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Dimethyl 2-chloro-3-tosylmaleate

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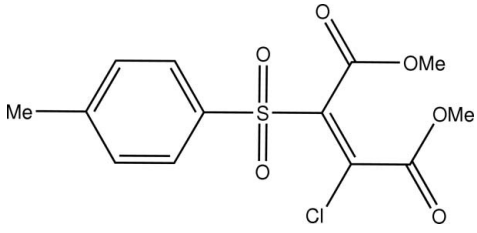
Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.127; data-to-parameter ratio = 17.0.

The title compound, $C_{13}H_{13}ClO_6S$, contains two methoxycarbonyl groups in a *cis* arrangement, with a dihedral angle of 79.9 (1)° between the least-squares planes defined by the two methoxycarbonyl fragments. Weak van der Waals interactions between the molecules are effective in the molecular packing. This is the first X-ray structure reported for a tosylmaleate derivative which can be used as a pharmaceutical primary material.

Related literature

For applications of vinylsulfonyl-containing compounds, see: Zhu *et al.* (1989).



Experimental

Crystal data

$C_{13}H_{13}ClO_6S$
M_r = 332.74
Monoclinic, $P2_1/c$
a = 12.4167 (14) Å
b = 12.9964 (13) Å
c = 9.5511 (10) Å
β = 100.879 (10)°
V = 1513.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 290 (2) K
0.5 × 0.2 × 0.1 mm

Data collection

Oxford Diffraction Xcalibur2 with a Sapphire-3 CCD detector diffractometer
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1997)
T_{min} = 0.815, T_{max} = 0.930
9792 measured reflections
3298 independent reflections
2475 reflections with $I > 2\sigma(I)$
R_{int} = 0.070

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.127
S = 0.99
3298 reflections
194 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected torsion angles (°).

C5—C1—C2—C3	−7.4 (3)	C5—C1—C2—Cl1	171.16 (14)
S1—C1—C2—C3	177.75 (12)	S1—C1—C2—Cl1	−3.7 (2)

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2129).

References

Brandenburg, K. (2001). *DIAMOND*. Version 2.1e. Crystal Impact GbR, Bonn, Germany.
Oxford Diffraction. (2003). *CrysAlis CCD* and *CrysAlis RED*. Version 1.171.29.2. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (1997). *X-RED*. Version 1.09. Stoe & Cie GmbH, Darmstadt, Germany.
Zhu, Z.-H., Chen, K.-C., Li, J.-X., Chen, S.-L. & Wang, S.-L. (1989). *Dyes Pigm.* **11**, 277–291.

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supplementary materials

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Dimethyl 2-chloro-3-tosylmaleate

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Comment

Vinylsulfonyl groups are valuable building blocks which can be used in fiber-reactive azo dyes, and also in plastic films and photographic materials (Zhu *et al.*, 1989). The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The two methoxycarbonyl groups are in *cis* configuration, shown by the torsion angle C5—C1—C2—C3 close to 0°. The interplanar angle between the least-squares planes defined by O6/C3/C2/O5/C4 and O4/C5/C1/O3/C6 is 79.9 (1)°. The four atoms connected to ethylene functionality (C1═C2) only slightly deviate from planarity. Relatively weak intermolecular van der Waals interactions are present between neighboring molecules, stabilizing the crystal structure.

Experimental

To a solution of dimethyl acetylenedicarboxylate (DMAD, 566 mg, 4.0 mmol) and *p*-toluenesulfonyl chloride (4.0 mmol) in dry THF (15 ml) under a nitrogen atmosphere, pyridine (48 mg, 0.6 mmol) was added and the reaction mixture was stirred for 16 h at room temperature. The solvent was evaporated and the residue was chromatographed on a silicagel column, using hexane-ethylacetate (90/10) as eluent, giving the pure product.

Refinement

All H atoms were positioned geometrically and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(carrier C) for aromatic and methyl group, respectively. C—H bond lengths were set to 0.93 (aromatic CH) and 0.96 Å (methyl CH_3), and methyl groups were allowed to rotate about their C—C σ bonds.

Figures

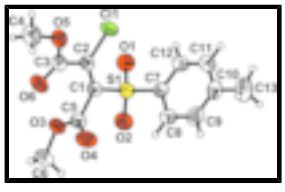

Fig. 1. Molecular structure of (I), with 50% probability displacement ellipsoids. H atoms are shown as circles of arbitrary radii.

Dimethyl 2-chloro-3-tosylmaleate

Crystal data

$C_{13}H_{13}ClO_6S$

M_r = 332.74

Monoclinic, $P2_1/c$

F_{000} = 688

D_x = 1.460 Mg m^{-3}

Mo $K\alpha$ radiation

λ = 0.71073 Å

Hall symbol: -P 2ybc	Cell parameters from 9792 reflections
a = 12.4167 (14) Å	θ = 3.8–32.1º
b = 12.9964 (13) Å	μ = 0.41 mm^{-1}
c = 9.5511 (10) Å	T = 290 (2) K
β = 100.879 (10)º	Needle, colourless
V = 1513.6 (3) $Å^3$	0.5 × 0.2 × 0.1 mm
Z = 4	

Data collection

Oxford Diffraction X-calibur2 with a Sapphire-3 CCD detector diffractometer	3298 independent reflections
Radiation source: fine-focus sealed tube	2475 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.070
Detector resolution: 12 pixels mm^{-1}	θ_{max} = 27.0º
T = 290(2) K	θ_{min} = 3.8º
ω scans at different φ	h = −15→11
Absorption correction: numerical (X-RED; Stoe & Cie, 1997)	k = −16→16
T_{min} = 0.815, T_{max} = 0.930	l = −12→12
9792 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$w = 1/[\sigma^2(F_o^2) + (0.0868P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.127	$(\Delta/\sigma)_{max} < 0.001$
S = 0.99	$\Delta\rho_{max}$ = 0.45 e $Å^{-3}$
3298 reflections	$\Delta\rho_{min}$ = −0.30 e $Å^{-3}$
194 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.040 (4)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
S1	0.29026 (4)	0.12068 (4)	0.04566 (5)	0.03976 (17)
Cl1	0.14986 (4)	0.12677 (4)	0.30129 (5)	0.04899 (19)
O1	0.23422 (11)	0.21700 (10)	0.04155 (14)	0.0519 (4)
O2	0.32811 (12)	0.08590 (13)	−0.07918 (14)	0.0561 (4)
O3	0.14474 (11)	−0.06627 (11)	−0.13420 (14)	0.0503 (4)
O4	0.26860 (14)	−0.14406 (13)	0.03464 (18)	0.0694 (5)

O5	0.03110 (11)	−0.05397 (11)	0.33034 (14)	0.0511 (4)
O6	0.03986 (14)	−0.12829 (11)	0.12083 (17)	0.0615 (4)
C1	0.20074 (14)	0.01944 (13)	0.08335 (18)	0.0362 (4)
C2	0.14118 (14)	0.02430 (13)	0.18619 (18)	0.0364 (4)
C3	0.06504 (14)	−0.06168 (14)	0.2081 (2)	0.0414 (4)
C4	−0.0495 (2)	−0.1296 (2)	0.3566 (3)	0.0689 (7)
H4A	−0.1066	−0.1351	0.2738	0.103*
H4B	−0.0805	−0.1082	0.4366	0.103*
H4C	−0.0145	−0.1952	0.3766	0.103*
C5	0.20747 (15)	−0.07510 (15)	−0.0063 (2)	0.0433 (4)
C6	0.1592 (2)	−0.1451 (2)	−0.2352 (3)	0.0730 (7)
H6A	0.2359	−0.1536	−0.2356	0.109*
H6B	0.1217	−0.1255	−0.3286	0.109*
H6C	0.1295	−0.2088	−0.2085	0.109*
C7	0.40081 (14)	0.11797 (14)	0.19050 (19)	0.0388 (4)
C8	0.47378 (16)	0.03585 (16)	0.2040 (2)	0.0505 (5)
H8	0.4648	−0.0168	0.1368	0.061*
C9	0.56025 (17)	0.03382 (18)	0.3196 (3)	0.0576 (6)
H9	0.6094	−0.0209	0.3293	0.069*
C10	0.57497 (16)	0.11112 (17)	0.4204 (2)	0.0513 (5)
C11	0.50243 (18)	0.19318 (18)	0.4025 (2)	0.0567 (5)
H11	0.5128	0.2467	0.4683	0.068*
C12	0.41541 (16)	0.19742 (16)	0.2894 (2)	0.0492 (5)
H12	0.3671	0.2528	0.2794	0.059*
C13	0.6695 (2)	0.1068 (2)	0.5454 (3)	0.0758 (8)
H13A	0.6709	0.0407	0.5905	0.114*
H13B	0.6607	0.1595	0.6127	0.114*
H13C	0.7371	0.1175	0.5125	0.114*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0468 (3)	0.0380 (3)	0.0358 (3)	−0.00102 (18)	0.01118 (19)	0.00405 (17)
Cl1	0.0638 (3)	0.0390 (3)	0.0490 (3)	−0.0072 (2)	0.0231 (2)	−0.01039 (19)
O1	0.0611 (8)	0.0384 (8)	0.0553 (8)	0.0041 (6)	0.0086 (7)	0.0135 (6)
O2	0.0679 (9)	0.0641 (10)	0.0404 (7)	−0.0087 (7)	0.0211 (7)	−0.0005 (7)
O3	0.0579 (8)	0.0505 (9)	0.0408 (7)	0.0002 (6)	0.0051 (6)	−0.0117 (6)
O4	0.0798 (11)	0.0528 (10)	0.0716 (11)	0.0271 (8)	0.0036 (8)	−0.0075 (8)
O5	0.0571 (8)	0.0542 (9)	0.0442 (8)	−0.0168 (6)	0.0148 (6)	−0.0004 (6)
O6	0.0792 (11)	0.0456 (9)	0.0629 (10)	−0.0202 (7)	0.0220 (8)	−0.0142 (7)
C1	0.0384 (9)	0.0351 (9)	0.0338 (9)	0.0020 (7)	0.0032 (7)	0.0015 (7)
C2	0.0391 (9)	0.0331 (9)	0.0355 (9)	0.0006 (7)	0.0036 (7)	−0.0006 (7)
C3	0.0447 (10)	0.0365 (10)	0.0425 (10)	−0.0002 (8)	0.0074 (8)	0.0030 (8)
C4	0.0793 (16)	0.0702 (17)	0.0635 (15)	−0.0296 (12)	0.0298 (12)	0.0013 (12)
C5	0.0459 (10)	0.0396 (11)	0.0451 (10)	0.0014 (8)	0.0101 (8)	−0.0040 (8)
C6	0.0771 (16)	0.0788 (18)	0.0636 (15)	−0.0067 (13)	0.0150 (12)	−0.0344 (13)
C7	0.0385 (9)	0.0402 (10)	0.0391 (9)	−0.0024 (7)	0.0112 (7)	0.0013 (7)
C8	0.0486 (11)	0.0470 (12)	0.0563 (12)	0.0059 (9)	0.0106 (9)	−0.0075 (9)

C9	0.0477 (11)	0.0534 (13)	0.0699 (14)	0.0085 (9)	0.0067 (10)	0.0039 (11)
C10	0.0436 (10)	0.0574 (13)	0.0519 (12)	−0.0085 (9)	0.0062 (9)	0.0077 (10)
C11	0.0583 (12)	0.0552 (13)	0.0552 (12)	−0.0025 (10)	0.0067 (10)	−0.0160 (10)
C12	0.0500 (11)	0.0412 (11)	0.0564 (12)	0.0035 (8)	0.0103 (9)	−0.0069 (9)
C13	0.0638 (15)	0.086 (2)	0.0697 (16)	−0.0091 (13)	−0.0085 (12)	0.0109 (14)

Geometric parameters (Å, °)

S1—O1	1.4291 (14)	C6—H6A	0.9600
S1—O2	1.4348 (15)	C6—H6B	0.9600
S1—C7	1.7557 (19)	C6—H6C	0.9600
S1—C1	1.8020 (18)	C7—C12	1.388 (3)
Cl1—C2	1.7172 (18)	C7—C8	1.390 (3)
O3—C5	1.324 (2)	C8—C9	1.387 (3)
O3—C6	1.442 (3)	C8—H8	0.9300
O4—C5	1.192 (2)	C9—C10	1.380 (3)
O5—C3	1.318 (2)	C9—H9	0.9300
O5—C4	1.457 (2)	C10—C11	1.385 (3)
O6—C3	1.201 (2)	C10—C13	1.509 (3)
C1—C2	1.338 (2)	C11—C12	1.377 (3)
C1—C5	1.509 (3)	C11—H11	0.9300
C2—C3	1.504 (2)	C12—H12	0.9300
C4—H4A	0.9600	C13—H13A	0.9600
C4—H4B	0.9600	C13—H13B	0.9600
C4—H4C	0.9600	C13—H13C	0.9600
O1—S1—O2	119.16 (9)	H6A—C6—H6B	109.5
O1—S1—C7	110.09 (9)	O3—C6—H6C	109.5
O2—S1—C7	108.57 (9)	H6A—C6—H6C	109.5
O1—S1—C1	109.24 (8)	H6B—C6—H6C	109.5
O2—S1—C1	104.31 (9)	C12—C7—C8	120.58 (18)
C7—S1—C1	104.36 (8)	C12—C7—S1	120.23 (15)
C5—O3—C6	115.46 (18)	C8—C7—S1	119.19 (15)
C3—O5—C4	116.47 (17)	C9—C8—C7	118.79 (19)
C2—C1—C5	123.54 (16)	C9—C8—H8	120.6
C2—C1—S1	124.02 (14)	C7—C8—H8	120.6
C5—C1—S1	112.27 (13)	C10—C9—C8	121.5 (2)
C1—C2—C3	121.07 (16)	C10—C9—H9	119.3
C1—C2—Cl1	122.01 (14)	C8—C9—H9	119.3
C3—C2—Cl1	116.91 (13)	C9—C10—C11	118.47 (19)
O6—C3—O5	125.76 (17)	C9—C10—C13	120.4 (2)
O6—C3—C2	121.84 (17)	C11—C10—C13	121.1 (2)
O5—C3—C2	112.40 (16)	C12—C11—C10	121.6 (2)
O5—C4—H4A	109.5	C12—C11—H11	119.2
O5—C4—H4B	109.5	C10—C11—H11	119.2
H4A—C4—H4B	109.5	C11—C12—C7	119.09 (19)
O5—C4—H4C	109.5	C11—C12—H12	120.5
H4A—C4—H4C	109.5	C7—C12—H12	120.5
H4B—C4—H4C	109.5	C10—C13—H13A	109.5
O4—C5—O3	126.52 (19)	C10—C13—H13B	109.5

O4—C5—C1	121.76 (18)	H13A—C13—H13B	109.5
O3—C5—C1	111.53 (16)	C10—C13—H13C	109.5
O3—C6—H6A	109.5	H13A—C13—H13C	109.5
O3—C6—H6B	109.5	H13B—C13—H13C	109.5
O1—S1—C1—C2	−46.44 (17)	S1—C1—C5—O4	92.7 (2)
O2—S1—C1—C2	−174.88 (15)	C2—C1—C5—O3	101.9 (2)
C7—S1—C1—C2	71.26 (16)	S1—C1—C5—O3	−82.72 (16)
O1—S1—C1—C5	138.23 (12)	O1—S1—C7—C12	6.95 (18)
O2—S1—C1—C5	9.79 (15)	O2—S1—C7—C12	139.03 (16)
C7—S1—C1—C5	−104.07 (13)	C1—S1—C7—C12	−110.17 (16)
C5—C1—C2—C3	−7.4 (3)	O1—S1—C7—C8	−172.52 (15)
S1—C1—C2—C3	177.75 (12)	O2—S1—C7—C8	−40.44 (18)
C5—C1—C2—Cl1	171.16 (14)	C1—S1—C7—C8	70.36 (17)
S1—C1—C2—Cl1	−3.7 (2)	C12—C7—C8—C9	1.2 (3)
C4—O5—C3—O6	−4.2 (3)	S1—C7—C8—C9	−179.35 (16)
C4—O5—C3—C2	175.59 (17)	C7—C8—C9—C10	0.0 (3)
C1—C2—C3—O6	−13.4 (3)	C8—C9—C10—C11	−1.5 (3)
Cl1—C2—C3—O6	167.92 (16)	C8—C9—C10—C13	179.8 (2)
C1—C2—C3—O5	166.77 (16)	C9—C10—C11—C12	1.8 (3)
Cl1—C2—C3—O5	−11.9 (2)	C13—C10—C11—C12	−179.5 (2)
C6—O3—C5—O4	−4.8 (3)	C10—C11—C12—C7	−0.6 (3)
C6—O3—C5—C1	170.36 (18)	C8—C7—C12—C11	−0.9 (3)
C2—C1—C5—O4	−82.7 (3)	S1—C7—C12—C11	179.65 (16)

Fig. 1