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(S)-(-)-Methyl 2-(*p*-tolylsulfonyloxy)-propanoate

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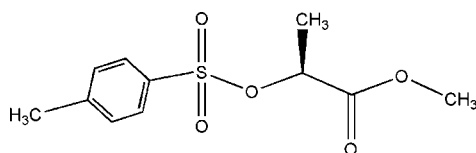
Received 31 March 2008; accepted 12 April 2008

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.064; wR factor = 0.154; data-to-parameter ratio = 16.8.

In the title compound, $\text{C}_{11}\text{H}_{14}\text{O}_5\text{S}$, there is an intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond, for which the $\text{C}-\text{C}-\text{S}-\text{O}$ torsion angle involving the acceptor and donor atoms is $2.4(4)^\circ$. The dihedral angle between the benzene ring and the methoxy-carbonyl plane is $52.7(4)^\circ$. In the crystal structure, molecules are linked *via* intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a molecular chain along the b axis.

Related literature

For related literature, see: Allen *et al.* (1987); Chan *et al.* (1975); Talbert *et al.* (1974).



Experimental

Crystal data

 $\text{C}_{11}\text{H}_{14}\text{O}_5\text{S}$ $M_r = 258.28$ Orthorhombic, $P2_12_12_1$ $a = 7.4890(15)$ Å $b = 10.150(2)$ Å $c = 17.362(4)$ Å $V = 1319.7(5)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.25$ mm⁻¹ $T = 298(2)$ K $0.40 \times 0.20 \times 0.20$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.906$, $T_{\max} = 0.951$
2933 measured reflections

2581 independent reflections
1703 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.154$
 $S = 1.01$
2581 reflections
154 parameters
H-atom parameters constrained

$\Delta\rho_{\max} = 0.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³
Absolute structure: Flack (1983),
1073 Friedel pairs
Flack parameter: 0.20 (16)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C4}-\text{H4A}\cdots\text{O1}$	0.93	2.53	2.910 (6)	104
$\text{C4}-\text{H4A}\cdots\text{O3}^i$	0.93	2.52	3.297 (5)	141
$\text{C6}-\text{H6A}\cdots\text{O1}^{ii}$	0.93	2.55	3.478 (6)	172

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

Data collection: *CAD-4 Software* (Enraf-Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: IS2285).

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supplementary materials

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(S)-(-)-Methyl 2-(*p*-tolylsulfonyloxy)propanoate

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Comment

(S)-(-)-methyl 2-(*p*-toluenesulfonyloxy)propanoate is an important fine chemical, which can be used for many fields such as chiral pesticide, organometallic chemistry, *etc.* (Talbert *et al.*, 1974). The bond lengths and angles of the title compound are within normal ranges (Allen *et al.*, 1987). In the crystal structure, molecules are linked *via* intermolecular C—H \cdots O hydrogen bonds, which with intramolecular C—H \cdots O hydrogen bonds seem to be effective in the stabilization of the crystal. As can be seen from the packing diagram (Fig. 2), the molecules are stacked along the *a* axis.

Experimental

The title compound was prepared according to the literature method (Chan *et al.*, 1975). The crystals were obtained by dissolving the title compound (500 mg, 2 mmol) in ethyl acetate (20 ml) and evaporating the solvent slowly at room temperature for about 7 d.

Refinement

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Figures

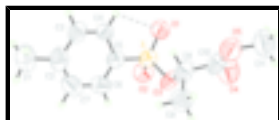


Fig. 1. A drawing of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The hydrogen bond is shown by dashed line.

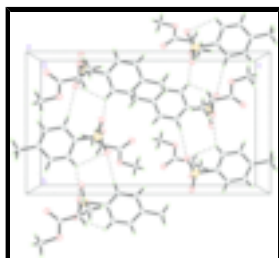


Fig. 2. A packing diagram of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

(S)-(-)-Methyl 2-(*p*-tolylsulfonyloxy)propanoate

Crystal data

C₁₁H₁₄O₅S
 $M_r = 258.28$

$F_{000} = 544$
 $D_x = 1.300 \text{ Mg m}^{-3}$

supplementary materials

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.4890$ (15) Å

$b = 10.150$ (2) Å

$c = 17.362$ (4) Å

$V = 1319.7$ (5) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9\text{--}14^\circ$

$\mu = 0.25$ mm⁻¹

$T = 298$ (2) K

Block, colorless

$0.40 \times 0.20 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.906$, $T_{\max} = 0.951$

2933 measured reflections

2581 independent reflections

1703 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.063$

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

$\theta_{\min} = 2.3^\circ$

$h = -9 \rightarrow 9$

$k = 0 \rightarrow 12$

$l = 0 \rightarrow 21$

3 standard reflections

every 200 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.063$

$wR(F^2) = 0.154$

$S = 1.01$

2581 reflections

154 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + P]$$

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

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

$\Delta\rho_{\max} = 0.31$ e Å⁻³

$\Delta\rho_{\min} = -0.29$ e Å⁻³

Extinction correction: none

Absolute structure: Flack (1983), 1073 Friedel pairs

Flack parameter: 0.20 (16)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S	0.06641 (15)	0.89958 (12)	0.71844 (7)	0.0628 (3)
O1	0.1083 (4)	0.7829 (3)	0.67754 (18)	0.0765 (10)
O2	0.1557 (5)	1.0219 (3)	0.7003 (2)	0.0830 (10)
O3	-0.1372 (4)	0.9339 (3)	0.70538 (18)	0.0689 (9)
O4	-0.3001 (6)	0.8618 (5)	0.5745 (2)	0.1125 (16)
O5	-0.4188 (5)	0.6878 (4)	0.6310 (2)	0.0984 (12)
C1	0.1382 (8)	0.7979 (7)	1.0589 (3)	0.102 (2)
H1B	0.1073	0.8750	1.0881	0.152*
H1C	0.2598	0.7741	1.0695	0.152*
H1D	0.0608	0.7266	1.0732	0.152*
C2	0.1173 (7)	0.8265 (7)	0.9740 (3)	0.0853 (17)
C3	0.1522 (6)	0.7282 (5)	0.9202 (3)	0.0723 (13)
H3A	0.1874	0.6457	0.9377	0.087*
C4	0.1371 (6)	0.7478 (4)	0.8434 (3)	0.0589 (11)
H4A	0.1627	0.6807	0.8086	0.071*
C5	0.0814 (5)	0.8730 (4)	0.8176 (3)	0.0561 (11)
C6	0.0417 (8)	0.9679 (5)	0.8676 (3)	0.0805 (14)
H6A	0.0034	1.0498	0.8500	0.097*
C7	0.0576 (9)	0.9444 (6)	0.9464 (3)	0.0910 (17)
H7A	0.0267	1.0106	0.9810	0.109*
C8	-0.2695 (6)	0.8272 (5)	0.7102 (3)	0.0715 (13)
H8A	-0.2174	0.7494	0.7350	0.086*
C9	-0.4204 (7)	0.8807 (6)	0.7576 (3)	0.0969 (17)
H9A	-0.3782	0.9008	0.8084	0.145*
H9B	-0.5139	0.8162	0.7607	0.145*
H9C	-0.4659	0.9593	0.7339	0.145*
C10	-0.3253 (6)	0.7955 (6)	0.6289 (3)	0.0758 (14)
C11	-0.5033 (10)	0.6511 (7)	0.5593 (4)	0.124 (3)
H11A	-0.5745	0.5736	0.5671	0.186*
H11B	-0.4134	0.6334	0.5213	0.186*
H11C	-0.5782	0.7219	0.5420	0.186*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0524 (6)	0.0593 (6)	0.0768 (7)	-0.0042 (6)	0.0029 (6)	0.0051 (6)
O1	0.077 (2)	0.080 (2)	0.072 (2)	0.0056 (18)	-0.0007 (17)	-0.0045 (17)
O2	0.078 (2)	0.078 (2)	0.093 (3)	-0.0125 (18)	0.0042 (18)	0.0086 (19)
O3	0.0576 (17)	0.0589 (18)	0.090 (2)	0.0055 (14)	-0.0090 (16)	0.0238 (16)
O4	0.142 (4)	0.119 (4)	0.076 (3)	-0.042 (3)	-0.004 (2)	0.026 (3)
O5	0.098 (3)	0.081 (2)	0.116 (3)	-0.011 (2)	-0.025 (2)	0.012 (2)
C1	0.092 (4)	0.137 (6)	0.075 (4)	-0.009 (4)	-0.007 (3)	-0.003 (4)

supplementary materials

C2	0.057 (3)	0.118 (5)	0.081 (4)	-0.014 (3)	-0.012 (3)	-0.005 (4)
C3	0.058 (3)	0.070 (3)	0.089 (4)	-0.009 (2)	-0.017 (2)	0.008 (3)
C4	0.061 (3)	0.043 (2)	0.072 (3)	-0.0061 (19)	-0.006 (2)	-0.001 (2)
C5	0.036 (2)	0.052 (3)	0.080 (3)	-0.0096 (19)	-0.002 (2)	0.001 (2)
C6	0.090 (4)	0.061 (3)	0.090 (4)	0.011 (3)	0.004 (3)	-0.005 (3)
C7	0.087 (4)	0.096 (4)	0.090 (4)	-0.012 (4)	0.007 (3)	-0.032 (3)
C8	0.053 (2)	0.076 (3)	0.085 (4)	-0.010 (2)	-0.008 (2)	0.023 (3)
C9	0.078 (3)	0.114 (4)	0.099 (4)	-0.010 (4)	0.017 (3)	0.005 (3)
C10	0.053 (3)	0.085 (4)	0.089 (4)	-0.005 (3)	-0.004 (3)	0.021 (3)
C11	0.140 (7)	0.111 (5)	0.121 (6)	-0.019 (5)	-0.036 (4)	-0.009 (4)

Geometric parameters (Å, °)

S—O1	1.416 (3)	C4—C5	1.411 (6)
S—O2	1.444 (3)	C4—H4A	0.9300
S—O3	1.581 (3)	C5—C6	1.331 (6)
S—C5	1.746 (5)	C6—C7	1.395 (8)
O3—C8	1.470 (5)	C6—H6A	0.9300
O4—C10	1.174 (6)	C7—H7A	0.9300
O5—C10	1.299 (6)	C8—C9	1.500 (7)
O5—C11	1.444 (7)	C8—C10	1.507 (7)
C1—C2	1.511 (7)	C8—H8A	0.9800
C1—H1B	0.9600	C9—H9A	0.9600
C1—H1C	0.9600	C9—H9B	0.9600
C1—H1D	0.9600	C9—H9C	0.9600
C2—C7	1.364 (8)	C11—H11A	0.9600
C2—C3	1.392 (8)	C11—H11B	0.9600
C3—C4	1.353 (6)	C11—H11C	0.9600
C3—H3A	0.9300		
O1—S—O2	120.5 (2)	C5—C6—H6A	120.1
O1—S—O3	109.0 (2)	C7—C6—H6A	120.1
O2—S—O3	103.07 (19)	C2—C7—C6	121.5 (5)
O1—S—C5	110.5 (2)	C2—C7—H7A	119.3
O2—S—C5	108.5 (2)	C6—C7—H7A	119.3
O3—S—C5	103.75 (18)	O3—C8—C9	105.8 (4)
C8—O3—S	118.7 (3)	O3—C8—C10	106.9 (4)
C10—O5—C11	115.4 (5)	C9—C8—C10	112.5 (4)
C2—C1—H1B	109.5	O3—C8—H8A	110.5
C2—C1—H1C	109.5	C9—C8—H8A	110.5
H1B—C1—H1C	109.5	C10—C8—H8A	110.5
C2—C1—H1D	109.5	C8—C9—H9A	109.5
H1B—C1—H1D	109.5	C8—C9—H9B	109.5
H1C—C1—H1D	109.5	H9A—C9—H9B	109.5
C7—C2—C3	117.0 (5)	C8—C9—H9C	109.5
C7—C2—C1	123.0 (6)	H9A—C9—H9C	109.5
C3—C2—C1	119.9 (6)	H9B—C9—H9C	109.5
C4—C3—C2	122.8 (5)	O4—C10—O5	126.3 (6)
C4—C3—H3A	118.6	O4—C10—C8	125.9 (5)
C2—C3—H3A	118.6	O5—C10—C8	107.6 (5)

C3—C4—C5	118.0 (4)	O5—C11—H11A	109.5
C3—C4—H4A	121.0	O5—C11—H11B	109.5
C5—C4—H4A	121.0	H11A—C11—H11B	109.5
C6—C5—C4	120.8 (4)	O5—C11—H11C	109.5
C6—C5—S	121.1 (4)	H11A—C11—H11C	109.5
C4—C5—S	118.1 (3)	H11B—C11—H11C	109.5
C5—C6—C7	119.8 (5)		
O1—S—O3—C8	44.2 (4)	C4—C5—C6—C7	0.9 (8)
O2—S—O3—C8	173.3 (3)	S—C5—C6—C7	-179.0 (4)
C5—S—O3—C8	-73.6 (4)	C3—C2—C7—C6	-3.5 (9)
C7—C2—C3—C4	3.2 (8)	C1—C2—C7—C6	179.3 (5)
C1—C2—C3—C4	-179.6 (5)	C5—C6—C7—C2	1.6 (9)
C2—C3—C4—C5	-0.8 (7)	S—O3—C8—C9	135.0 (4)
C3—C4—C5—C6	-1.3 (6)	S—O3—C8—C10	-104.8 (4)
C3—C4—C5—S	178.7 (3)	C11—O5—C10—O4	-2.2 (9)
O1—S—C5—C6	-177.6 (4)	C11—O5—C10—C8	172.4 (5)
O2—S—C5—C6	48.2 (5)	O3—C8—C10—O4	-15.7 (7)
O3—S—C5—C6	-60.9 (4)	C9—C8—C10—O4	100.0 (7)
O1—S—C5—C4	2.4 (4)	O3—C8—C10—O5	169.7 (4)
O2—S—C5—C4	-131.7 (3)	C9—C8—C10—O5	-74.6 (6)
O3—S—C5—C4	119.2 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4A \cdots O1	0.93	2.53	2.910 (6)	104
C4—H4A \cdots O3 ⁱ	0.93	2.52	3.297 (5)	141
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Symmetry codes: (i) $-x, y-1/2, -z+3/2$; (ii) $-x, y+1/2, -z+3/2$.

Fig. 1

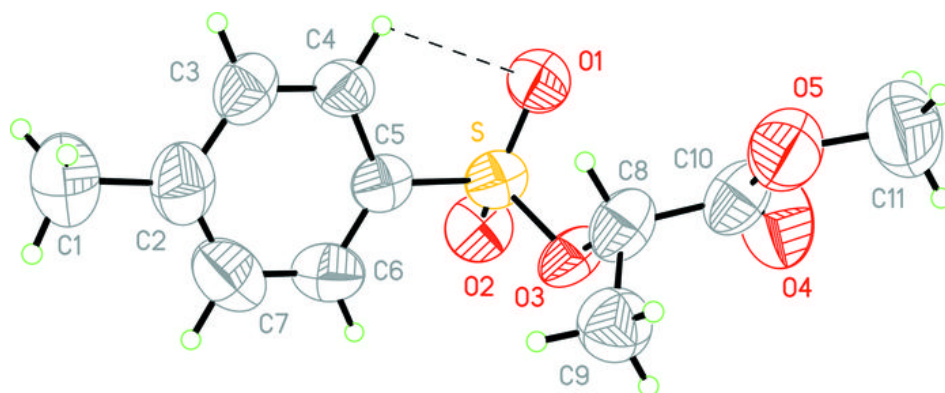


Fig. 2

