17—H17C	109.5
O3—C1—H1B	110.4	H17A—C17—H17C	109.5
C2—C1—H1B	110.4	H17B—C17—H17C	109.5
H1A—C1—H1B	108.6		

O2—S—O3—C1	−42.19 (16)	O2—S—C11—C16	145.72 (17)
O1—S—O3—C1	−170.05 (14)	O1—S—C11—C16	−82.58 (19)
C11—S—O3—C1	74.60 (16)	O3—S—C11—C16	28.16 (19)
S—O3—C1—C2	−177.32 (13)	O2—S—C11—C12	−35.86 (19)
C4—O4—C2—C1	−122.29 (19)	O1—S—C11—C12	95.84 (18)
C4—O4—C2—C3	−2.1 (2)	O3—S—C11—C12	−153.41 (16)
O3—C1—C2—O4	−64.2 (2)	C16—C11—C12—C13	0.1 (3)
O3—C1—C2—C3	−179.29 (16)	S—C11—C12—C13	−178.31 (16)
C4—O5—C3—C2	32.7 (2)	C11—C12—C13—C14	−1.5 (3)
O4—C2—C3—O5	−18.5 (2)	C12—C13—C14—C15	1.6 (3)
C1—C2—C3—O5	99.1 (2)	C12—C13—C14—C17	−177.4 (2)
C3—O5—C4—O4	−34.7 (2)	C13—C14—C15—C16	−0.3 (3)
C3—O5—C4—C6	−151.42 (19)	C17—C14—C15—C16	178.8 (2)
C3—O5—C4—C5	83.0 (2)	C14—C15—C16—C11	−1.0 (3)
C2—O4—C4—O5	22.3 (2)	C12—C11—C16—C15	1.1 (3)
C2—O4—C4—C6	138.77 (18)	S—C11—C16—C15	179.51 (16)
C2—O4—C4—C5	−97.16 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O1^{i}	0.95	2.60	3.212 (2)	122
C17—H17*C*···O1^{ii}	0.98	2.65	3.369 (3)	131
C17—H17*B*···O1^{iii}	0.98	2.66	3.558 (3)	153
C5—H5*A*···O2^{iv}	0.98	2.64	3.509 (3)	148

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*, *y*−1/2, −*z*+1/2; (iii) *x*, −*y*+3/2, *z*−1/2; (iv) *x*, −*y*+3/2, *z*+1/2.