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2,4-Dinitrophenyl 3-nitrobenzenesulfonate

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In the title molecule, $C_{12}H_7N_3O_9S$, there are weak C—H···O interactions which generate rings of motifs $S(5)$, $R_1^2(8)$ and $R_1^2(4)$.

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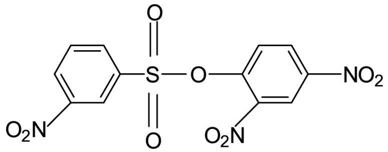
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.059
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.

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 structures of 3-nitrobenzenesulfonyl chloride have been recently reported (Vembu, Nallu, Spencer & Howard, 2003*c*). 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.



(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 0.005 (3), −0.248 (4) and 0.267 (4) Å, respectively. Atoms N2, O6, O7, N3, O8 and O9 deviate from the mean plane formed by atoms C7–C12 by 0.069 (3), −0.849 (3), 1.051 (3), 0.026 (3), 0.341 (4) and −0.277 (4) Å, respectively. The dihedral angle between the above-mentioned planes is 36.10 (7)°. This shows their non-coplanar orientation, similar to that reported for some aromatic sulfonates (Vembu, Nallu, Garrison & Youngs, 2003*b*–*e*; Vembu, Nallu, Spencer &

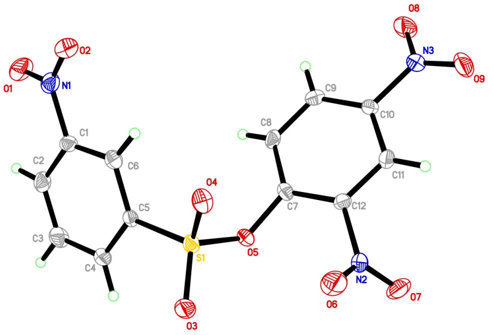


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme..

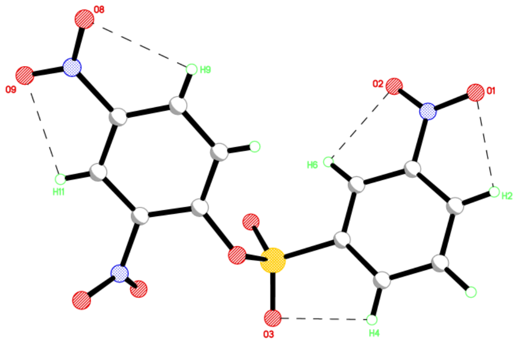


Figure 2
Diagram showing hydrogen bonds 1–3, 5 and 6 as dashed lines (the numbers correspond to the sequence of entries in Table 2).

Howard, 2003*a*,*b*,*d*,*e*), and in contrast with the near-coplanar orientation found in 2,4-dinitrophenyl 4-toluenesulfonate (Vembu, Nallu, Garrison & Youngs, 2003*a*) and 4-methoxyphenyl 4-toluenesulfonate (Vembu *et al.*, 2003).

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 C2—H2···O1, C4—H4···O3, C6—H6···O2, C6—H6···O4, C9—H9···O8 and C11—H11···O9 interaction 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 can be seen in Fig. 3, the C3—H3···O3^{ii} and C3—H3···O6^{ii} interactions constitute a pair of bifurcated donor bonds generating an $R_1^2(8)$ motif. The C6—H6···N3^{iv}, C6—H6···O8^{iv} and C6—H6···O9^{iv} interactions together constitute a set of trifurcated donor bonds. The C6—H6···O8^{iv} and C6—H6···O9^{iv} interactions generate a symmetrical three-centred hydrogen-bonded chelate motif of graph set $R_1^2(4)$. There are several other weak C—H···O interactions which contribute to the supramolecular aggregation of (I) (Table 2). Other short intermolecular contacts are C9···O9($1-x$, $1-y$, $z-\frac{1}{2}$) 3.130 (3) Å, N1···O8($1-x$, $2-y$, $z-\frac{1}{2}$) 2.818 (2) Å and N1···O7(x, y, $z-1$) 3.037 (2) Å. A view of the molecular packing down the *b* axis is shown in Fig. 4.

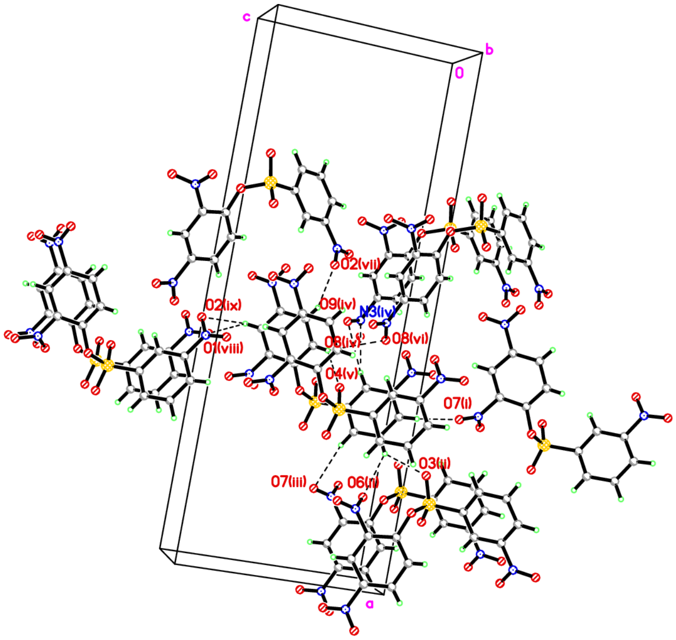


Figure 3
Diagram showing hydrogen bonds 7–18 as dashed lines (the numbers correspond to the sequence of entries in Table 2). Symmetry codes are as in Table 2.

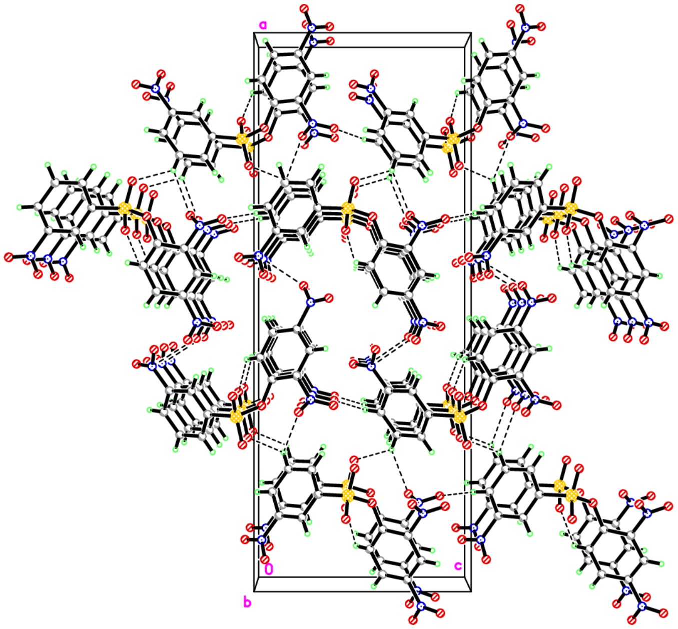


Figure 4
Packing of the molecules in the unit cell of (I), viewed along the *b* axis, showing the network of hydrogen bonds as dashed lines.

Experimental

2,4-Dinitrophenol (5 mmol) and triethylamine (5 mmol) were dissolved in acetone (20 ml). To this solution, 3-nitrobenzenesulfonyl chloride (5 mmol) in acetone (20 ml) was added. The resulting solution was allowed to evaporate. The residue was washed several times with aqueous triethylamine solution. The crude product of the title compound, (I), was recrystallized from a 1:1 mixture of petroleum ether and ethanol.

Crystal data

$C_{12}H_7N_3O_9S$
$M_r = 369.27$
Orthorhombic, $Pca2_1$
$a = 27.730$ (2) Å
$b = 4.7374$ (3) Å
$c = 10.7800$ (8) Å
$V = 1416.16$ (17) Å^3
$Z = 4$
$D_x = 1.732$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3073 reflections
$\theta = 2.4$–27.1°
$\mu = 0.29$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.28 × 0.25 × 0.11 mm

Data collection

Bruker SMART CCD 6 K area-detector diffractometer
ω scans
8989 measured reflections
2952 independent reflections
2619 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.033$
$\theta_{\max} = 27.1°$
$h = -35 \rightarrow 27$
$k = -6 \rightarrow 5$
$l = -13 \rightarrow 12$

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.059$
$S = 0.97$
2952 reflections
247 parameters
Only coordinates of H atoms refined

$w = 1/[\sigma^2(F_o^2) + (0.0274P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}
Absolute structure: (Flack, 1983); 1310 Friedel pairs
Flack parameter = −0.05 (6)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.473 (3)	N1—O1	1.225 (2)
C5—S1	1.753 (2)	N1—O2	1.227 (2)
C10—N3	1.481 (2)	N2—O6	1.211 (2)
C12—N2	1.477 (2)	N2—O7	1.224 (2)
S1—O3	1.4147 (15)	N3—O8	1.216 (2)
S1—O4	1.4182 (13)	N3—O9	1.216 (2)
S1—O5	1.6222 (15)		
O3—S1—O4	122.42 (8)	O2—N1—C1	117.88 (17)
O3—S1—O5	104.06 (8)	O6—N2—O7	125.83 (17)
O4—S1—O5	107.75 (8)	O6—N2—C12	118.07 (17)
O3—S1—C5	108.77 (9)	O7—N2—C12	116.08 (17)
O4—S1—C5	108.53 (10)	O8—N3—O9	124.40 (18)
O5—S1—C5	103.71 (8)	O8—N3—C10	118.16 (17)
O1—N1—O2	124.03 (18)	O9—N3—C10	117.44 (17)
O1—N1—C1	118.10 (18)	C7—O5—S1	120.15 (12)
C5—S1—O5—C7	103.41 (15)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1	0.95 (2)	2.43 (2)	2.742 (3)	99.0 (15)
C4—H4···O3	0.94 (2)	2.63 (2)	2.944 (3)	100.4 (15)
C6—H6···O2	0.95 (2)	2.50 (2)	2.715 (3)	92.7 (15)
C6—H6···O4	0.95 (2)	2.49 (2)	2.898 (2)	106.4 (16)
C9—H9···O8	0.86 (2)	2.53 (2)	2.745 (3)	94.9 (18)
C11—H11···O9	0.90 (2)	2.46 (2)	2.699 (3)	95.3 (16)
C2—H2···O7$^{\text{i}}$	0.95 (2)	2.45 (2)	3.374 (3)	166.2 (19)
C3—H3···O3$^{\text{ii}}$	0.97 (2)	2.43 (2)	3.208 (3)	137.0 (18)
C3—H3···O6$^{\text{ii}}$	0.97 (2)	2.47 (2)	3.263 (3)	138.2 (18)
C4—H4···O7$^{\text{iii}}$	0.94 (2)	2.99 (2)	3.781 (3)	142.9 (17)
C6—H6···N3$^{\text{iv}}$	0.95 (2)	2.93 (2)	3.836 (3)	161.2 (18)
C6—H6···O8$^{\text{iv}}$	0.95 (2)	2.77 (2)	3.652 (3)	155.7 (19)
C6—H6···O9$^{\text{iv}}$	0.95 (2)	2.70 (2)	3.572 (3)	153.6 (17)
C8—H8···O4$^{\text{v}}$	0.81 (2)	2.43 (2)	3.008 (2)	129.2 (19)
C8—H8···O8$^{\text{vi}}$	0.81 (2)	2.96 (2)	3.681 (3)	150.0 (18)
C9—H9···O2$^{\text{vii}}$	0.86 (2)	2.97 (2)	3.726 (3)	147 (2)
C11—H11···O1$^{\text{viii}}$	0.90 (2)	2.58 (2)	3.362 (3)	145.1 (18)
C11—H11···O2$^{\text{ix}}$	0.90 (2)	2.62 (2)	3.240 (3)	126.4 (17)

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

All H atoms were located from difference Fourier maps and their positional parameters were refined, with $U_{\text{iso}} = 1.2U_{\text{eq}}$(parent atom). The C—H bond lengths are in the range 0.81 (2)–0.97 (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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