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2-Nitrophenyl 3-nitrobenzenesulfonate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.070
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.

In the title molecule, $C_{12}H_8N_2O_7S$, there are weak C—H$\cdots$O interactions which generate rings of motifs $S(5)$, $R_2^1(5)$, $R_1^2(4)$ and $R_1^2(8)$.

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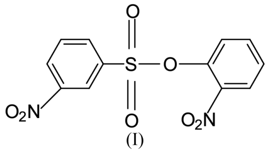
Comment

Aromatic sulfonates are used in monitoring the merging of lipids (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 recently reported (Vembu, Nallu, Spencer & Howard, 2003*c*). In view of the biological importance of its analogues and also to compare 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 N1, O1 and O2 deviate from the mean plane formed by atoms C1–C6 by 0.031 (3), 0.015 (4) and 0.085 (4) Å, respectively, while atoms N2, O6, O7 deviate from the mean plane formed by atoms C7–C12 by 0.146 (3), 0.874 (4) and −0.456 (4) Å, respectively. The dihedral angle between the C1–C6 and C7–C12 planes is 39.68 (8)°. This shows their non-coplanar orientation, similar to that reported for other aromatic sulfonates (Vembu, Nallu, Garrison & Youngs, 2003*b*,*c*,*d*,*e*; Vembu, Nallu, Spencer & Howard, 2003*a*,*b*,*d*,*e*), but in contrast to the near coplanar orientation found in 2,4-dinitrophenyl 4-toluenesulfonate (Vembu, Nallu, Garrison &

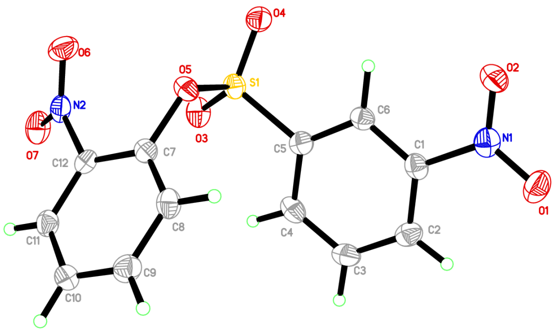


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

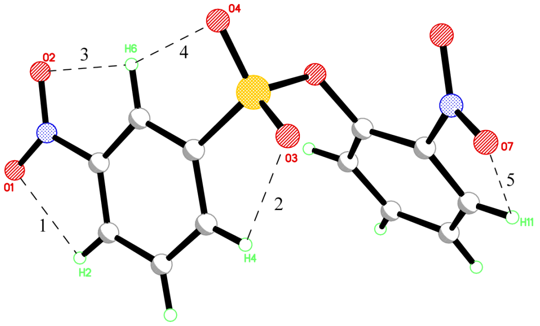


Figure 2
Diagram showing hydrogen bonds 1–5 as dashed lines(the numbers correspond to sequence of entries in Table 2).

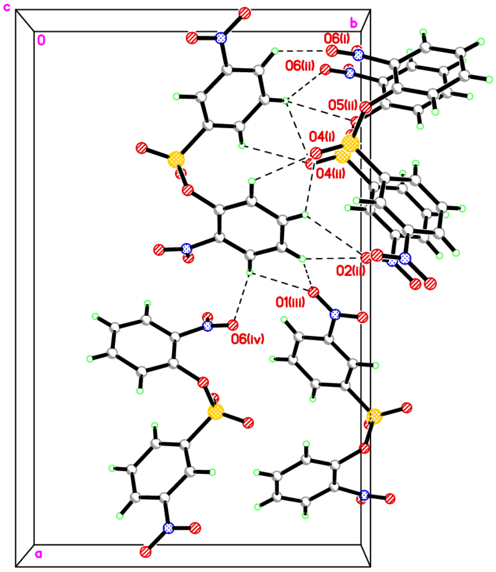


Figure 3
Diagram showing hydrogen bonds 6–17 as dashed lines (the numbers correspond to sequence of entries in Table 2). Symmetry codes are as in Table 2.

Youngs, 2003*a*) and 4-methoxyphenyl 4-toluenesulfonate (Vembu *et al.*, 2003).

The crystal structure 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···O3, C6—H6···O2, C6—H6···O4 and C11—H11···O7 interactions generates an S(5) graph-set motif (Bernstein *et al.*, 1995; Etter, 1990). The C6—H6···O2 and C6—H6···O4 interactions constitute a pair of bifurcated donor bonds. As seen in Fig. 3, the C8—H8···O4$^{\rm i}$ and C9—H9···O4$^{\rm i}$ interactions constitute a pair of bifurcated acceptor bonds generating an $R_2^1(5)$ motif. The C3—H3···O6$^{\rm ii}$ and C3—H3···O4$^{\rm ii}$ interactions constitute a pair of bifurcated donor bonds generating an $R_1^2(8)$ motif. The C3—H3···O4$^{\rm ii}$ and C4—H4···O4$^{\rm ii}$ interactions constitute a pair of bifurcated acceptor bonds generating an $R_2^1(5)$ motif. The C3—H3···O4$^{\rm ii}$ and C3—H3···O5$^{\rm ii}$ interactions constitute a pair of bifurcated donor bonds generating a symmetrical hydrogen-bonded chelate motif of graph-set $R_1^2(4)$. The C9—H9···O2$^{\rm ii}$ and C10—H10···O2$^{\rm ii}$ interactions together form a pair of bifurcated acceptor bonds generating an $R_2^1(5)$ motif. The C10—H10···O1$^{\rm iii}$ and C11—H11···O1$^{\rm iii}$ interactions together constitute a pair of bifurcated acceptor bonds generating an $R_2^1(5)$ motif. The C2—H2···O6$^{\rm i}$ and C11—H11···O6$^{\rm iv}$ interactions also contribute towards the supramolecular aggregation of the title compound (Fig. 4).

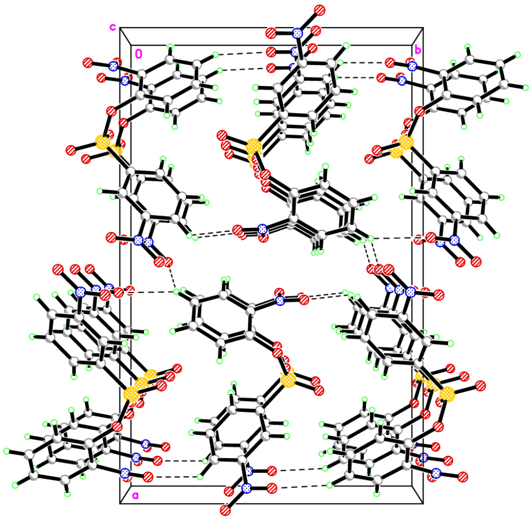


Figure 4
The packing of the molecules in the unit cell, viewed along the *c* axis, showing the network of hydrogen bonds.

Experimental

3–Nitrobenzenesulfonyl chloride (4.9 mmol) dissolved in acetone (5 ml) was added to 2-nitrophenol (5.0 mmol) in NaOH (4 ml, 5%) and shaken well. The crude title compound (2.6 mmol, yield: 53%) precipitated from solution. Diffraction quality crystals were obtained by recrystallizing the crude product from aqueous ethanol.

Crystal data

$C_{12}H_8N_2O_7S$
$M_r = 324.26$
Orthorhombic, $Pna2_1$
$a = 19.9671$ (8) Å
$b = 12.7083$ (5) Å
$c = 5.1306$ (2) Å
$V = 1301.88$ (9) Å^3
$Z = 4$
$D_x = 1.654$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2719 reflections
$\theta = 2.6$–26.7°
$\mu = 0.29$ mm^{-1}
$T = 120$ (2) K
Block **OR plate???**, colourless
0.14 × 0.12 × 0.08 mm

Data collection

Bruker SMART CCD 6K area-detector diffractometer
ω scans
9331 measured reflections
2832 independent reflections
2355 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.059$
$\theta_{\rm max} = 27.1^\circ$
$h = -24 \rightarrow 25$
$k = -16 \rightarrow 15$
$l = -6 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.070$
$S = 0.96$
2832 reflections
223 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.33$ e Å^{-3}
Absolute structure: (Flack, 1983), 1230 Friedel pairs
Flack parameter = −0.10 (7)

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Table 1
Selected geometric parameters (Å, °).

C1—N1	1.471 (3)	S1—O5	1.5992 (16)
C5—S1	1.765 (2)	O1—N1	1.227 (2)
C7—O5	1.408 (2)	O2—N1	1.228 (2)
C12—N2	1.469 (3)	O6—N2	1.226 (2)
S1—O4	1.4195 (16)	O7—N2	1.229 (3)
S1—O3	1.4250 (16)		
O4—S1—O3	121.34 (10)	O1—N1—O2	124.0 (2)
O4—S1—O5	103.53 (9)	O1—N1—C1	117.7 (2)
O3—S1—O5	108.83 (9)	O2—N1—C1	118.28 (16)
O4—S1—C5	109.93 (9)	O6—N2—O7	124.76 (19)
O3—S1—C5	108.15 (10)	O6—N2—C12	117.54 (18)
O5—S1—C5	103.59 (9)	O7—N2—C12	117.67 (19)
C7—O5—S1	118.08 (13)		
C5—S1—O5—C7	−71.90 (16)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1	0.93 (2)	2.40 (3)	2.708 (3)	99.2 (18)
C4—H4···O3	0.94 (3)	2.57 (2)	2.948 (3)	104.5 (17)
C6—H6···O2	1.03 (2)	2.43 (2)	2.711 (3)	94.2 (14)
C6—H6···O4	1.03 (2)	2.58 (2)	2.944 (3)	100.4 (16)
C11—H11···O7	0.91 (2)	2.57 (2)	2.788 (3)	94.4 (16)
C8—H8···O4$^{\rm i}$	0.97 (2)	2.72 (2)	3.359 (3)	123.8 (17)
C9—H9···O4$^{\rm i}$	0.95 (3)	2.73 (3)	3.374 (3)	125.3 (18)
C2—H2···O6$^{\rm i}$	0.93 (2)	2.48 (2)	3.092 (3)	123.5 (18)
C3—H3···O6$^{\rm ii}$	0.98 (3)	2.98 (3)	3.836 (3)	147.1 (19)
C3—H3···O4$^{\rm ii}$	0.98 (3)	2.78 (3)	3.409 (3)	122.5 (18)
C9—H9···O2$^{\rm ii}$	0.95 (3)	2.88 (2)	3.398 (3)	115.6 (18)
C4—H4···O4$^{\rm ii}$	0.94 (3)	2.86 (2)	3.401 (3)	117.3 (18)
C10—H10···O2$^{\rm ii}$	0.93 (2)	2.54 (2)	3.236 (3)	131.4 (18)
C3—H3···O5$^{\rm ii}$	0.98 (3)	2.93 (2)	3.842 (3)	156 (2)
C10—H10···O1$^{\rm iii}$	0.93 (2)	2.51 (2)	3.177 (3)	129.2 (19)
C11—H11···O1$^{\rm iii}$	0.91 (2)	2.79 (2)	3.300 (3)	116.2 (17)
C11—H11···O6$^{\rm iv}$	0.91 (2)	2.60 (2)	3.484 (3)	163.5 (19)

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

All 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 0.91 (2)–1.03 (2) Å.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (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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