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1-Methyl-4-methylsulfonyl-2-nitrobenzene

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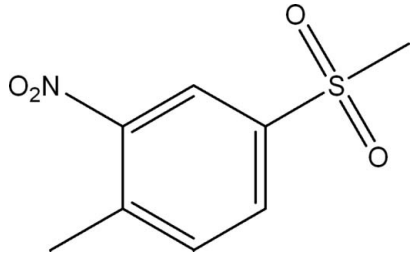
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.077; data-to-parameter ratio = 16.3.

The title compound, $C_8H_9NO_4S$, was prepared by the alkylation and nitration of 4-methylbenzenesulfonyl chloride. The aryl ring and its substituent atoms are essentially coplanar. The dihedral angle between this plane and the NO_2 planes is 42.4 (3)°

Related literature

For related literature, see: Brown (1991); Katz *et al.* (1953); Wichert *et al.* (2006).



Experimental

Crystal data

$C_8H_9NO_4S$
M_r = 215.22
Orthorhombic, $Pca2_1$
a = 14.957 (11) Å
b = 8.268 (5) Å
c = 7.795 (5) Å
V = 964.1 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 298 (1) K
0.45 × 0.40 × 0.35 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.856, T_{max} = 0.893
8883 measured reflections
2109 independent reflections
1430 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.077
S = 1.02
2109 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack (1983), 927 Friedel pairs
Flack parameter: 0.002 (8)

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); 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: SG2167).

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supplementary materials

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1-Methyl-4-methylsulfonyl-2-nitrobenzene

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Comment

1-Methyl-4-methylsulfonyl-2-nitrobenzene is an important intermediate for the synthesis of Mesotrione which is a useful corn herbicide for pre- and post-emergence control of grass and broadleaf weeds (Wichert *et al.*, 2006). It was obtained by the alkylation and nitration of 4-methylbenzenesulfonyl chloride.

The molecular structure of (1) is illustrated in Fig. 1. Atoms C1, C2, C3, C4, C5, C6, C7, N1 and S1 are coplanar, the largest deviation being 0.0415 (13) Å for C7.

Experimental

The title compound was prepared according to the procedure of Brown (1991) and Katz *et al.* (1953). A solution of the compound in ethanol was concentrated gradually at room temperature to afford colourless chunks (m.p. 391–392 K).

Refinement

H atoms were added at calculated positions and refined using a riding model. H atoms were given isotropic displacement parameters equal to 1.2(or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameters of their parent atoms and C—H distances were restrained to 0.93 Å for those bonded to phenyl ring, 0.96 Å for those bonded to methyl.

Figures

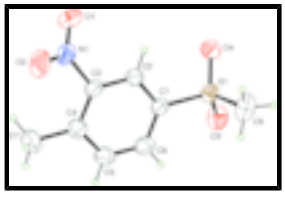

Fig. 1. The structure of (1), shown with 30% probability displacement ellipsoids.

1-Methyl-4-methylsulfonyl-2-nitrobenzene

Crystal data

$C_8H_9NO_4S$	F_{000} = 448.00
M_r = 215.22	D_x = 1.483 Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation λ = 0.71075 Å
Hall symbol: P 2c -2ac	Cell parameters from 7747 reflections
a = 14.957 (11) Å	θ = 3.6–27.5º
b = 8.268 (5) Å	μ = 0.32 mm^{-1}

c = 7.795 (5) Å	T = 298 (1) K
V = 964.1 (11) Å^3	Chunk, colorless
Z = 4	0.45 × 0.40 × 0.35 mm

Data collection

Rigaku R-AXIS RAPID diffractometer	1430 reflections with $F^2 > 2\sigma(F^2)$
Detector resolution: 10.00 pixels mm^{-1}	R_{int} = 0.039
ω scans	θ_{max} = 27.5º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$h = -19 \rightarrow 19$
T_{min} = 0.856, T_{max} = 0.893	$k = -10 \rightarrow 10$
8883 measured reflections	$l = -9 \rightarrow 10$
2109 independent reflections	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$wR(F^2)$ = 0.077	$\Delta\rho_{min}$ = −0.31 e Å^{-3}
S = 1.02	Extinction correction: Larson (1970), equation 22
2109 reflections	Extinction coefficient: 78 (10)
129 parameters	Absolute structure: Flack (1983), 927 Friedel pairs
H-atom parameters constrained	Flack parameter: 0.002 (8)
$w = 1/[0.0001F_o^2 + \sigma(F_o^2)]/(4F_o^2)$	

Special details

Refinement. Refinement using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (S) are based on F^2. *R*-factor (gt) are based on F. The threshold expression of F^2 > 2.0 sigma(F^2) is used only for calculating *R*-factor (gt).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.14758 (4)	0.09312 (6)	0.41547 (12)	0.04659 (16)
O1	0.48784 (12)	0.2707 (2)	0.4356 (3)	0.0711 (6)
O2	0.48852 (12)	0.4559 (2)	0.6321 (2)	0.0735 (7)
O3	0.07388 (12)	0.1408 (2)	0.3105 (2)	0.0659 (6)
O4	0.20666 (12)	−0.02992 (19)	0.3535 (2)	0.0663 (6)
N1	0.45069 (14)	0.3727 (2)	0.5241 (3)	0.0530 (7)
C1	0.21356 (14)	0.2679 (2)	0.4542 (3)	0.0403 (6)
C2	0.30390 (16)	0.2549 (2)	0.4770 (2)	0.0419 (7)
C3	0.35365 (14)	0.3938 (2)	0.5021 (2)	0.0424 (6)
C4	0.31618 (18)	0.5478 (2)	0.5019 (3)	0.0504 (8)
C5	0.2253 (2)	0.5549 (3)	0.4783 (4)	0.0609 (9)
C6	0.17398 (16)	0.4192 (3)	0.4536 (3)	0.0581 (9)

C7	0.3700 (2)	0.7023 (3)	0.5207 (4)	0.0733 (10)
C8	0.1062 (2)	0.0349 (3)	0.6141 (3)	0.0690 (10)
H2	0.3315	0.1541	0.4756	0.050*
H5	0.1974	0.6554	0.4792	0.073*
H6	0.1127	0.4290	0.4365	0.070*
H71	0.3851	0.7181	0.6392	0.088*
H72	0.4238	0.6941	0.4541	0.088*
H73	0.3352	0.7922	0.4808	0.088*
H81	0.0782	0.1258	0.6686	0.083*
H82	0.0631	−0.0500	0.5990	0.083*
H83	0.1545	−0.0034	0.6844	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0413 (3)	0.0524 (3)	0.0461 (3)	−0.0051 (2)	−0.0017 (3)	−0.0033 (4)
O1	0.0432 (10)	0.0778 (12)	0.0924 (17)	0.0151 (9)	0.0107 (13)	−0.0011 (14)
O2	0.0488 (12)	0.0821 (16)	0.0896 (17)	−0.0209 (11)	−0.0162 (12)	−0.0064 (13)
O3	0.0552 (12)	0.0772 (12)	0.0652 (13)	−0.0104 (10)	−0.0213 (10)	0.0067 (11)
O4	0.0604 (13)	0.0469 (9)	0.0916 (17)	−0.0024 (9)	0.0119 (10)	−0.0192 (10)
N1	0.0408 (12)	0.0589 (15)	0.0592 (15)	−0.0022 (11)	0.0029 (11)	0.0176 (12)
C1	0.0359 (12)	0.0442 (13)	0.0408 (16)	0.0015 (10)	0.0013 (11)	0.0024 (11)
C2	0.0424 (15)	0.0401 (13)	0.0431 (15)	0.0035 (10)	0.0045 (11)	0.0012 (11)
C3	0.0328 (12)	0.0478 (14)	0.0464 (13)	0.0007 (11)	0.0009 (11)	0.0023 (12)
C4	0.0518 (16)	0.0462 (15)	0.0532 (16)	−0.0002 (12)	−0.0001 (13)	0.0039 (12)
C5	0.0508 (16)	0.0472 (15)	0.085 (2)	0.0113 (13)	−0.0065 (14)	−0.0078 (14)
C6	0.0395 (14)	0.0574 (15)	0.077 (2)	0.0077 (12)	−0.0054 (13)	−0.0051 (15)
C7	0.070 (2)	0.0471 (16)	0.103 (2)	−0.0069 (15)	−0.0101 (19)	−0.0022 (17)
C8	0.0611 (19)	0.094 (2)	0.0516 (17)	−0.0259 (17)	0.0001 (15)	0.0019 (17)

Geometric parameters (Å, °)

S1—O3	1.428 (2)	C4—C7	1.517 (3)
S1—O4	1.4316 (18)	C5—C6	1.373 (3)
S1—C1	1.776 (2)	C2—H2	0.930
S1—C8	1.735 (2)	C5—H5	0.930
O1—N1	1.223 (3)	C6—H6	0.930
O2—N1	1.226 (3)	C7—H71	0.960
N1—C3	1.472 (3)	C7—H72	0.960
C1—C2	1.367 (3)	C7—H73	0.960
C1—C6	1.384 (3)	C8—H81	0.960
C2—C3	1.383 (3)	C8—H82	0.960
C3—C4	1.391 (3)	C8—H83	0.960
C4—C5	1.373 (4)		
O3—S1—O4	118.64 (13)	C1—C6—C5	119.9 (2)
O3—S1—C1	107.56 (11)	C1—C2—H2	120.5
O3—S1—C8	108.20 (13)	C3—C2—H2	120.5
O4—S1—C1	107.02 (10)	C4—C5—H5	118.8

O4—S1—C8	108.91 (13)	C6—C5—H5	118.8
C1—S1—C8	105.78 (13)	C1—C6—H6	120.0
O1—N1—O2	124.4 (2)	C5—C6—H6	120.0
O1—N1—C3	117.7 (2)	C4—C7—H71	109.5
O2—N1—C3	117.9 (2)	C4—C7—H72	109.5
S1—C1—C2	120.50 (17)	C4—C7—H73	109.5
S1—C1—C6	119.82 (17)	H71—C7—H72	109.5
C2—C1—C6	119.6 (2)	H71—C7—H73	109.5
C1—C2—C3	119.0 (2)	H72—C7—H73	109.5
N1—C3—C2	116.7 (2)	S1—C8—H81	109.5
N1—C3—C4	120.4 (2)	S1—C8—H82	109.5
C2—C3—C4	122.9 (2)	S1—C8—H83	109.5
C3—C4—C5	116.0 (2)	H81—C8—H82	109.5
C3—C4—C7	123.8 (2)	H81—C8—H83	109.5
C5—C4—C7	120.2 (2)	H82—C8—H83	109.5
C4—C5—C6	122.5 (2)		
O3—S1—C1—C2	−149.2 (2)	C2—C1—C6—C5	−1.0 (4)
O3—S1—C1—C6	27.8 (2)	C6—C1—C2—C3	1.2 (3)
O4—S1—C1—C2	−20.7 (2)	C1—C2—C3—N1	−179.4 (2)
O4—S1—C1—C6	156.3 (2)	C1—C2—C3—C4	−1.4 (3)
C8—S1—C1—C2	95.3 (2)	N1—C3—C4—C5	179.2 (2)
C8—S1—C1—C6	−87.7 (2)	N1—C3—C4—C7	1.0 (3)
O1—N1—C3—C2	38.9 (3)	C2—C3—C4—C5	1.3 (3)
O1—N1—C3—C4	−139.2 (2)	C2—C3—C4—C7	−177.0 (2)
O2—N1—C3—C2	−139.7 (2)	C3—C4—C5—C6	−1.0 (4)
O2—N1—C3—C4	42.2 (3)	C7—C4—C5—C6	177.3 (2)
S1—C1—C2—C3	178.19 (18)	C4—C5—C6—C1	0.9 (4)
S1—C1—C6—C5	−178.0 (2)		

Fig. 1