Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Phenyl 3-nitrobenzenesulfonate

Nagarajan Vembu,[a] Maruthai Nallu,[a]* Elinor C. Spencer[b] and Judith A. K. Howard[b]

[a]Department of Chemistry, Bharathidasan University, Tiruchirappalli 620 024, India, and [b]Department of Chemistry, Durham University, Durham DH1 3LE, England

Correspondence e-mail:
mnalv2003@yahoo.com

Received 14 July 2003
Accepted 21 July 2003
Online 31 July 2003

Key indicators

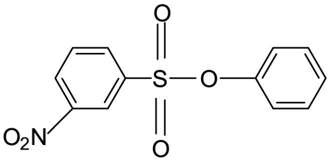
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.060
Data-to-parameter ratio = 12.0

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

In the title molecule, $C_{12}H_9NO_5S$, there are weak C—H$\cdots$O interactions which generate rings of motifs $S(5)$, $S(6)$, $R_1^2(4)$, $R_2^1(5)$, $R_2^2(7)$ and $R_2^2(13)$. The supramolecular aggregation is completed by the presence of C—H$\cdots\pi$ interactions.

Comment

Aromatic sulfonates are used in monitoring the merging of lipids (Yachi *et al.*, 1989) and in many other fields. An X-ray study of the title compound, (I), was undertaken in view of the biological importance of its analogues and also to compare its structural parameters with those of its precursor, 3-nitrobenzenesulfonyl chloride (Vembu, Nallu, Spencer & Howard, 2003*c*).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters in Table 1. The dihedral angle between the mean planes of the 3-nitrobenzene and phenyl rings is 38.76 (8)°. This non-coplanar orientation is similar to that found in some other aromatic sulfonates (Vembu, Nallu, Garrison & Youngs, 2003*b*,*c*,*d*,*e*; Vembu, Nallu, Spencer & Howard, 2003*a*,*b*), and is in contrast to the near coplanar orientation found in the 2,4-dinitrophenyl (Vembu, Nallu, Garrison & Youngs, 2003*a*) and 4-methoxyphenyl (Vembu, Nallu, Garrison, Hindi & Youngs, 2003) derivatives.

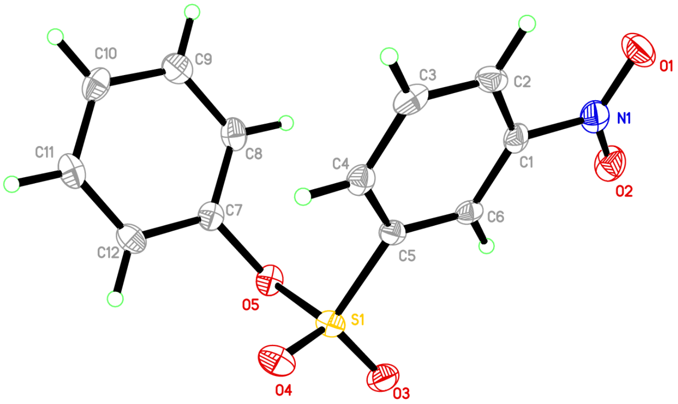


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

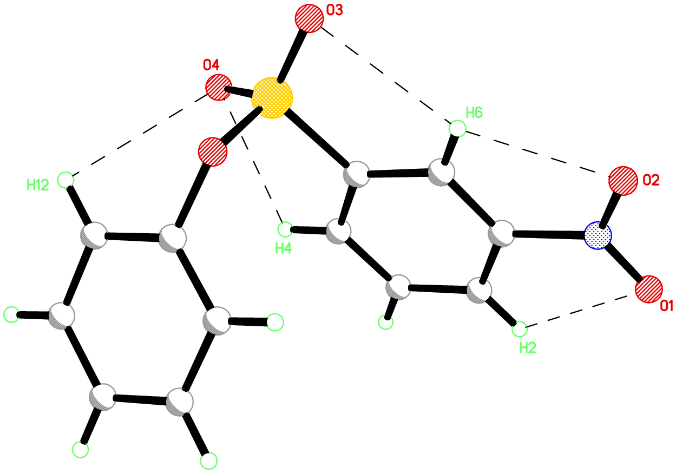


Figure 2
Diagram showing hydrogen bonds 1–5 (the numbers relate to the sequence of entries in Table 2).

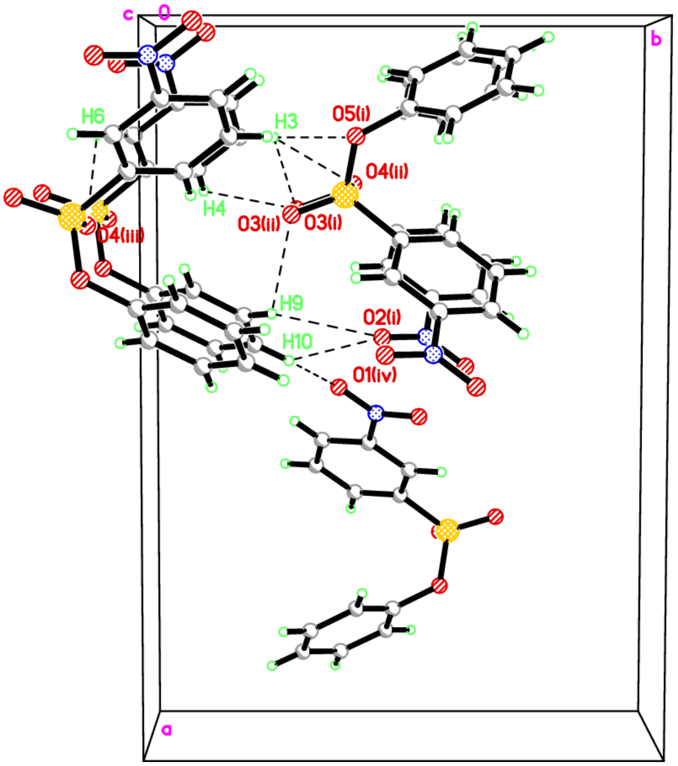


Figure 3
Diagram showing hydrogen bonds 6–14 (the numbers relate to the sequence of entries in Table 2). Symmetry codes are as in Table 2.

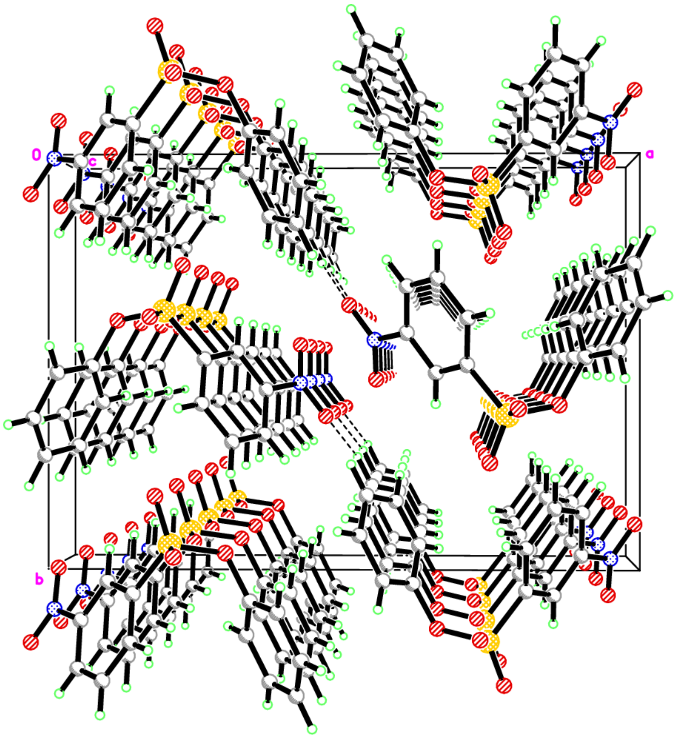


Figure 4
The packing of molecules, viewed along the *c* axis, showing the C—H$\cdots\pi$ interactions.

The crystal structure of (I) is stabilized by weak C—H···O interactions (Table 2). The range of H···O distances found in (I) agrees with that found for weak C—H···O bonds (Desiraju & Steiner, 1999). As shown in Fig. 2, each of the C2—H2···O1, C4—H4···O4, C6—H6···O2 and C6—H6···O3 interactions generates rings of graph-set motif *S*(5) (Etter, 1990; Bernstein *et al.*, 1995). The C6—H6···O2 and C6—H6···O3 interactions together constitute a pair of bifurcated donor bonds. The C12—H12···O4 interaction generates an *S*(6) motif. The C12—H12···O4 and C4—H4···O4 interactions together constitute a pair of bifurcated acceptor bonds. The C3—H3···O3$^{\rm i}$ and C3—H3···O5$^{\rm i}$ interactions constitute a pair of bifurcated donor bonds, generating a symmetrical three-centre hydrogen-bonded chelate motif (Fig. 3) of graph-set $R_1^2(4)$ (symmetry codes are as in Table 2). The C4—H4···O3$^{\rm i}$ and C3—H3···O3$^{\rm i}$ interactions constitute a pair of bifurcated acceptor bonds, generating a ring of graph-set $R_2^1(5)$. The C3—H3···O5$^{\rm i}$ and C4—H4···O3$^{\rm i}$ interactions together generate an $R_2^2(7)$ motif, which consists of $R_1^2(4)$ and $R_2^1(5)$ motifs. The C9—H9···O3$^{\rm ii}$ and C3—H3···O4$^{\rm ii}$ interactions together form a sulfonyl bifurcated motif of graph-set $R_2^2(13)$. There are several other C—H···O interactions which contribute to the supramolecular aggregation of this structure. The supramolecular aggregation is completed by the presence of two C—H$\cdots\pi$ interactions (Fig. 4 and Table 2; Spek, 1998).

Experimental

3–Nitrobenzenesulfonyl chloride (5 mmol) dissolved in acetone (4 ml) was added to phenol (5 mmol) in NaOH solution (2.5 ml, 8%) with constant shaking. The precipitated title compound, (I) (3.9 mmol, yield 78%), was filtered off and recrystallized from ethanol.

Crystal data

$C_{12}H_9NO_5S$
M_r = 279.26
Orthorhombic, $Pna2_1$
a = 17.458 (4) Å
b = 12.287 (3) Å
c = 5.4891 (14) Å
V = 1177.4 (5) Å^3
Z = 4
D_x = 1.575 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 713 reflections
θ = 2.9–25.7°
μ = 0.29 mm^{-1}
T = 120 (2) K
Block, colourless
0.16 × 0.14 × 0.09 mm

o1214 Nagarajan Vembu *et al.* • $C_{12}H_9NO_5S$ *Acta Cryst.* (2003). E**59**, o1213–o1215

Data collection

Bruker Proteum *M* diffractometer
ω scans
Absorption correction: none
7868 measured reflections
2392 independent reflections
1838 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.059$
$\theta_{\rm max} = 27.5°$
$h = -22 \rightarrow 22$
$k = -15 \rightarrow 12$
$l = -7 \rightarrow 5$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.060$
$S = 0.91$
2392 reflections
199 parameters
Only coordinates of H atoms refined

$w = 1/[\sigma^2(F_o^2) + (0.0153P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.44$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.47$ e Å^{-3}
Absolute structure: (Flack, 1983), 897 Friedel pairs
Flack parameter = −0.01 (8)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.480 (4)	N1—O1	1.230 (3)
C5—S1	1.763 (3)	O3—S1	1.4175 (18)
C7—O5	1.427 (3)	O4—S1	1.418 (2)
N1—O2	1.226 (3)	O5—S1	1.600 (2)
O2—N1—O1	124.3 (3)	O3—S1—O5	103.67 (10)
O2—N1—C1	118.1 (2)	O4—S1—O5	108.94 (11)
O1—N1—C1	117.6 (2)	O3—S1—C5	110.74 (13)
C7—O5—S1	117.36 (16)	O4—S1—C5	107.98 (13)
O3—S1—O4	120.78 (11)	O5—S1—C5	103.28 (11)
C7—O5—S1—C5	−60.2 (2)		

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

*Cg*2 is the centroid of the C7–C12 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1	0.89 (2)	2.42 (3)	2.707 (4)	98.8 (18)
C4—H4···O4	0.90 (2)	2.57 (2)	2.932 (3)	104.5 (17)
C6—H6···O2	0.94 (2)	2.39 (2)	2.715 (4)	100.2 (15)
C6—H6···O3	0.94 (2)	2.69 (2)	2.968 (3)	97.7 (16)
C12—H12···O4	0.90 (3)	2.80 (2)	3.095 (3)	100.4 (18)
C3—H3···O3$^{\rm i}$	1.00 (2)	2.80 (3)	3.426 (4)	121.2 (17)
C3—H3···O5$^{\rm i}$	1.00 (2)	2.67 (3)	3.647 (3)	165 (2)
C9—H9···O2$^{\rm i}$	0.93 (3)	2.87 (2)	3.399 (4)	117 (2)
C10—H10···O2$^{\rm i}$	0.94 (2)	2.58 (2)	3.260 (3)	129.6 (19)
C4—H4···O3$^{\rm i}$	0.90 (2)	2.75 (2)	3.364 (4)	127.0 (18)
C9—H9···O3$^{\rm ii}$	0.93 (3)	2.78 (3)	3.366 (4)	121.7 (19)
C3—H3···O4$^{\rm ii}$	1.00 (2)	2.65 (2)	3.170 (3)	112.2 (19)
C6—H6···O4$^{\rm iii}$	0.94 (2)	2.84 (2)	3.317 (3)	112.9 (15)
C10—H10···O1$^{\rm iv}$	0.94 (2)	2.48 (3)	3.219 (4)	135 (2)
C4—H4···*Cg*2	0.90 (2)	3.38	3.72	105
C12—H12···*Cg*2$^{\rm v}$	0.90 (3)	3.13	3.71	124

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

All the H atoms were located from difference Fourier maps and their positional parameters were refined with $U_{\rm iso} = 1.2U_{\rm eq}$(parent atom). The C—H bond lengths are in the range 0.89 (3)–1.00 (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*.

NV thanks the University Grants Commission–SERO, Government of India, for the award of Faculty Improvement Programme Grant [TFTNBD097 dt., 07.07.99]. JAKH thanks the EPRSC for a Senior Research Fellowship. ECS thanks the EPRSC for support.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (1998). *SMART-NT* and *SAINT-NT*. Versions 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.

Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*. New York: Oxford University Press.

Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Sheldrick, G. M. (1998). *SHELXTL*. University of Göttingen, Germany.

Spek, A. L. (1998). *PLATON*. Utrecht University, The Netherlands.

Vembu, N., Nallu, M., Garrison, J., Hindi, K. & Youngs, W. J. (2003). *Acta Cryst.* E**59**, o830–o832.

Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003*a*). *Acta Cryst.* E**59**, o378–o380.

Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003*b*). *Acta Cryst.* E**59**, o503–o505.

Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003*c*). *Acta Cryst.* E**59**, o776–o779.

Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003*d*). *Acta Cryst.* E**59**, o936–o938.

Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003*e*). *Acta Cryst.* E**59**, o1019–o1021.

Vembu, N., Nallu, M., Spencer, E. C. & Howard, J. A. K. (2003*a*). *Acta Cryst.* E**59**, o1009–o1011.

Vembu, N., Nallu, M., Spencer, E. C. & Howard, J. A. K. (2003*b*). *Acta Cryst.* E**59**, o1033–o1035.

Vembu, N., Nallu, M., Spencer, E. C. & Howard, J. A. K. (2003*c*). *Acta Cryst.* E**59**, o1036–o1038.

Yachi, K., Sugiyama, Y., Sawada, Y., Iga, T., Ikeda, Y., Toda, G. & Hanano, M. (1989). *Biochim. Biophys. Acta*, **978**, 1–7.

Acta Cryst. (2003). E**59**, o1213–o1215 Nagarajan Vembu *et al.* • $C_{12}H_9NO_5S$ **o1215**