

3-Oxapentane-1,5-diyl bis(allylsulfonate)

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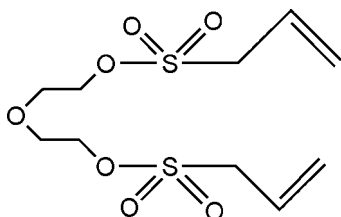
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.039; wR factor = 0.111; data-to-parameter ratio = 16.3.

The title compound, $\text{C}_{10}\text{H}_{18}\text{O}_7\text{S}_2$, was synthesized by reacting diethylene glycol with allyl chlorosulfonate in the presence of pyridine. The asymmetric unit consists of half a molecule, which is located on a twofold rotation axis. In the crystal structure, the molecules are involved in several weak C—H \cdots O interactions.

Related literature

For related work on monomers and polymers for nuclear track detection purposes, see: Mascarenhas *et al.* (2006). For related literature, see: Bondi (1964).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{18}\text{O}_7\text{S}_2$
 $M_r = 314.38$
Monoclinic, $C2/c$
 $a = 12.022$ (3) Å
 $b = 8.3484$ (18) Å
 $c = 14.894$ (3) Å
 $\beta = 101.096$ (3)°

$V = 1466.8$ (5) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.39$ mm⁻¹
 $T = 298$ (2) K
 $0.38 \times 0.38 \times 0.22$ mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
 $T_{\min} = 0.867$, $T_{\max} = 0.920$
4456 measured reflections
1437 independent reflections
1233 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.111$
 $S = 1.06$
1437 reflections
88 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2A}\cdots\text{O3}^{\text{ii}}$	0.97	2.53	3.219 (2)	128
$\text{C2}-\text{H2B}\cdots\text{O4}^{\text{iii}}$	0.97	2.41	3.332 (2)	159
$\text{C3}-\text{H3A}\cdots\text{O3}^{\text{iv}}$	0.97	2.65	3.594 (2)	163
$\text{C5}-\text{H5B}\cdots\text{O2}^{\text{v}}$	0.93	2.64	3.447 (2)	146

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2062).

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supplementary materials

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3-Oxapentane-1,5-diyl bis(allylsulfonate)

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Comment

As part of an ongoing research programme, we are designing monomers and polymers for nuclear track detection purpose (Mascarenhas *et al.*, 2006). During the course of this study we have synthesized two monomers namely diethylene glycol bis(allylsulfonate) (I) and the corresponding isomer allyl diglycol sulphite (ADS) having the same molecular formula [C₁₀H₁₈O₇S₂] but different functional groups. Interestingly compound (I) is a solid while the functional isomer (ADS) is a liquid at room temperature. The structure of (I) is described in this report.

In the crystal structure of the title compound the molecules are located with the ether oxygen atom (O1) on a 2-fold axis with one half of the molecule constituting the asymmetric unit (Fig. 1). An analysis of the structure reveals that each molecule of (I) is hydrogen bonded to four symmetry related molecules with the aid of C—H···O interactions (Fig. 2). All these O···H contacts are shorter than the sum of their van der Waals radii (Bondi, 1964) (Table 1).

Experimental

Diethylene glycol (10 g, 0.094 mol) was condensed with allyl chlorosulphonate (26.695 g, 0.19 mol) in the presence of pyridine (16.53 g, 0.209 mol). The product obtained was purified by column chromatography. Yield: 25.61 g (86%) of a colourless solid (m.p. 43–45°C). Crystals suitable for structure determination were prepared by recrystallizing from a mixture of 1:1 ethyl acetate and petroleum ether.

Refinement

The H atoms were positioned with idealized geometry (C—H = 0.93 and 0.97 Å and were refined isotropic ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$) using a riding model.

Figures

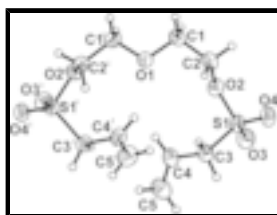


Fig. 1. Crystal structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at 30% probability level. The O1 atom is situated on a twofold axis. Symmetry code: i) $-x + 1, y, -z + 3/2$.

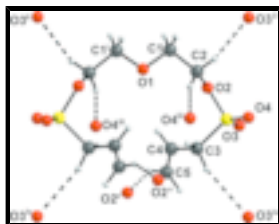


Fig. 2. A view of the surroundings of (I) showing its linking to four symmetry related molecules. (C—H...O bonds are shown as dashed lines). Symmetry codes: i) $-x + 1, y, -z + 3/2$; ii) $-x + 1/2, -y + 1/2, -z + 1$; iii) $-x + 1, -y, -z + 1$; iv) $-x + 1/2, -y - 1/2, -z + 1$; v) $-x + 1/2, y - 1/2, -z + 3/2$.

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$C_{10}H_{18}O_7S_2$

$M_r = 314.38$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 12.022 (3) \text{ \AA}$

$b = 8.3484 (18) \text{ \AA}$

$c = 14.894 (3) \text{ \AA}$

$\beta = 101.096 (3)^\circ$

$V = 1466.8 (5) \text{ \AA}^3$

$Z = 4$

$F_{000} = 664$

$D_x = 1.424 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2760 reflections

$\theta = 2.8\text{--}26.0^\circ$

$\mu = 0.39 \text{ mm}^{-1}$

$T = 298 (2) \text{ K}$

Block, colourless

$0.38 \times 0.38 \times 0.22 \text{ mm}$

Data collection

Bruker SMART APEX CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2) \text{ K}$

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2004)

$T_{\min} = 0.867, T_{\max} = 0.920$

4456 measured reflections

1437 independent reflections

1233 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\max} = 26.0^\circ$

$\theta_{\min} = 2.8^\circ$

$h = -14 \rightarrow 13$

$k = -10 \rightarrow 9$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.111$

$S = 1.06$

1437 reflections

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 0.3626P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.23 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$

88 parameters

Extinction correction: SHELXTL (Sheldrick, 2001),

$$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.0102 (14)

Secondary atom site location: difference Fourier map

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.47766 (16)	0.3496 (2)	0.66959 (12)	0.0598 (5)
H1A	0.5433	0.4147	0.6654	0.072*
H1B	0.4140	0.4203	0.6711	0.072*
C2	0.45113 (15)	0.2405 (2)	0.58925 (12)	0.0583 (5)
H2A	0.4486	0.3002	0.5330	0.070*
H2B	0.5090	0.1584	0.5931	0.070*
C3	0.38595 (16)	-0.1408 (2)	0.60957 (12)	0.0575 (5)
H3A	0.3680	-0.2459	0.5830	0.069*
H3B	0.4666	-0.1230	0.6142	0.069*
C4	0.35810 (16)	-0.1363 (2)	0.70316 (12)	0.0633 (5)
H4	0.3820	-0.0481	0.7400	0.076*
C5	0.3028 (2)	-0.2478 (3)	0.73597 (16)	0.0864 (7)
H5A	0.2778	-0.3374	0.7008	0.104*
H5B	0.2880	-0.2383	0.7948	0.104*
O1	0.5000	0.25490 (19)	0.7500	0.0588 (5)
O2	0.34023 (10)	0.16683 (15)	0.59020 (8)	0.0564 (4)
O3	0.19171 (12)	-0.01518 (16)	0.53390 (11)	0.0709 (4)
O4	0.34969 (13)	0.01089 (16)	0.45264 (9)	0.0689 (4)
S1	0.30943 (4)	0.00679 (5)	0.53678 (3)	0.0551 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0652 (12)	0.0545 (11)	0.0604 (10)	0.0051 (8)	0.0139 (8)	0.0084 (8)
C2	0.0543 (10)	0.0676 (12)	0.0549 (10)	0.0098 (8)	0.0153 (7)	0.0064 (8)
C3	0.0533 (10)	0.0643 (11)	0.0548 (9)	0.0123 (8)	0.0098 (7)	0.0046 (8)
C4	0.0588 (11)	0.0730 (13)	0.0572 (10)	0.0088 (9)	0.0091 (8)	0.0067 (9)
C5	0.0861 (17)	0.0939 (17)	0.0859 (15)	0.0038 (13)	0.0334 (12)	0.0152 (12)

supplementary materials

O1	0.0705 (12)	0.0506 (10)	0.0527 (9)	0.000	0.0054 (8)	0.000
O2	0.0527 (7)	0.0643 (8)	0.0539 (7)	0.0107 (6)	0.0147 (5)	-0.0047 (5)
O3	0.0506 (8)	0.0839 (10)	0.0737 (9)	0.0082 (6)	0.0008 (6)	0.0005 (7)
O4	0.0794 (11)	0.0815 (10)	0.0454 (7)	0.0187 (7)	0.0109 (6)	-0.0017 (5)
S1	0.0523 (3)	0.0662 (4)	0.0451 (3)	0.01240 (18)	0.0054 (2)	-0.00064 (17)

Geometric parameters (Å, °)

C1—O1	1.4170 (19)	C3—H3B	0.9700
C1—C2	1.489 (3)	C4—C5	1.293 (3)
C1—H1A	0.9700	C4—H4	0.9300
C1—H1B	0.9700	C5—H5A	0.9300
C2—O2	1.471 (2)	C5—H5B	0.9300
C2—H2A	0.9700	O1—C1 ⁱ	1.4169 (19)
C2—H2B	0.9700	O2—S1	1.5626 (13)
C3—C4	1.495 (2)	O3—S1	1.4196 (15)
C3—S1	1.7764 (18)	O4—S1	1.4279 (15)
C3—H3A	0.9700		
O1—C1—C2	108.31 (14)	S1—C3—H3B	109.3
O1—C1—H1A	110.0	H3A—C3—H3B	107.9
C2—C1—H1A	110.0	C5—C4—C3	123.9 (2)
O1—C1—H1B	110.0	C5—C4—H4	118.0
C2—C1—H1B	110.0	C3—C4—H4	118.0
H1A—C1—H1B	108.4	C4—C5—H5A	120.0
O2—C2—C1	107.61 (13)	C4—C5—H5B	120.0
O2—C2—H2A	110.2	H5A—C5—H5B	120.0
C1—C2—H2A	110.2	C1 ⁱ —O1—C1	112.15 (19)
O2—C2—H2B	110.2	C2—O2—S1	118.61 (10)
C1—C2—H2B	110.2	O3—S1—O4	118.74 (10)
H2A—C2—H2B	108.5	O3—S1—O2	105.25 (7)
C4—C3—S1	111.66 (13)	O4—S1—O2	109.83 (8)
C4—C3—H3A	109.3	O3—S1—C3	108.96 (9)
S1—C3—H3A	109.3	O4—S1—C3	109.21 (8)
C4—C3—H3B	109.3	O2—S1—C3	103.77 (8)

Symmetry codes: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C2—H2A \cdots O3 ⁱⁱ	0.97	2.53	3.219 (2)	128
C2—H2B \cdots O4 ⁱⁱⁱ	0.97	2.41	3.332 (2)	159
C3—H3A \cdots O3 ^{iv}	0.97	2.65	3.594 (2)	163
C5—H5B \cdots O2 ^v	0.93	2.64	3.447 (2)	146

Symmetry codes: (ii) $-x+1/2, -y+1/2, -z+1$; (iii) $-x+1, -y, -z+1$; (iv) $-x+1/2, -y-1/2, -z+1$; (v) $-x+1/2, y-1/2, -z+3/2$.

Fig. 1

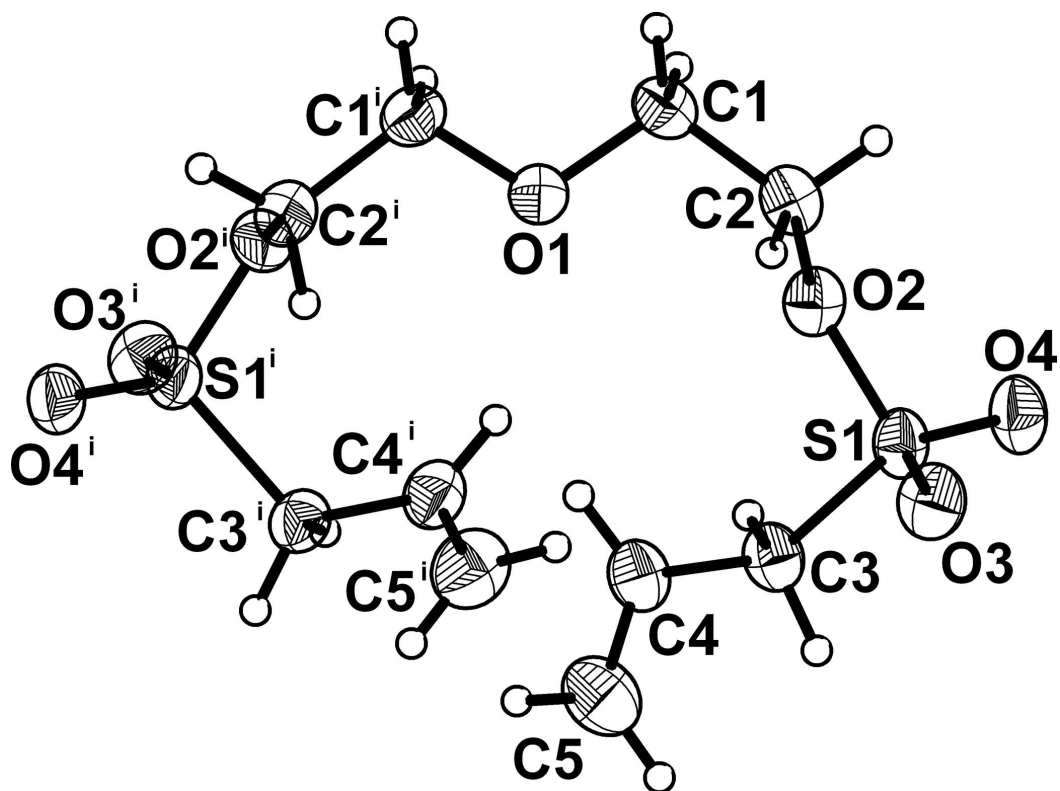


Fig. 2

