

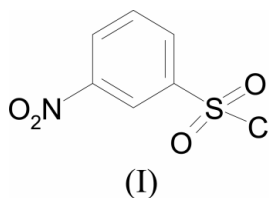
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Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.039
 wR factor = 0.106
Data-to-parameter ratio = 14.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.3-Nitrobenzenesulfonyl chloride:
molecular aggregation through
C—H···O interactionsIn the title compound, $\text{C}_6\text{H}_4\text{ClNO}_2\text{S}$, the molecular structure and molecular packing in the crystal are stabilized by C—H···O interactions. The C—H···O interactions form $S(5)$ and R_2^1 ring motifs.

Comment

Structural studies on aryl sulfonyl chlorides are essential for understanding the mechanisms of nucleophilic substitution reactions at the sulfonyl reaction centre (Kislov & Ivanov, 2000). Substitution reactions of sulfonyl chlorides bridge inorganic and organic chemistry (Koo *et al.*, 2000). A protein complex of 4-toluenesulfonyl chloride had been reported as a ruminant feed material (Miller, US patent No. 3711289). Sulfonyl halides are used as initiators in the metal-catalysed living radical polymerization of acrylonitrile (Barboiu & Percec, 2002). Arene sulfonyl halides were reported to be a universal class of functional initiators for metal-catalysed living radical polymerization of styrenes, methacrylates and acrylates (Percec *et al.*, 1998). The present crystal structure determination of the title compound, (I), was undertaken owing to the biological activity and synthetic utility of its analogues.A search of the July 2002 release of the Cambridge Structural Database (Allen, 2002) revealed 11 structures (with the following refcodes: COKCOU, FAQJOW, FOHYAC, KADZOE, NAQLUM, NAXFEX, NAXFIB, NBZSOC, QQHJA, QQHJA01 and SUTYAH) which are closely related to the title compound. The S=O, S—C and S—Cl bond lengths (Table 1) are comparable to those found in these structures. The crystal structure of (I) is stabilized by weak C—H···O interactions. The range of H···O distances (Table 2) found in (I) agrees with those found for weak C—H···O bonds (Desiraju & Steiner, 1999). The C6—H6···O4 and C6—H6···O2 (Fig. 1) interactions constitute a pair of bifurcated donor bonds, generating two rings fused to each other, each of graph-set motif $S(5)$ (Etter, 1990; Bernstein *et al.*, 1995). Each of the C4—H4···O3 and C2—H2···O1 (Fig. 2) interactions generates an $S(5)$ motif. The C3—H3···O2ⁱⁱⁱ and C2—H2···O2ⁱⁱⁱ (Fig. 2 and Table 2 for symmetry codes) interac-Received 2 June 2003
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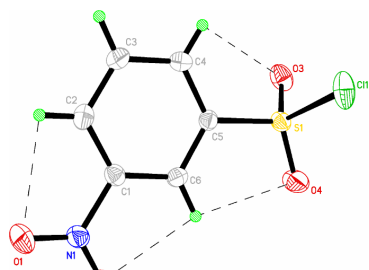


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and intramolecular C—H...O interactions.

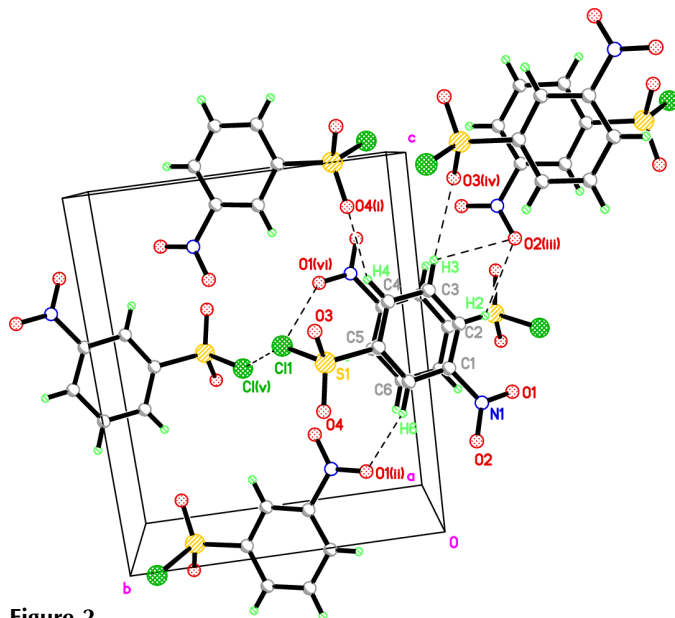


Figure 2
Part of the molecular network, showing intermolecular C—H...O interactions and also Cl...Cl and Cl...O contacts. [Symmetry codes: (v) $1-x, 1-y, 1-z$; (vi) $1-x, -y, 1-z$; other codes are given in Table 2.]

tions constitute a pair of bifurcated acceptor bonds, generating a ring of graph set $R_2^2(5)$. The molecular aggregation is completed by several other weak C—H...O interactions (Table 2 and Fig. 2). Other short intermolecular contacts are Cl1...O1($1-x, -y, 1-z$) of 3.231 (2) Å and Cl1...Cl1($1-x, 1-y, 1-z$) of 3.437 (1) Å.

Experimental

The title compound was purchased from National Chemicals, Vadodara, Gujarat, India. Diffraction quality crystals were obtained by recrystallizing the sample from a 1:1 mixture of petroleum ether and acetone.

Crystal data

$C_6H_4ClNO_4S$
 $M_r = 221.61$
Monoclinic, $P2_1/n$
 $a = 7.8868$ (4) Å
 $b = 9.4298$ (4) Å
 $c = 11.8789$ (5) Å
 $\beta = 107.318$ (2)°
 $V = 843.40$ (7) Å³
 $Z = 4$

$D_x = 1.745$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 823 reflections
 $\theta = 2.8$ – 27.5 °
 $\mu = 0.68$ mm⁻¹
 $T = 120$ (2) K
Block, colourless
 $0.39 \times 0.24 \times 0.13$ mm

Data collection

Bruker SMART 6K CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1998)
 $T_{\min} = 0.822, T_{\max} = 0.916$
9420 measured reflections

1928 independent reflections
1667 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$
 $\theta_{\text{max}} = 27.5$ °
 $h = -10 \rightarrow 10$
 $k = -12 \rightarrow 12$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.106$
 $S = 1.08$
1928 reflections
134 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.872P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 1.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.69$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

S1—O3	1.4182 (17)	C5—C6	1.387 (3)
S1—O4	1.4193 (18)	C5—C4	1.392 (3)
S1—C5	1.761 (2)	C1—C6	1.384 (3)
S1—Cl1	2.0198 (8)	C1—C2	1.385 (3)
O1—N1	1.222 (3)	C3—C4	1.386 (3)
N1—O2	1.224 (3)	C3—C2	1.391 (3)
N1—C1	1.472 (3)		
O3—S1—O4	120.87 (12)	C5—S1—Cl1	102.24 (7)
O3—S1—C5	109.41 (10)	O1—N1—O2	124.23 (19)
O4—S1—C5	109.45 (10)	O1—N1—C1	117.94 (18)
O3—S1—Cl1	105.20 (8)	O2—N1—C1	117.83 (18)
O4—S1—Cl1	107.98 (10)		

Table 2

Geometry of C—H...O interactions (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6—H6...O4	0.95 (3)	2.55 (3)	2.897 (3)	101.9 (18)
C6—H6...O2	0.95 (3)	2.42 (3)	2.712 (3)	97.1 (18)
C4—H4...O3	0.91 (3)	2.79 (3)	3.051 (3)	97.8 (18)
C2—H2...O1	0.90 (3)	2.48 (3)	2.725 (3)	96.0 (19)
C4—H4...O4 ⁱ	0.91 (3)	2.38 (3)	3.116 (3)	138 (2)
C6—H6...O1 ⁱⁱ	0.95 (3)	2.52 (3)	3.251 (3)	134 (2)
C3—H3...O2 ⁱⁱⁱ	0.93 (3)	2.97 (3)	3.504 (3)	118 (2)
C2—H2...O2 ⁱⁱⁱ	0.90 (3)	2.71 (3)	3.387 (3)	133 (2)
C3—H3...O3 ^{iv}	0.93 (3)	2.54 (3)	3.412 (3)	156 (3)

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

H atoms were located from a difference Fourier map and both positional and isotropic displacement parameters were refined. The C—H bond lengths are in the range 0.90 (3)–0.95 (3) Å and the C—C—H angles for the aromatic ring are in the range 118 (2)–123 (2)°. The highest peak in the difference Fourier map lies close to the Cl atom (0.84 Å).

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998); program(s) used to solve structure: SHELXTL (Bruker, 1998); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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