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4-Methylphenyl 3-nitrobenzenesulfonate

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In $C_{13}H_{11}NO_5S$, there are weak C—H$\cdots$O interactions which generate rings of motifs $S(5)$, $S(6)$, $R_2^2(8)$, $R_2^2(11)$ and $R_2^2(14)$.

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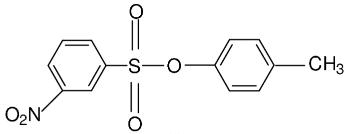
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.091
Data-to-parameter ratio = 12.7

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

Comment

Aromatic sulfonates are used in detecting specific organic anion binding protein(s) in the liver plasma membrane (Yachi *et al.*, 1989) and in many other fields (Spungin *et al.*, 1992; Tharakan *et al.*, 1992; Alford *et al.*, 1991; Jiang *et al.*, 1990; Narayanan & Krakow, 1983). The molecular and crystal structure of 3-nitrobenzenesulfonyl chloride has been reported recently (Vembu, Nallu, Spencer & Howard, 2003*c*). In view of the biological importance of its analogues and also to allow for comparison of its structural parameters with those of its precursor, 3-nitrobenzenesulfonyl chloride, an X-ray study of the title compound, (I), was undertaken.



(I)

The molecular structure of (I) is shown in Fig. 1, and selected geometric parameters are given in Table 1. Atoms N, O1 and O2 deviate from the mean plane formed by atoms C1–C6 by 0.015 (2), 0.008 (2) and 0.019 (2) Å, respectively. The dihedral angle between the above plane and that of the 4-methylphenyl ring is 53.44 (4)°. This shows their non-coplanar orientation is similar to that reported for other aromatic sulfonates (Vembu, Nallu, Garrison & Youngs, 2003*b*,*c*,*d*,*e*; Vembu, Nallu, Spencer & Howard, 2003*a*,*b*,*c*,*d*,*e*,*f*,*g*) and in contrast to the near-coplanar orientation found in 2,4-dinitrophenyl 4-toluenesulfonate (Vembu, Nallu, Garrison & Youngs, 2003*a*) and 4-methoxyphenyl 4-toluenesulfonate (Vembu, Nallu, Garrison, Hindi & Youngs, 2003).

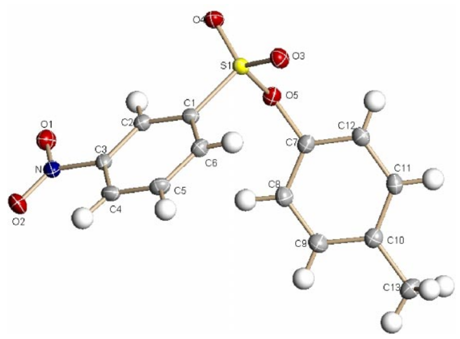


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

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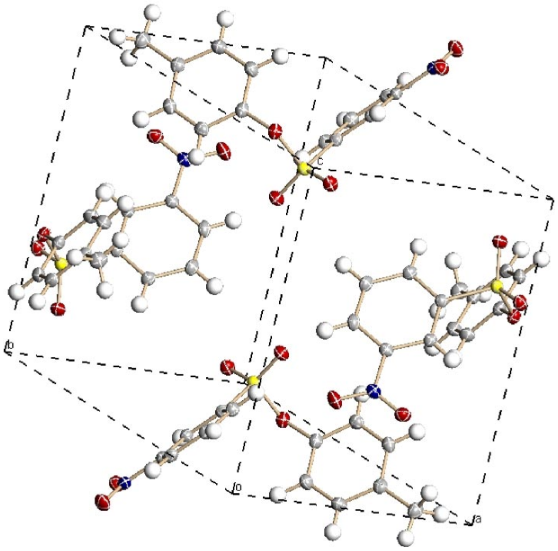

Figure 2
View of the unit cell along the *b* axis.

The crystal structure of (I) is stabilized by weak C—H···O interactions (Table 2). The H···O distances found in (I) agree with those found for weak C—H···O bonds (Desiraju & Steiner, 1999). In (I), each of the C4—H4···O2, C2—H2···O1, C2—H2···O4 and C6—H6···O3 interactions generates an *S*(5) graph-set motif (Bernstein *et al.*, 1995; Etter, 1990). The *S*(5) rings generated by C4—H4···O2 and C2—H2···O1 are planar. The *S*(5) rings generated by C2—H2···O4 and C6—H6···O3 are non-planar, with atoms O4 and O3 deviating by 0.732 (13) and 0.301 (15) Å, respectively, from the corresponding mean planes formed by the other four atoms. The C2—H2···O1 and C2—H2···O4 interactions constitute a pair of bifurcated donor bonds. The C12—H12···O3 interaction generates an *S*(6) motif. The C6—H6···O3 and C12—H12···O3 interactions form a pair of bifurcated acceptor bonds. The C2—H2···O5$^{\rm ii}$ and C8—H8···O4$^{\rm ii}$ interactions together generate an $R_2^2(11)$ motif (symmetry codes as in Table 2). The C11—H11···O4$^{\rm iii}$ and C13—H13*C*···O3$^{\rm iii}$ interactions together generate a sulfonyl fork motif of graph set $R_2^2(8)$. The C13—H13*B*···O2$^{\rm iv}$ and C9—H9···O1$^{\rm iv}$ interactions form a nitro-fork motif of graph set $R_2^2(8)$. The C6—H6···O2$^{\rm v}$ and C13—H13*A*···O1$^{\rm v}$ interactions together generate an $R_2^2(14)$ motif. There are a few more C—H···O interactions which contribute to the supramolecular aggregation of the title compound. Other short intermolecular contacts are N···C2$(1 - x, 2 - y, 1 - z)$ of 3.236 (2) Å, N···C11$(x + \frac{1}{2}, \frac{3}{2} - y, z + \frac{1}{2})$ of 3.190 (2) Å and C4···O3$(x + \frac{1}{2}, \frac{3}{2} - y, z + \frac{1}{2})$ of 3.008 (2) Å. The packing of molecules is shown in Fig. 2.

Experimental

3-Nitrobenzenesulfonyl chloride (5 mmol) dissolved in acetone (5 ml) was added to *p*-cresol (4.6 mmol) dissolved in aqueous NaOH solution (4 ml, 5%) and shaken well. The precipitated solid product (3 mmol; yield: 65%) was recrystallized from ethanol.

Crystal data

$C_{13}H_{11}NO_5S$
$M_r = 293.29$
Monoclinic, $P2_1/n$
$a = 9.2127$ (13) Å
$b = 12.9944$ (19) Å
$c = 10.5892$ (15) Å
$\beta = 93.267$ (2)°
$V = 1265.6$ (3) Å^3
$Z = 4$
$D_x = 1.539$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6112 reflections
$\theta = 2.5$–$28.3°$
$\mu = 0.28$ mm^{-1}
$T = 100$ (2) K
Block, colorless
0.41 × 0.27 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min} = 0.896$, $T_{\rm max} = 0.937$
7309 measured reflections
2864 independent reflections
2611 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\rm max} = 28.4°$
$h = -12 \rightarrow 12$
$k = -16 \rightarrow 14$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.091$
$S = 1.05$
2864 reflections
225 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 0.5196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.37$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O3	1.4243 (10)	O1—N	1.2249 (16)
S1—O4	1.4227 (11)	O2—N	1.2239 (16)
S1—O5	1.5921 (10)	O5—C7	1.4218 (17)
S1—C1	1.7575 (14)	N—C3	1.4744 (17)
O4—S1—O3	120.52 (6)	O5—S1—C1	103.35 (6)
O4—S1—O5	103.23 (6)	C7—O5—S1	119.37 (8)
O3—S1—O5	109.60 (6)	O2—N—O1	124.10 (12)
O4—S1—C1	110.29 (6)	O2—N—C3	117.95 (12)
O3—S1—C1	108.45 (6)	O1—N—C3	117.96 (11)
C1—S1—O5—C7	−68.40 (11)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2	0.93 (2)	2.400 (18)	2.7244 (19)	100.4 (13)
C2—H2···O1	0.950 (17)	2.407 (17)	2.6981 (17)	97.3 (12)
C2—H2···O4	0.950 (17)	2.727 (17)	3.0085 (17)	97.9 (11)
C12—H12···O3	0.928 (18)	2.872 (17)	3.1433 (17)	98.3 (12)
C6—H6···O3	0.920 (18)	2.526 (17)	2.9107 (18)	105.5 (12)
C4—H4···O3$^{\rm i}$	0.93 (2)	2.438 (18)	3.0081 (17)	119.7 (14)
C2—H2···O5$^{\rm ii}$	0.950 (17)	2.356 (17)	3.2469 (16)	156.0 (14)
C8—H8···O4$^{\rm ii}$	0.975 (18)	2.714 (17)	3.3047 (18)	119.4 (13)
C11—H11···O4$^{\rm iii}$	0.962 (19)	2.739 (19)	3.6521 (18)	158.7 (14)
C13—H13*C*···O3$^{\rm iii}$	0.94 (2)	2.83 (2)	3.482 (2)	127.5 (17)
C13—H13*B*···O2$^{\rm iv}$	0.95 (3)	2.79 (3)	3.730 (2)	174.4 (19)
C9—H9···O1$^{\rm iv}$	0.936 (19)	2.695 (19)	3.6154 (18)	167.9 (15)
C6—H6···O2$^{\rm v}$	0.920 (18)	2.583 (18)	3.3515 (18)	141.4 (14)
C13—H13*A*···O1$^{\rm v}$	0.96 (2)	2.79 (2)	3.4887 (19)	130.5 (17)
C5—H5···O4$^{\rm vi}$	0.962 (19)	2.727 (19)	3.5354 (18)	142.0 (14)

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

All the H atoms were located in a difference Fourier map and their positional coordinates and isotropic displacement parameters were refined. The C—H bond lengths are in the range 0.92 (2)–0.97 (2) Å.

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

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