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Methyl 4-methylsulfonyl-2-nitrobenzoate

Yan-Jun Hou, Wen-Yi Chu, Jun Sui and Zhi-Zhong Sun*

College of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China
Correspondence e-mail: hljusunzhizhong@163.com

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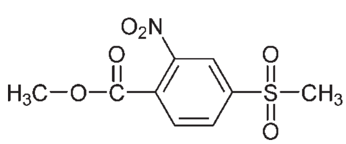
Key indicators: single-crystal X-ray study; *T* = 273 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.041; *wR* factor = 0.111; data-to-parameter ratio = 17.8.

The title compound, $C_9H_9NO_6S$, was prepared by the reaction of methanol and thionyl chloride with 4-methylsulfonyl-2-nitrobenzoic acid under mild conditions. The dihedral angle between the nitro group and benzene ring is 21.33 (19)° and that between the carboxylate group and the benzene ring is 72.09 (17)°. The crystal structure is stabilized by weak intermolecular bifurcated C—H⋯O interactions occurring in the (100) plane.

Related literature

For general background to the synthesis and properties of 4-methylsulfonyl-2-nitro-benzoic acid methyl ester, see: Carter *et al.* (1991). For the biological activity of 4-methylsulfonyl-2-nitro-benzoic acid methyl ester derivatives, see: Kopsell *et al.* (2009).



Experimental

Crystal data

$C_9H_9NO_6S$
M_r = 259.23
Monoclinic, $P2_1/c$
a = 9.0108 (12) Å
b = 8.7671 (11) Å
c = 14.4761 (19) Å
β = 98.955 (2)°
V = 1129.7 (3) Å^3
Z = 4
Mo *K*α radiation
μ = 0.30 mm^{-1}
T = 273 K
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEXII CCD detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.942, T_{max} = 0.948
9661 measured reflections
2783 independent reflections
2042 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.111
S = 1.05
2783 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C6—H6⋯O2^{i}	0.93	2.54	3.370 (2)	148
C6—H6⋯O3^{ii}	0.93	2.59	3.216 (2)	125

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; 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: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2147).

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supporting information

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S1. Comment

4-methylsulfonyl-2-nitro-benzoic acid methyl ester is used for preparation of mesotrione, which inhibits a critical enzyme, phytoene desaturase, in plant carotenoidbiosynthesis (Kopsell *et al.*, 2009).

The structure of the title compound is shown in Fig. 1. The dihedral angle between the nitro group and benzene ring is 21.33 (19)°. The dihedral angle between the carboxyl group and benzene ring is 72.09 (17)°. The crystal structure is stabilized by weak intermolecular bifurcated C—H···O interactions (the sum of the angles involving H6 as the central atom is 360 (3)°) occurring in the (100) plane (Table 1), resulting in a two-dimensional network (Fig. 2).

S2. Experimental

Thionyl chloride (250 mmol) was added to a solution of 4-methylsulfonyl-2-nitro-benzoic acid (50 mmol) in anhydrous toluene (250 ml). After stirring the reaction mixture for 10 h at room temperature, the solvent was removed and methanol (100 ml) was added. The reaction mixture was further stirred for 3 h at 323 K. The resulting oil was washed with water (100 ml). After separation from the water phase, the product was concentrated under reduced pressure and the residue was recrystallized from methanol to give the title compound in a yield of 80% (Carter *et al.*, 1991). Crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from ethanol at room temperature in a yield of 60%. Analysis found: C 41.7, H 3.4, N 5.3%; $C_9H_9NO_6S$ requires: C 41.7, H 3.5, N 5.4%.

S3. Refinement

All H atoms were placed in idealized positions [C—H = 0.96 (methyl) and 0.93 Å(aromatic)] and included in the refinement in the riding-model approximation, with U_{iso}(H)= 1.5 U_{eq}(methyl C) and 1.2 U_{eq}(aromatic C).

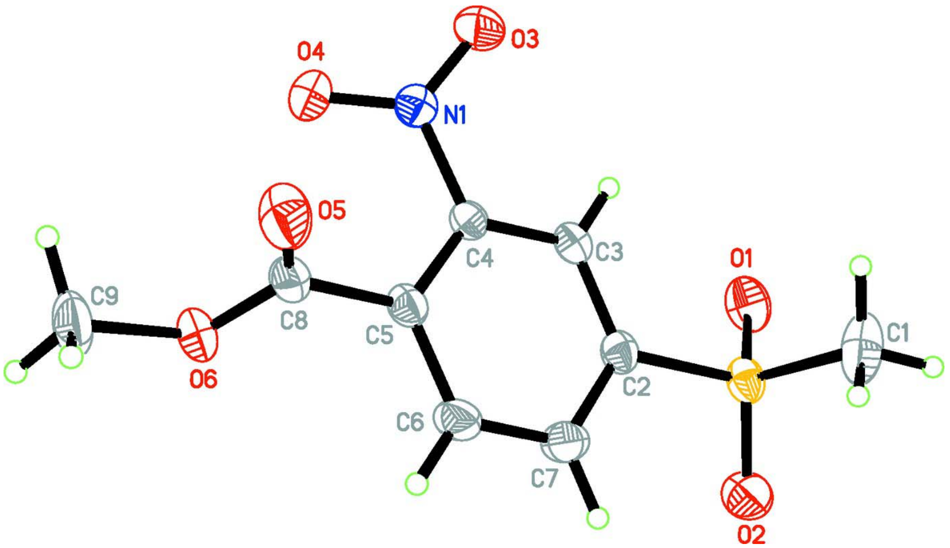

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

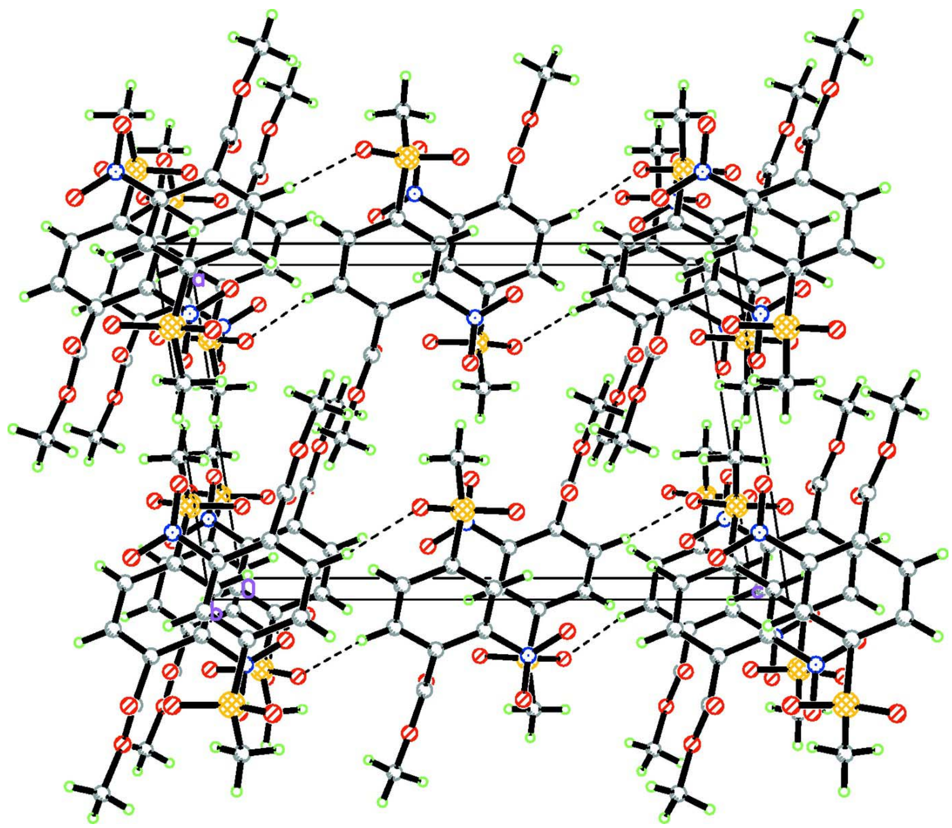

Figure 2

Part of packing of the crystal structure of the title compound, viewed down the b direction. Dashed lines indicate hydrogen bonds.

Methyl 4-methylsulfonyl-2-nitrobenzoate

Crystal data

$C_9H_9NO_6S$
M_r = 259.23
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.0108 (12) Å
b = 8.7671 (11) Å
c = 14.4761 (19) Å
β = 98.955 (2)°
V = 1129.7 (3) Å^3
Z = 4
$F(000)$ = 536
D_x = 1.524 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2311 reflections
θ = 2.9–25.0°
μ = 0.30 mm^{-1}
T = 273 K
Block, colorless
0.20 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEX CCD detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.942, T_{max} = 0.948
9661 measured reflections
2783 independent reflections
2042 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 28.3°, θ_{min} = 2.7°
h = −11→11
k = −11→10
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.111
S = 1.05
2783 reflections
156 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
$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.3279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 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	$U_{iso}*/U_{eq}$
S1	−0.24972 (5)	0.42896 (5)	0.03839 (4)	0.04280 (16)
O6	0.42147 (14)	0.11875 (17)	0.20037 (9)	0.0491 (4)
O5	0.28877 (18)	−0.09358 (17)	0.15947 (13)	0.0665 (5)
O4	0.32570 (17)	0.07267 (19)	−0.01616 (11)	0.0608 (4)

O3	0.12224 (18)	0.08499 (19)	−0.11609 (9)	0.0581 (4)
O2	−0.26056 (19)	0.53991 (19)	0.10923 (12)	0.0694 (5)
O1	−0.24582 (17)	0.48263 (19)	−0.05419 (11)	0.0613 (4)
N1	0.19276 (18)	0.10350 (18)	−0.03781 (11)	0.0399 (4)
C1	−0.3960 (2)	0.2976 (3)	0.0338 (2)	0.0737 (8)
H1A	−0.4905	0.3496	0.0188	0.111*
H1B	−0.3909	0.2482	0.0934	0.111*
H1C	−0.3872	0.2227	−0.0134	0.111*
C2	−0.0851 (2)	0.3192 (2)	0.07342 (12)	0.0383 (4)
C4	0.11351 (19)	0.16778 (19)	0.03452 (11)	0.0333 (4)
C3	−0.01499 (19)	0.2509 (2)	0.00566 (12)	0.0361 (4)
H3	−0.0535	0.2607	−0.0575	0.043*
C5	0.1726 (2)	0.1479 (2)	0.12854 (12)	0.0380 (4)
C8	0.3013 (2)	0.0425 (2)	0.16253 (13)	0.0417 (4)
C7	−0.0276 (2)	0.3049 (3)	0.16756 (13)	0.0502 (5)
H7	−0.0750	0.3526	0.2124	0.060*
C6	0.1005 (2)	0.2193 (2)	0.19439 (13)	0.0489 (5)
H6	0.1388	0.2094	0.2576	0.059*
C9	0.5498 (2)	0.0281 (3)	0.24167 (17)	0.0684 (7)
H9A	0.5205	−0.0387	0.2882	0.103*
H9B	0.6285	0.0945	0.2703	0.103*
H9C	0.5853	−0.0313	0.1939	0.103*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0366 (3)	0.0341 (3)	0.0543 (3)	0.00290 (19)	−0.0033 (2)	0.0009 (2)
O6	0.0375 (7)	0.0557 (9)	0.0497 (8)	−0.0009 (6)	−0.0073 (6)	0.0037 (6)
O5	0.0590 (10)	0.0406 (9)	0.0924 (12)	0.0075 (7)	−0.0115 (9)	0.0010 (8)
O4	0.0422 (8)	0.0762 (11)	0.0641 (10)	0.0123 (8)	0.0087 (7)	−0.0088 (8)
O3	0.0607 (9)	0.0771 (11)	0.0362 (8)	0.0002 (8)	0.0061 (7)	−0.0069 (7)
O2	0.0670 (11)	0.0601 (10)	0.0752 (11)	0.0246 (8)	−0.0075 (8)	−0.0210 (8)
O1	0.0565 (10)	0.0612 (10)	0.0608 (9)	0.0055 (8)	−0.0081 (7)	0.0182 (8)
N1	0.0409 (9)	0.0378 (9)	0.0414 (9)	−0.0030 (7)	0.0072 (7)	0.0010 (6)
C1	0.0395 (12)	0.0476 (14)	0.134 (2)	−0.0002 (10)	0.0131 (13)	0.0101 (14)
C2	0.0344 (9)	0.0383 (10)	0.0400 (10)	0.0025 (8)	−0.0006 (7)	0.0024 (7)
C4	0.0332 (9)	0.0318 (9)	0.0341 (9)	−0.0039 (7)	0.0029 (7)	−0.0008 (7)
C3	0.0353 (9)	0.0368 (10)	0.0341 (9)	−0.0032 (7)	−0.0010 (7)	0.0025 (7)
C5	0.0376 (9)	0.0357 (10)	0.0380 (9)	0.0016 (7)	−0.0022 (7)	−0.0004 (7)
C8	0.0387 (10)	0.0458 (12)	0.0383 (10)	0.0032 (8)	−0.0015 (7)	0.0010 (8)
C7	0.0546 (12)	0.0569 (13)	0.0381 (10)	0.0170 (10)	0.0038 (9)	−0.0044 (9)
C6	0.0561 (12)	0.0555 (12)	0.0318 (9)	0.0145 (10)	−0.0037 (8)	−0.0028 (9)
C9	0.0412 (12)	0.0959 (19)	0.0622 (14)	0.0130 (12)	−0.0100 (10)	0.0143 (13)

Geometric parameters (Å, º)

S1—O1	1.4260 (16)	C2—C3	1.383 (3)
S1—O2	1.4278 (16)	C2—C7	1.386 (2)

S1—C1	1.744 (2)	C4—C3	1.377 (2)
S1—C2	1.7749 (18)	C4—C5	1.393 (2)
O6—C8	1.317 (2)	C3—H3	0.9300
O6—C9	1.453 (2)	C5—C6	1.384 (3)
O5—C8	1.198 (2)	C5—C8	1.504 (3)
O4—N1	1.220 (2)	C7—C6	1.381 (3)
O3—N1	1.221 (2)	C7—H7	0.9300
N1—C4	1.469 (2)	C6—H6	0.9300
C1—H1A	0.9600	C9—H9A	0.9600
C1—H1B	0.9600	C9—H9B	0.9600
C1—H1C	0.9600	C9—H9C	0.9600

O1—S1—O2	117.69 (11)	C4—C3—C2	117.99 (15)
O1—S1—C1	108.11 (12)	C4—C3—H3	121.0
O2—S1—C1	109.99 (13)	C2—C3—H3	121.0
O1—S1—C2	107.80 (9)	C6—C5—C4	117.83 (16)
O2—S1—C2	108.17 (9)	C6—C5—C8	118.24 (16)
C1—S1—C2	104.24 (10)	C4—C5—C8	123.69 (17)
C8—O6—C9	116.36 (18)	O5—C8—O6	125.95 (18)
O4—N1—O3	123.86 (17)	O5—C8—C5	122.42 (17)
O4—N1—C4	117.96 (15)	O6—C8—C5	111.49 (17)
O3—N1—C4	118.18 (15)	C6—C7—C2	119.59 (18)
S1—C1—H1A	109.5	C6—C7—H7	120.2
S1—C1—H1B	109.5	C2—C7—H7	120.2
H1A—C1—H1B	109.5	C7—C6—C5	120.91 (17)
S1—C1—H1C	109.5	C7—C6—H6	119.5
H1A—C1—H1C	109.5	C5—C6—H6	119.5
H1B—C1—H1C	109.5	O6—C9—H9A	109.5
C3—C2—C7	121.07 (17)	O6—C9—H9B	109.5
C3—C2—S1	119.07 (13)	H9A—C9—H9B	109.5
C7—C2—S1	119.85 (15)	O6—C9—H9C	109.5
C3—C4—C5	122.57 (16)	H9A—C9—H9C	109.5
C3—C4—N1	117.78 (15)	H9B—C9—H9C	109.5
C5—C4—N1	119.60 (15)		

O1—S1—C2—C3	−25.21 (18)	N1—C4—C5—C6	175.52 (17)
O2—S1—C2—C3	−153.45 (16)	C3—C4—C5—C8	172.23 (17)
C1—S1—C2—C3	89.52 (18)	N1—C4—C5—C8	−10.2 (3)
O1—S1—C2—C7	153.91 (17)	C9—O6—C8—O5	0.2 (3)
O2—S1—C2—C7	25.7 (2)	C9—O6—C8—C5	175.83 (17)
C1—S1—C2—C7	−91.4 (2)	C6—C5—C8—O5	103.2 (2)
O4—N1—C4—C3	157.53 (17)	C4—C5—C8—O5	−71.0 (3)
O3—N1—C4—C3	−22.0 (2)	C6—C5—C8—O6	−72.6 (2)
O4—N1—C4—C5	−20.2 (2)	C4—C5—C8—O6	113.1 (2)
O3—N1—C4—C5	160.31 (17)	C3—C2—C7—C6	−0.9 (3)
C5—C4—C3—C2	1.4 (3)	S1—C2—C7—C6	179.96 (17)
N1—C4—C3—C2	−176.25 (15)	C2—C7—C6—C5	0.2 (3)
C7—C2—C3—C4	0.2 (3)	C4—C5—C6—C7	1.2 (3)

S1—C2—C3—C4	179.26 (13)	C8—C5—C6—C7	−173.4 (2)
C3—C4—C5—C6	−2.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.93	2.54	3.370 (2)	148
C6—H6···O3^{ii}	0.93	2.59	3.216 (2)	125

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