

4-Chloro-3-nitrophenyl methyl sulfone

Xin-Yan Zhang,^a Sheng-Rong Guo^{b*} and Yan-Qin Yuan^b^aLishui Vocational and Technical College, 323000 Lishui Zhejiang, People's Republic of China, and ^bDepartment of Chemistry, Lishui College, 323000 Lishui Zhejiang, People's Republic of China

Correspondence e-mail: guosr9608@163.com

Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.036

wR factor = 0.095

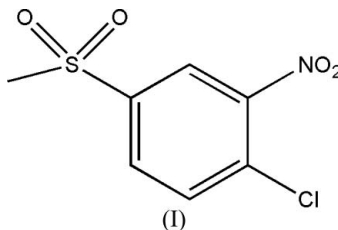
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_7\text{H}_6\text{ClNO}_4\text{S}$, the average S—C and S=O bond lengths are 1.762 (3) and 1.4352 (18) Å, respectively. In the crystal packing, the molecules are linked by intermolecular C—H···O hydrogen bonds to form layers parallel to the (101) plane.

Comment

In the course of our continuing studies aimed at the synthesis and characterization of new phenyl methyl sulfones, the title compound, (I), was obtained and its crystal structure is reported here.



The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles agree well with those observed in similar compounds (Podlaha *et al.*, 1986; Julia *et al.*, 1986; Hauback & Mo, 1990; Li *et al.*, 2003; Guo & Yuan, 2005). The S=O bond lengths within the SO_2 group are not significantly different, with an average value of 1.4352 (18) Å. The average bond length for the two C—S bonds is 1.762 (3) Å. The dihedral angle formed by the nitro group with the benzene ring is 27.90 (10)°. There is an intramolecular C—H···O hydrogen bond stabilizing the molecular structure (Table 2).

In the crystal packing, molecules related by a centre of symmetry are linked through intermolecular C—H···O hydrogen-bonding interactions (Table 2) to form two-dimensional sheets running parallel to the (101) plane (Fig. 2).

Experimental

A mixture of anhydrous sodium sulfite (0.25 mol), sodium bicarbonate (0.30 mol) and water (100 ml) was put in a beaker provided with a mechanical stirrer and thermometer, and heated on a hot plate at 343–353 K, while 3-nitro-4-chlorobenzenesulfonyl chloride (0.1 mol) was added dropwise with stirring. The solid sodium-substituted benzene sulfinate that separated was collected by filtration and mixed with sodium bicarbonate (0.1 mol) and dimethyl sulfate (0.08 mol) in a three-necked round-bottomed flask equipped with a mechanical stirrer, a reflux condenser and a separatory funnel. Water (10–20 ml) was added dropwise, and the solution was refluxed for 5 h. After cooling to room temperature, the mixture was extracted with benzene (30 ml \times 3) and dried with anhydrous calcium chloride. The drying

Received 30 October 2006

Accepted 16 November 2006

agent was removed by filtration and the remaining solid was washed with benzene. Benzene was then removed from the filtrate by distillation under reduced pressure, and the solid compound (I) that separated was dried further at about 10 mmHg. The product was finally purified by recrystallization from an ethanol-water mixture (1:1 v/v). Single crystals suitable for X-ray data collection were obtained by slow evaporation of an ethyl acetate–toluene (4:1 v/v) solution (m.p. 391–393 K).

Crystal data

$C_7H_6ClNO_4S$ $V = 455.43 (12) \text{ \AA}^3$
 $M_r = 235.65$ $Z = 2$
 Triclinic, $P\bar{1}$ $D_x = 1.718 \text{ Mg m}^{-3}$
 $a = 6.9754 (11) \text{ \AA}$ $Mo K\alpha$ radiation
 $b = 8.2311 (13) \text{ \AA}$ $\mu = 0.63 \text{ mm}^{-1}$
 $c = 8.7890 (14) \text{ \AA}$ $T = 298 (2) \text{ K}$
 $\alpha = 89.296 (2)^\circ$ Block, colourless
 $\beta = 79.266 (2)^\circ$ $0.34 \times 0.15 \times 0.14 \text{ mm}$
 $\gamma = 67.020 (2)^\circ$

Data collection

Bruker APEX area-detector 3381 measured reflections
 diffractometer 1651 independent reflections
 φ and ω scans 1566 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{int} = 0.012$
 (SADABS; Bruker, 2002) $\theta_{max} = 25.3^\circ$
 $T_{min} = 0.833, T_{max} = 0.916$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.2421P]$
 $R[F^2 > 2\sigma(F^2)] = 0.036$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.095$ $(\Delta/\sigma)_{max} < 0.001$
 $S = 1.09$ $\Delta\rho_{max} = 0.44 \text{ e \AA}^{-3}$
 1651 reflections $\Delta\rho_{min} = -0.27 \text{ e \AA}^{-3}$
 128 parameters
 H-atom parameters constrained

Table 1 Selected geometric parameters (\AA , $^\circ$).

C11–C4	1.719 (2)	S1–C1	1.774 (2)
S1–O1	1.4347 (18)	O3–N1	1.221 (3)
S1–O2	1.4358 (17)	O4–N1	1.211 (3)
S1–C7	1.751 (3)		
O1–S1–O2	118.60 (11)	O1–S1–C1	107.51 (10)
O1–S1–C7	108.83 (13)	O2–S1–C1	107.25 (10)
O2–S1–C7	108.71 (13)	C7–S1–C1	105.12 (11)

Table 2 Hydrogen-bond geometry (\AA , $^\circ$).

D–H...A	D–H	H...A	D...A	D–H...A
C6–H6...O2	0.93	2.55	2.909 (3)	103
C2–H2...O1 ⁱ	0.93	2.43	3.309 (2)	159
C6–H6...O4 ⁱⁱ	0.93	2.59	3.442 (3)	153
C7–H7C...O2 ⁱⁱⁱ	0.96	2.46	3.316 (4)	149

Symmetry codes: (i) $-x + 1, -y + 2, -z + 2$; (ii) $x, y - 1, z$; (iii) $-x + 1, -y + 1, -z + 2$.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C–H distances in the range 0.93–0.96 \AA and $U_{iso}(H) = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for methyl H atoms.

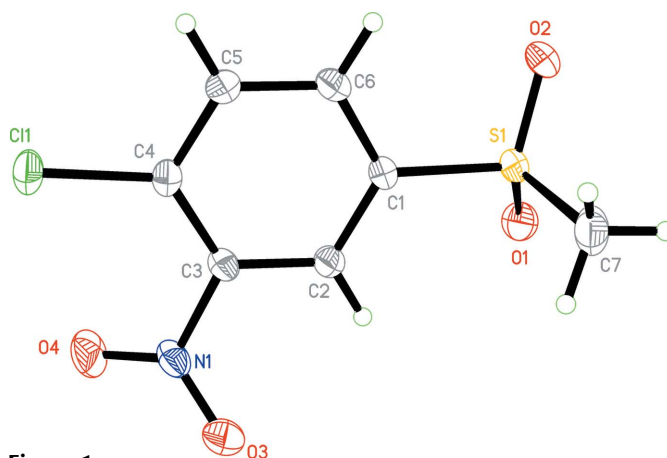


Figure 1 The molecular structure of (I) showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

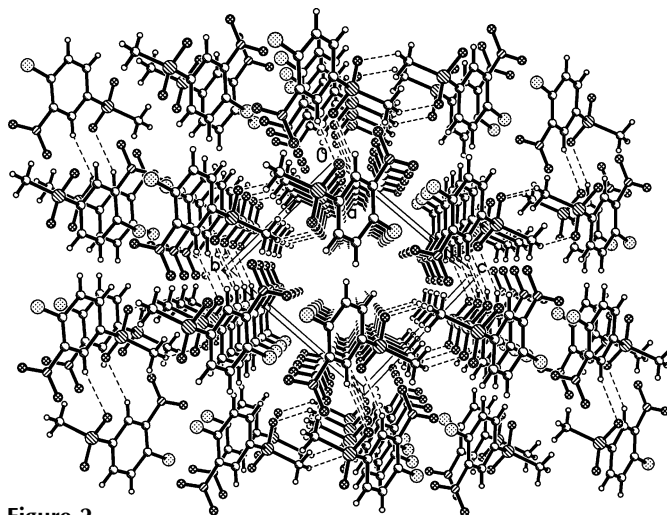


Figure 2 Packing diagram viewed down the a axis. Hydrogen bonds are shown as dashed lines.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP (Bruker, 2002); software used to prepare material for publication: SHELXL97.

This work was supported by the Youth Foundation of Lishui Vocational and Technical College, Lishui Zhejiang, China.

References

Bruker (2002). SADABS (Version 2.03), SAINT (Version 6.02), SMART (Version 5.62) and XP. Bruker AXS Inc., Madison, Wisconsin, USA.
 Guo, S.-R. & Yuan, Y.-Q. (2005). *Acta Cryst.* **E61**, o4032–o4033.
 Hauback, B. C. & Mo, F. (1990). *Z. Kristallogr.* **191**, 195–207.
 Julia, M., Lauron, H., Stacino, J.-P., Verpeaux, J.-N., Jeannin, Y. & Dromzee, Y. (1986). *Tetrahedron*, **42**, 2475–2484.
 Li, J.-R., Zheng, Y., Xie, Y.-B., Zou, R.-Q., Zhang, R.-H. & Bu, X.-H. (2003). *Acta Cryst.* **E59**, o70–o71.
 Podlaha, J., Podlahová, J., Kratochvíl, B., Tulsani, N. B., Khawaja, A. M. & Malý, K. (1986). *Acta Cryst.* **C42**, 491–493.
 Sheldrick, G. M. (1997). SHELXL97 and SHELXS97. University of Göttingen, Germany.