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N-Methyl-*N*-methylsulfonyl-2-nitrobenzenesulfonamide

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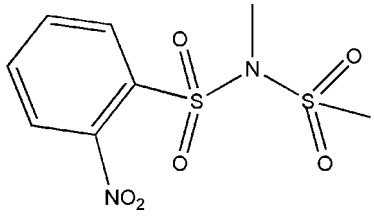
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.024; wR factor = 0.065; data-to-parameter ratio = 12.4.

In the title compound, $C_8H_{10}N_2O_6S_2$, all bond lengths and angles are normal. The molecules are packed into a three-dimensional network by intermolecular C—H···O hydrogen bonds and weak π–π aromatic stacking interactions [centroid separation = 4.078 (2) Å].

Related literature

For literature on the biological activities of sulfonimide compounds, see: Kamoshita *et al.* (1987). For the crystal structure of related compounds, see: Henschel *et al.* (1996). For related literature, see: Allen *et al.* (1987); Zhang *et al.* (2007).



Experimental

Crystal data

$C_8H_{10}N_2O_6S_2$
M_r = 294.30
Monoclinic, $P2_1/c$
a = 6.9540 (14) Å
b = 20.492 (4) Å
c = 8.1272 (16) Å
β = 92.53 (3)°
V = 1157.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.48 mm^{-1}
T = 153 (2) K
0.44 × 0.23 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.818, T_{max} = 0.945
9002 measured reflections
2040 independent reflections
1937 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.065
S = 1.08
2040 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O3	0.95	2.40	2.807 (2)	106
C8—H8A···O4	0.98	2.30	2.811 (2)	112
C9—H9B···O3	0.98	2.60	3.243 (2)	124
C2—H2A···O2^{i}	0.95	2.58	3.435 (2)	149
C4—H4B···O6^{ii}	0.95	2.52	3.261 (2)	135
C8—H8A···O5^{iii}	0.98	2.50	3.194 (2)	128

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2153).

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supplementary materials

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N-Methyl-*N*-methylsulfonyl-2-nitrobenzenesulfonamide

X.-Y. Li, Z.-W. Song, W.-P. Shi and Z.-Q. Hu

Comment

Many compounds containing sulfonimide groups possess a broad spectrum of biological activities and can be widely used as herbicides (Kamoshita *et al.*, 1987). Their use as catalysts is also reported (Zhang *et al.*, 2007). Here, we report the synthesis and crystal structure of the title compound.

In the title compound (Fig. 1), all bond lengths and angles are normal (Allen *et al.*, 1987) and in good agreement with those reported previously for similar molecules (Henschel *et al.*, 1996). The molecular structure is stabilized by intramolecular C—H···O hydrogen bonds involving the oxygen atoms bound to atom S1 (Table 1). In the crystal structure (Fig. 2), molecules are linked into a three-dimensional network by weak intermolecular C—H···O hydrogen bonds and aromatic π-π stacking interactions ($Cg\cdots Cg^{i}$ = 4.078 (2) Å; *Cg* is the centroid of the C1—C6 benzene ring; symmetry code: (i) $x, 3/2-y, -1/2+z$).

Experimental

A solution of methylsulfonyl chloride (1 mmol) dissolved in anhydrous CH_2Cl_2 (10 ml) was added dropwise over a period of 10 min to a solution of 2-nitro-*N*-methyl-benzenesulfonamide (1 mmol) and $EtN(^{i}Pr)_2$ (3 mmol) in CH_2Cl_2 (10 ml) at 273 K. The mixture was stirred at room temperature for 4 h. The organic phase was washed twice with 2 N HCl and dried over anhydrous Na_2SO_4. The solvent was removed and the residue was purified by flash chromatography (1:1 cyclohexane/dichloromethane) to give the title compound as a white solid (147 mg, yield 64%). Single crystals suitable for X-ray measurements were obtained by slow evaporation of an ethanol/dichloromethane solution (1:1 *v*/*v*) at room temperature.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95–0.98 Å and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) for methyl groups.

Figures

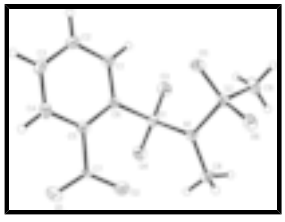

Fig. 1. The molecular structure of the title compound, with atom labels and 40% probability displacement ellipsoids.

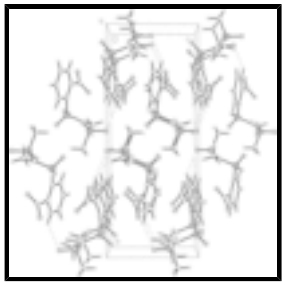

Fig. 2. Packing diagram of the title compound viewed down the *a* axis, showing the intermolecular C—H···O hydrogen bonding network (dashed lines).

N-Methyl-*N*-methylsulfonyl-2-nitrobenzenesulfonamide

Crystal data

$C_8H_{10}N_2O_6S_2$	F_{000} = 608
M_r = 294.30	D_x = 1.689 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2541 reflections
a = 6.9540 (14) Å	θ = 6.2–55.0º
b = 20.492 (4) Å	μ = 0.48 mm^{-1}
c = 8.1272 (16) Å	T = 153 (2) K
β = 92.53 (3)º	Block, colourless
V = 1157.0 (4) Å^3	0.44 × 0.23 × 0.12 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	2040 independent reflections
Radiation source: Rotating Anode	1937 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.018
T = 153(2) K	θ_{max} = 25.0º
ω oscillation scans	θ_{min} = 3.1º
Absorption correction: multi-scan (ABSCOR; Higashi 1995)	h = −8→7
T_{min} = 0.818, T_{max} = 0.945	k = −24→24
9002 measured reflections	l = −9→9

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.024	$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2 + 0.8042P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.065	$(\Delta/\sigma)_{max}$ = 0.002
S = 1.08	$\Delta\rho_{max}$ = 0.32 e Å^{-3}

2040 reflections	$\Delta\rho_{min} = -0.32$ e Å^{-3}
164 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: 0.0257 (16)
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 $F^2 > 2\text{sigma}(F^2)$ 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	0.36867 (5)	0.613945 (18)	0.45674 (5)	0.01797 (13)
S2	0.17510 (5)	0.544118 (18)	0.18890 (5)	0.01806 (13)
O1	0.55828 (18)	0.80933 (6)	0.35423 (15)	0.0297 (3)
O2	0.52506 (17)	0.71668 (6)	0.22789 (14)	0.0270 (3)
O3	0.27241 (18)	0.56917 (6)	0.56005 (14)	0.0278 (3)
O4	0.56186 (16)	0.63251 (6)	0.49936 (14)	0.0256 (3)
O5	0.01285 (16)	0.57654 (6)	0.25138 (15)	0.0256 (3)
O6	0.19992 (17)	0.54204 (6)	0.01525 (14)	0.0255 (3)
N1	0.37021 (18)	0.58253 (6)	0.26787 (16)	0.0185 (3)
N2	0.47199 (19)	0.75836 (7)	0.32402 (16)	0.0196 (3)
C1	0.0432 (2)	0.68091 (9)	0.51230 (19)	0.0229 (4)
H1A	−0.0040	0.6393	0.5429	0.027*
C2	−0.0693 (2)	0.73588 (9)	0.5334 (2)	0.0275 (4)
H2A	−0.1934	0.7318	0.5766	0.033*
C3	−0.0008 (3)	0.79664 (9)	0.4915 (2)	0.0279 (4)
H3B	−0.0772	0.8343	0.5079	0.033*
C4	0.1787 (2)	0.80291 (8)	0.4258 (2)	0.0240 (4)
H4B	0.2259	0.8447	0.3971	0.029*
C5	0.2881 (2)	0.74774 (8)	0.40254 (18)	0.0176 (3)
C6	0.2241 (2)	0.68580 (8)	0.44701 (18)	0.0172 (3)
C8	0.5563 (2)	0.57088 (9)	0.1902 (2)	0.0275 (4)
H8A	0.6578	0.5961	0.2483	0.041*
H8B	0.5458	0.5845	0.0745	0.041*
H8C	0.5880	0.5243	0.1963	0.041*
C9	0.1866 (3)	0.46428 (8)	0.2671 (2)	0.0297 (4)
H9A	0.0748	0.4394	0.2244	0.045*
H9B	0.1866	0.4656	0.3876	0.045*

H9C	0.3049	0.4432	0.2328	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0191 (2)	0.0174 (2)	0.0173 (2)	0.00059 (15)	−0.00127 (15)	0.00110 (14)
S2	0.0168 (2)	0.0145 (2)	0.0225 (2)	−0.00014 (14)	−0.00308 (15)	0.00002 (14)
O1	0.0297 (7)	0.0259 (7)	0.0335 (7)	−0.0118 (5)	0.0004 (5)	0.0025 (5)
O2	0.0259 (6)	0.0306 (7)	0.0253 (6)	−0.0021 (5)	0.0078 (5)	−0.0032 (5)
O3	0.0357 (7)	0.0235 (6)	0.0243 (6)	−0.0020 (5)	0.0034 (5)	0.0068 (5)
O4	0.0219 (6)	0.0263 (6)	0.0278 (6)	0.0009 (5)	−0.0085 (5)	−0.0014 (5)
O5	0.0154 (5)	0.0256 (6)	0.0355 (7)	0.0006 (5)	−0.0006 (5)	−0.0029 (5)
O6	0.0306 (6)	0.0227 (6)	0.0229 (6)	−0.0026 (5)	−0.0038 (5)	−0.0019 (5)
N1	0.0133 (6)	0.0201 (7)	0.0220 (7)	0.0000 (5)	0.0017 (5)	−0.0045 (5)
N2	0.0202 (7)	0.0214 (7)	0.0170 (6)	−0.0029 (6)	−0.0020 (5)	0.0039 (5)
C1	0.0219 (8)	0.0258 (9)	0.0212 (8)	−0.0041 (7)	0.0045 (7)	−0.0030 (7)
C2	0.0193 (8)	0.0362 (10)	0.0275 (9)	0.0001 (7)	0.0048 (7)	−0.0101 (8)
C3	0.0233 (8)	0.0263 (9)	0.0335 (9)	0.0064 (7)	−0.0038 (7)	−0.0122 (7)
C4	0.0253 (8)	0.0194 (8)	0.0266 (8)	−0.0001 (7)	−0.0053 (7)	−0.0040 (7)
C5	0.0168 (7)	0.0212 (8)	0.0145 (7)	−0.0020 (6)	−0.0020 (6)	−0.0016 (6)
C6	0.0185 (8)	0.0186 (8)	0.0145 (7)	0.0006 (6)	0.0002 (6)	−0.0026 (6)
C8	0.0164 (8)	0.0332 (10)	0.0336 (9)	0.0025 (7)	0.0063 (7)	−0.0085 (8)
C9	0.0372 (10)	0.0156 (8)	0.0356 (10)	−0.0030 (7)	−0.0075 (8)	0.0044 (7)

Geometric parameters (Å, °)

S1—O4	1.4242 (12)	C1—H1A	0.9500
S1—O3	1.4299 (12)	C2—C3	1.381 (3)
S1—N1	1.6650 (13)	C2—H2A	0.9500
S1—C6	1.7829 (16)	C3—C4	1.385 (3)
S2—O5	1.4222 (12)	C3—H3B	0.9500
S2—O6	1.4300 (13)	C4—C5	1.380 (2)
S2—N1	1.6721 (14)	C4—H4B	0.9500
S2—C9	1.7556 (17)	C5—C6	1.398 (2)
O1—N2	1.2241 (18)	C8—H8A	0.9800
O2—N2	1.2254 (18)	C8—H8B	0.9800
N1—C8	1.484 (2)	C8—H8C	0.9800
N2—C5	1.470 (2)	C9—H9A	0.9800
C1—C2	1.386 (2)	C9—H9B	0.9800
C1—C6	1.390 (2)	C9—H9C	0.9800
O4—S1—O3	119.31 (8)	C2—C3—C4	120.33 (16)
O4—S1—N1	106.45 (7)	C2—C3—H3B	119.8
O3—S1—N1	108.38 (7)	C4—C3—H3B	119.8
O4—S1—C6	108.32 (7)	C5—C4—C3	119.24 (16)
O3—S1—C6	106.16 (7)	C5—C4—H4B	120.4
N1—S1—C6	107.76 (7)	C3—C4—H4B	120.4
O5—S2—O6	119.90 (8)	C4—C5—C6	121.63 (15)
O5—S2—N1	106.62 (7)	C4—C5—N2	115.69 (14)

O6—S2—N1	105.17 (7)	C6—C5—N2	122.66 (14)
O5—S2—C9	109.29 (9)	C1—C6—C5	117.90 (14)
O6—S2—C9	108.90 (8)	C1—C6—S1	116.20 (12)
N1—S2—C9	106.05 (8)	C5—C6—S1	125.17 (12)
C8—N1—S1	119.72 (11)	N1—C8—H8A	109.5
C8—N1—S2	117.96 (11)	N1—C8—H8B	109.5
S1—N1—S2	119.86 (8)	H8A—C8—H8B	109.5
O1—N2—O2	124.33 (14)	N1—C8—H8C	109.5
O1—N2—C5	117.86 (13)	H8A—C8—H8C	109.5
O2—N2—C5	117.75 (13)	H8B—C8—H8C	109.5
C2—C1—C6	120.88 (16)	S2—C9—H9A	109.5
C2—C1—H1A	119.6	S2—C9—H9B	109.5
C6—C1—H1A	119.6	H9A—C9—H9B	109.5
C3—C2—C1	119.99 (16)	S2—C9—H9C	109.5
C3—C2—H2A	120.0	H9A—C9—H9C	109.5
C1—C2—H2A	120.0	H9B—C9—H9C	109.5
O4—S1—N1—C8	8.06 (14)	O1—N2—C5—C4	34.0 (2)
O3—S1—N1—C8	−121.44 (13)	O2—N2—C5—C4	−143.33 (15)
C6—S1—N1—C8	124.08 (13)	O1—N2—C5—C6	−147.77 (15)
O4—S1—N1—S2	169.91 (9)	O2—N2—C5—C6	34.9 (2)
O3—S1—N1—S2	40.41 (11)	C2—C1—C6—C5	−0.4 (2)
C6—S1—N1—S2	−74.08 (10)	C2—C1—C6—S1	170.38 (13)
O5—S2—N1—C8	−163.93 (12)	C4—C5—C6—C1	1.6 (2)
O6—S2—N1—C8	−35.62 (14)	N2—C5—C6—C1	−176.56 (13)
C9—S2—N1—C8	79.67 (14)	C4—C5—C6—S1	−168.26 (12)
O5—S2—N1—S1	33.91 (11)	N2—C5—C6—S1	13.6 (2)
O6—S2—N1—S1	162.22 (8)	O4—S1—C6—C1	−139.46 (12)
C9—S2—N1—S1	−82.49 (11)	O3—S1—C6—C1	−10.20 (14)
C6—C1—C2—C3	−0.9 (3)	N1—S1—C6—C1	105.75 (13)
C1—C2—C3—C4	1.1 (3)	O4—S1—C6—C5	30.54 (15)
C2—C3—C4—C5	0.1 (3)	O3—S1—C6—C5	159.80 (13)
C3—C4—C5—C6	−1.4 (2)	N1—S1—C6—C5	−84.25 (14)
C3—C4—C5—N2	176.82 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1A···O3	0.95	2.40	2.807 (2)	106
C8—H8A···O4	0.98	2.30	2.811 (2)	112
C9—H9B···O3	0.98	2.60	3.243 (2)	124
C2—H2A···O2^{i}	0.95	2.58	3.435 (2)	149
C4—H4B···O6^{ii}	0.95	2.52	3.261 (2)	135
C8—H8A···O5^{iii}	0.98	2.50	3.194 (2)	128

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

Fig. 1

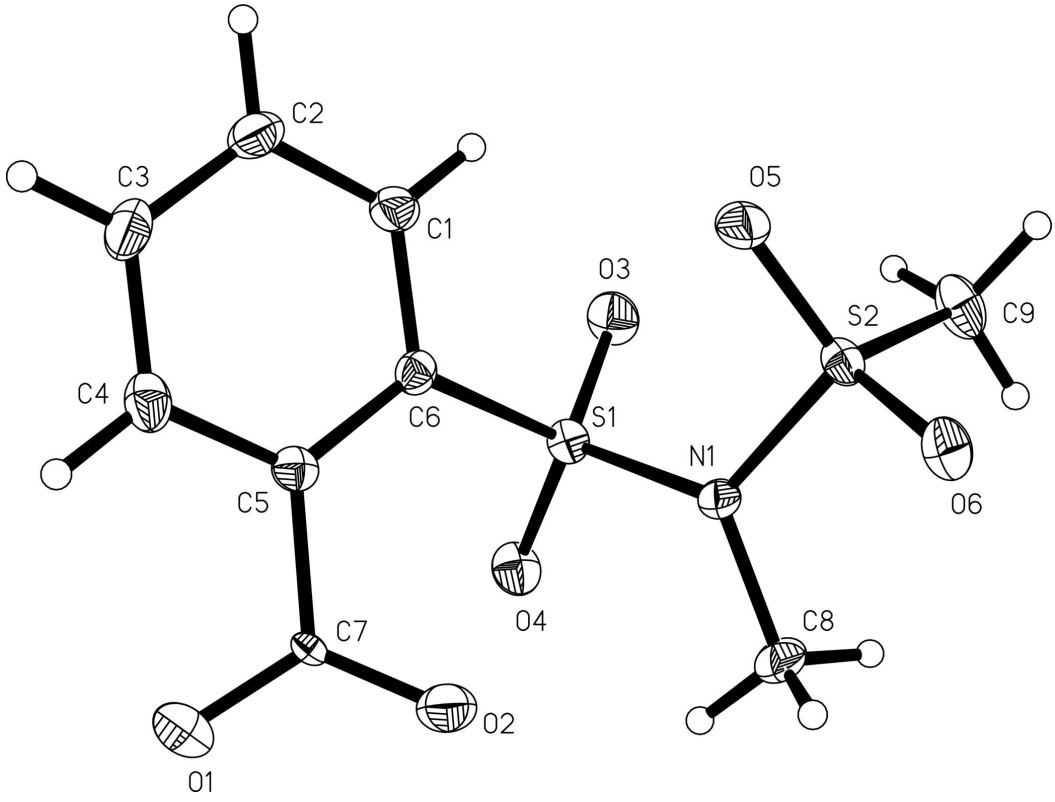

Fig. 2