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Methyl 4-(3-nitrobenzenesulfonyloxy)benzoate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.082
Data-to-parameter ratio = 13.1

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

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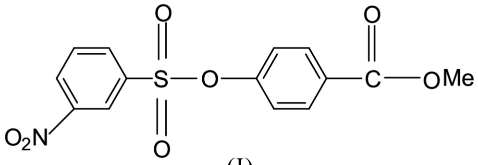
In the title molecule, $C_{14}H_{11}NO_7S$, (I), there are weak C—H⋯O interactions which generate rings of motifs *S*(5), *S*(6), $R_2^1(5)$ and $R_2^2(7)$. The supramolecular aggregation is completed by the presence of C—H⋯π and π–π interactions.

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*) and its analogue phenyl 3-nitrobenzenesulfonate (Vembu, Nallu, Spencer & Howard, 2003*d*).


(I)

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters in Table 1. Atoms C13, O6, O7 and C14 deviate by −0.189 (3), −0.512 (3), 0.015 (3) and −0.229 (4) Å, respectively, from the mean plane formed by the atoms C7–C12. The dihedral angle between the mean planes of the 3-nitrobenzene and benzoate phenyl rings is 58.4 (5)°. This non-coplanar orientation is similar to that found in previous 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

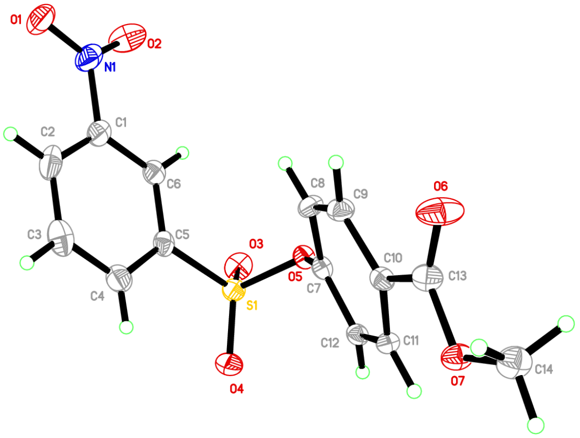


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

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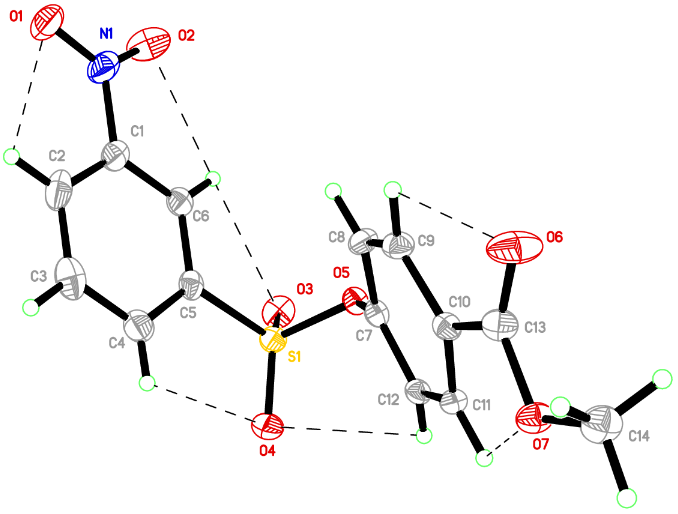

Figure 2
Diagram showing hydrogen bonds 1–7 (the numbers relate to the sequence of entries in Table 2).

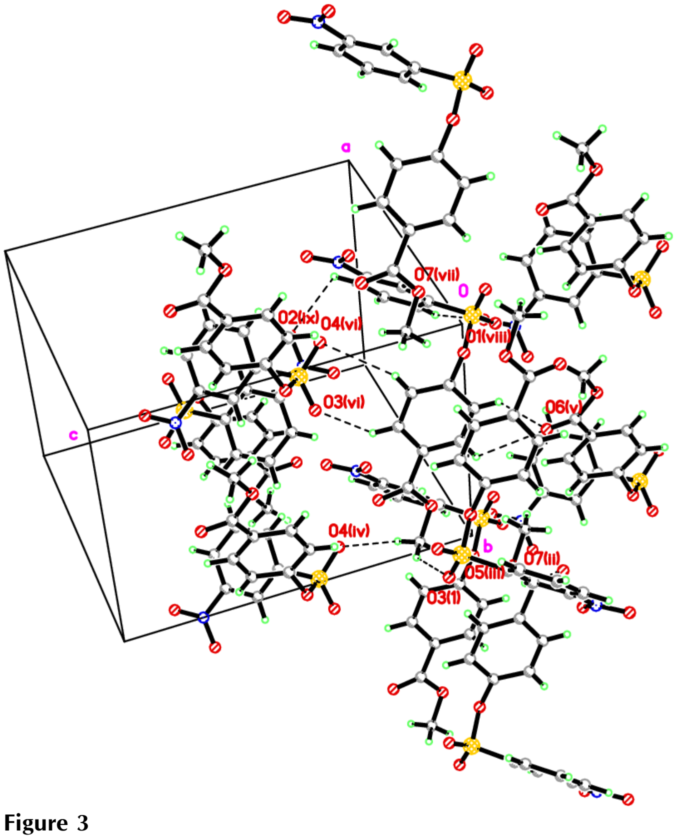

Figure 3
Diagram showing the intermolecular hydrogen bonds. Symmetry codes are given in Table 2.

orientation found in the 2,4-dinitrophenyl (Vembu, Nallu, Garrison & Youngs, 2003*a*) and 4-methoxyphenyl (Vembu, Nallu, Garrison, Hindi & Youngs, 2003) derivatives. 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).

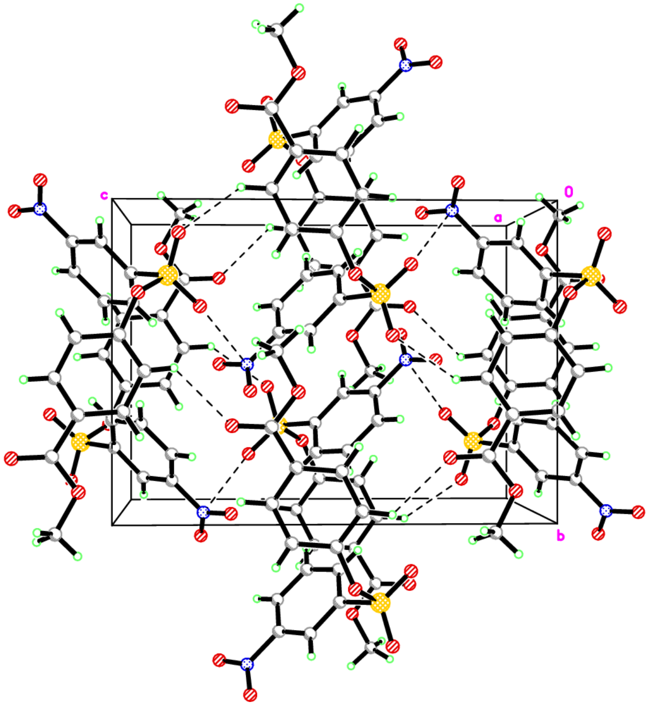

Figure 4
Packing of the molecule in the unit cell, viewed along the *a* axis, showing the network of C—H···O interactions.

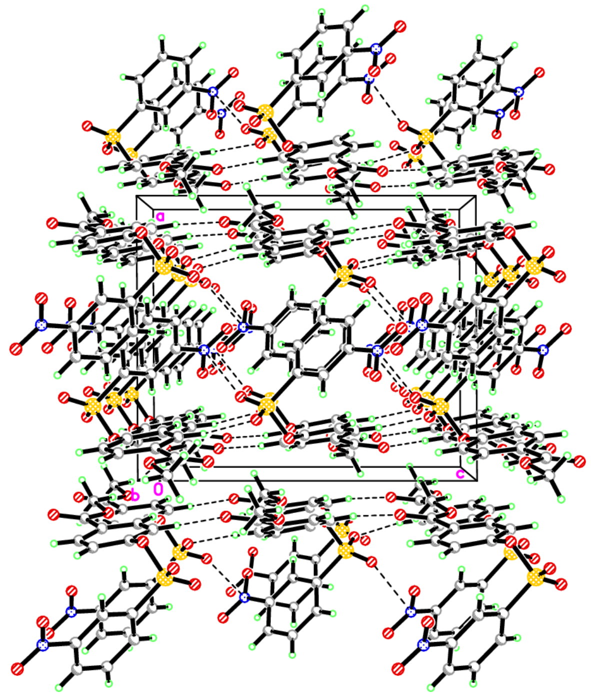

Figure 5
Packing of the molecule in the unit cell, viewed along the *b* axis, showing the C—H···π and π–π-stacking interactions.

As shown in Fig. 2, each of the C2—H2···O1, C4—H4···O4, C6—H6···O2, C6—H6···O3, C9—H9···O6, C11—H11···O7 and C12—H12···O4 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$\cdots$O4 interaction generates a ring of graph-set motif *S*(6). As can be seen in Fig. 3, the C9—H9$\cdots$O3^{iv} and C8—H8$\cdots$O4^{iv} interactions together generate a sulfonyl bifurcated motif of graph-set $R_2^2(7)$. The C12—H12$\cdots$O6^{v} and C11—H11$\cdots$O6^{v} interactions constitute a pair of bifurcated acceptor bonds, generating a ring of graph-set $R_2^1(5)$. There are several other C—H$\cdots$O interactions which contribute to the supramolecular aggregation of the structure. In the crystal structure (Figs. 4 and 5), the inversion-related benzoate phenyl rings (symmetry code: $-x$, $1 - y$, z) are stacked with a typical centroid–centroid separation of 3.663 Å, suggesting weak π–π interactions. The supramolecular aggregation is completed by the presence of a C—H$\cdots\pi$ interaction (Table 2; Spek, 1998).

Experimental

3-Nitrobenzenesulfonyl chloride (5 mmol) dissolved in acetone (4 ml) was added to methyl 4-hydroxybenzoate (5 mmol) in NaOH solution (2.5 ml, 8%) with constant shaking. The precipitated title compound (I) (2.6 mmol, yield 52%) was filtered and recrystallized from a 1:1 mixture of acetone and petroleum ether.

Crystal data

$C_{14}H_{11}NO_7S$
$M_r = 337.30$
Monoclinic, $P2_1/c$
$a = 11.125$ (2) Å
$b = 9.643$ (2) Å
$c = 13.236$ (3) Å
$\beta = 90.257$ (5)°
$V = 1419.9$ (5) Å^3
$Z = 4$

$D_x = 1.578$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 928 reflections
$\theta = 3.2$–27.1°
$\mu = 0.27$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.22 × 0.13 × 0.12 mm

Data collection

Bruker Proteum *M* diffractometer
ω scans
Absorption correction: none
9472 measured reflections
3150 independent reflections
2405 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.030$
$\theta_{\rm max} = 27.2°$
$h = -13 \rightarrow 14$
$k = -12 \rightarrow 9$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.082$
$S = 0.95$
3150 reflections
241 parameters

Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.39$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O3	1.4163 (12)	N1—C1	1.474 (2)
S1—O4	1.4206 (13)	O5—C7	1.4219 (19)
S1—O5	1.5894 (12)	O6—C13	1.205 (2)
S1—C5	1.7573 (17)	O7—C13	1.338 (2)
N1—O2	1.225 (2)	O7—C14	1.447 (2)
N1—O1	1.225 (2)		
O3—S1—O4	120.90 (8)	O2—N1—O1	124.60 (17)
O3—S1—O5	102.93 (7)	O2—N1—C1	117.83 (16)
O4—S1—O5	109.67 (7)	O1—N1—C1	117.57 (17)
O3—S1—C5	109.59 (8)	C7—O5—S1	120.64 (9)
O4—S1—C5	109.51 (8)	C13—O7—C14	114.92 (14)
O5—S1—C5	102.53 (7)		
C5—S1—O5—C7	64.78 (13)		

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

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C2—H2$\cdots$O1	0.93 (2)	2.438 (19)	2.717 (3)	97.2 (13)
C4—H4$\cdots$O4	0.886 (19)	2.571 (18)	2.938 (2)	105.8 (13)
C6—H6$\cdots$O2	0.920 (18)	2.422 (18)	2.702 (2)	97.6 (13)
C6—H6$\cdots$O3	0.920 (18)	2.696 (18)	2.996 (2)	100.0 (13)
C9—H9$\cdots$O6	0.946 (18)	2.543 (17)	2.816 (2)	96.8 (11)
C11—H11$\cdots$O7	0.928 (16)	2.479 (16)	2.771 (2)	98.4 (11)
C12—H12$\cdots$O4	0.915 (18)	2.855 (16)	3.184 (2)	102.7 (12)
C2—H2$\cdots$O2^{i}	0.93 (2)	2.796 (19)	3.309 (3)	115.7 (14)
C4—H4$\cdots$O1^{ii}	0.886 (19)	2.596 (19)	3.454 (3)	163.2 (15)
C6—H6$\cdots$O7^{iii}	0.920 (18)	2.707 (19)	3.599 (2)	163.5 (15)
C9—H9$\cdots$O3^{iv}	0.946 (18)	2.401 (18)	3.198 (2)	141.6 (13)
C8—H8$\cdots$O4^{iv}	0.932 (18)	2.998 (18)	3.870 (2)	156.4 (14)
C12—H12$\cdots$O6^{v}	0.915 (18)	2.489 (18)	3.152 (2)	129.6 (13)
C11—H11$\cdots$O6^{v}	0.928 (16)	2.805 (16)	3.324 (2)	116.5 (11)
C14—H14*A*$\cdots$O4^{vi}	0.98 (2)	2.74 (2)	3.712 (2)	173.3 (15)
C14—H14*B*$\cdots$O5^{vii}	0.953 (19)	2.67 (2)	3.528 (2)	149.7 (15)
C14—H14*B*$\cdots$O7^{viii}	0.953 (19)	2.839 (19)	3.497 (2)	127.1 (13)
C14—H14*C*$\cdots$O3^{ix}	0.96 (2)	2.721 (19)	3.536 (2)	143.2 (15)
C3—H3$\cdots$*Cg*2^{x}	0.95 (2)	2.85	3.70	149

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

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 0.89 (2)–0.98 (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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