

Nagarajan Vembu,^a Maruthai Nallu,^{a*} Elinor C. Spencer^b and Judith A. K. Howard^b

^aDepartment of Chemistry, Bharathidasan University, Tiruchirappalli 620 024, India, and

^bDepartment of Chemistry, Durham University, Durham DH1 3LE, England

Correspondence e-mail: mnalv2003@yahoo.com

Key indicators

Single-crystal X-ray study

$T = 120$ K

Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å

R factor = 0.038

wR factor = 0.078

Data-to-parameter ratio = 12.6

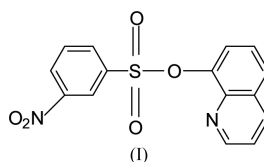
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

8-Quinolyl 3-nitrobenzenesulfonate

In the title molecule, $\text{C}_{15}\text{H}_{10}\text{N}_2\text{O}_5\text{S}$, there are weak $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{N}$ interactions which generate rings of motifs $S(5)$, $S(6)$, $S(8)$, $R_2^1(5)$, $R_2^1(6)$, $R_1^1(4)$, $R_2^2(6)$, $R_2^2(7)$, $R_2^2(8)$ and $R_2^2(13)$. The supramolecular aggregation is completed by the presence of $\pi-\pi$ interactions.

Comment

Aromatic sulfonates are used in monitoring the merging of lipids (Yachi *et al.*, 1989), studying membrane fusion during an acrosome reaction (Spungin *et al.*, 1992), in the development of immunoaffinity chromatography for the purification of human coagulation factor (Tharakan *et al.*, 1992), in chemical studies of viruses (Alford *et al.*, 1991), in the development of technology for linking photosensitizers to model monoclonal antibodies (Jiang *et al.*, 1990) and in the chemical modification of σ sub-units of *E. coli* RNA polymerase (Narayanan & Krakow, 1983). The molecular and crystal structure of 3-nitrobenzenesulfonyl chloride has recently been reported (Vembu, Nallu, Spencer & Howard, 2003c). An X-ray study of the title compound, (I), has now been 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.



The title compound, (I), crystallizes with two independent molecules per asymmetric unit (Fig. 1). Selected geometric parameters are given in Table 1. The dihedral angles between the mean planes of the 3-nitrobenzene moiety and the quinoline rings are $8.28(5)$ and $5.18(5)^\circ$ in molecules 1 and 2,

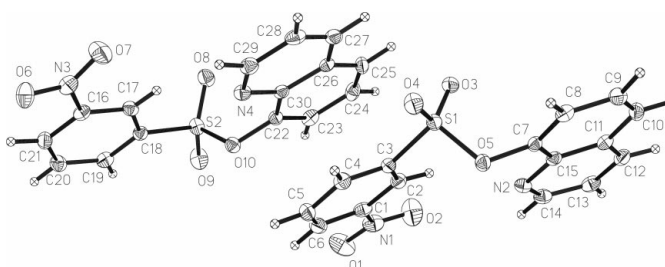


Figure 1

The structure of the two independent molecules of (I), showing 50% probability displacement ellipsoids.

respectively. This near-coplanar orientation is similar to that found in related aromatic sulfonates (Vembu *et al.*, 2003; Vembu, Nallu, Garrison & Youngs, 2003*a*), and is in contrast with the non-coplanar orientation reported for similar compounds (Vembu, Nallu, Garrison & Youngs, 2003*b,c,e,f*; Vembu, Nallu, Spencer & Howard, 2003*a,b*). The crystal structure of (I) is stabilized by weak C—H···O and C—H···N 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···O2, C2—H2···O3, C4—H4···O4, C6—H6···O1, C17—H17···O7, C17—H17···O10, C19—H19···O9 and C21—H21···O6 interaction generates rings of graph-set motif $S(5)$ (Etter, 1990; Bernstein *et al.*, 1995). The C2—H2···N2, C2—H2···O2 and C2—H2···O3 interactions constitute one set of heteroatomic trifurcated donor bonds, and the C17—H17···N4, C17—H17···O7 and C17—H17···O10 interactions constitute a second set of heteroatomic trifurcated donor bonds. The C25—H25···O3 and C27—H27···O3 interactions constitute a pair of bifurcated acceptor bonds generating an $S(6)$ motif. Each C2—H2···N2 and C17—H17···N4 interaction generates rings of graph-set motif $S(8)$.

As can be seen in Fig. 3, the C4—H4···O8ⁱ and C5—H5···O9ⁱ interactions generate a sulfonyl fork motif of graph set $R_2^2(7)$. The C9—H9···O6ⁱⁱⁱ and C8—H8···N3ⁱⁱⁱ interactions generate an $R_2^2(6)$ motif. The C9—H9···O6ⁱⁱⁱ and C8—H8···O6ⁱⁱⁱ interactions constitute a pair of bifurcated acceptor bonds generating an $R_2^1(5)$ motif. The C9—H9···O6ⁱⁱⁱ and C8—H8···O7ⁱⁱⁱ interactions generate a nitro fork motif of graph set $R_2^2(7)$. The C8—H8···O6ⁱⁱⁱ and C8—H8···O7ⁱⁱⁱ interactions constitute a pair of bifurcated donor bonds generating a three-centred hydrogen-bonded chelate motif of graph set $R_2^1(4)$ (Vembu *et al.*, 2003; Vembu, Nallu, Garrison & Youngs, 2003*b,c,d,e,f,g*; Vembu, Nallu, Spencer & Howard, 2003*a,b,d,e*). The C8—H8···N3ⁱⁱⁱ, C8—H8···O6ⁱⁱⁱ and C8—H8···O7ⁱⁱⁱ interactions together constitute a set of heteroatomic trifurcated donor bonds. The C9—H9···O10^{iv} and C10—H10···N4^{iv} interactions together generate an $R_2^2(8)$ ring

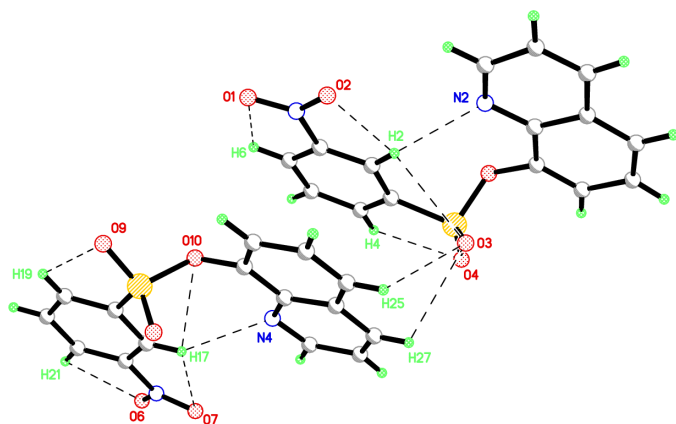


Figure 2
Hydrogen bonds 1–12 as dashed lines (the numbers relate to the sequence of entries in Table 2).

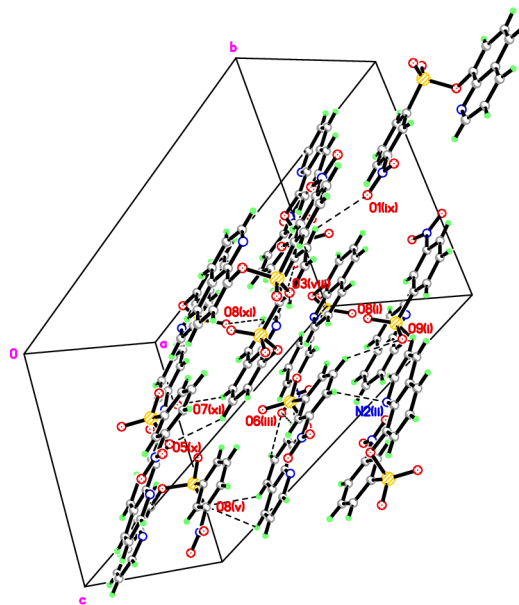


Figure 3
Hydrogen bonds 14–16, 18, 23, 24, 31, 32, 33, 34 and 35 as dashed lines (the numbers relate to the sequence of entries in Table 2). Symmetry codes are as in Table 2.

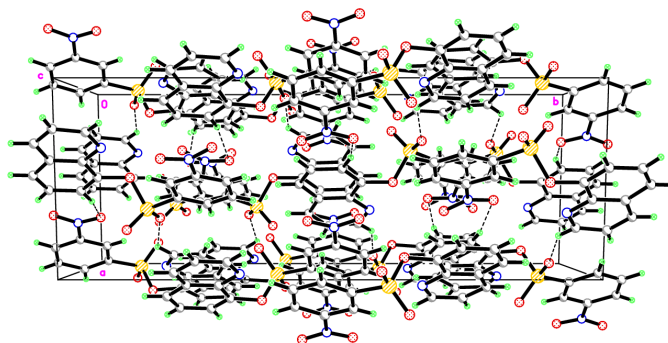


Figure 4
Packing of the molecules in the unit cell of (I), viewed along the *c* axis, showing the π – π interactions as dashed lines.

motif. The C10—H10···O8^v and C12—H12···O8^v interactions constitute a pair of bifurcated acceptor bonds generating an $R_2^1(6)$ motif. The C12—H12···O4^{vi} and C13—H13···O3^{vi} interactions generate a sulfonyl fork motif of graph set $R_2^2(7)$. The C24—H24···O1^{vii} and C23—H23···O2^{vii} interactions generate a nitro fork motif of graph set $R_2^2(7)$. The C14—H14···O9^{viii} and C13—H13···O9^{viii} interactions constitute a pair of bifurcated acceptor bonds forming an $R_2^1(5)$ motif. The C28—H28···O8^{xi} and C25—H25···O7^{xi} interactions together generate an $R_2^2(13)$ ring motif. There are several other C—H···O interactions which contribute to the supramolecular aggregation of the structure (Table 2).

In the crystal structure of (I) (Fig. 4), there are five pairs of π – π interactions, with separations of 3.604 (1), 3.450 (1), 3.786 (1), 3.596 (1) and 3.781 (1) Å between Cg_1 and $Cg_5(1-x, y-\frac{1}{2}, \frac{3}{2}-z)$, Cg_2 and $Cg_2(1-x, 1-y, 1-z)$, Cg_2 and Cg_3 , Cg_3 and Cg_6 , and Cg_4 and $Cg_5(2-x, y-\frac{1}{2}, \frac{3}{2}-z)$, respec-

tively, where Cg1, Cg2, Cg3, Cg4, Cg5 and Cg6 are the centroids of the rings formed by the atoms N2/C11–C15, N4/C26–C30, C1–C6, C7–C11/C15, C16–C21 and C22–C26/C30, respectively (Spek, 1998).

Experimental

3-Nitrobenzenesulfonyl chloride (5 mmol) was dissolved in acetone (5 ml) and added to 8-hydroxyquinoline (5 mmol), which had been dissolved in NaOH (4 ml, 5%). The precipitate, (I) (5 mmol, yield 60%), was recrystallized from ethyl acetate.

Crystal data

C ₁₅ H ₁₀ N ₂ O ₅ S	$D_x = 1.614 \text{ Mg m}^{-3}$
$M_r = 330.31$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2947 reflections
$a = 8.6001(6) \text{ \AA}$	$\theta = 3.0\text{--}26.6^\circ$
$b = 23.0681(16) \text{ \AA}$	$\mu = 0.27 \text{ mm}^{-1}$
$c = 13.9192(9) \text{ \AA}$	$T = 120(2) \text{ K}$
$\beta = 100.132(2)^\circ$	Block, colourless
$V = 2718.3(3) \text{ \AA}^3$	$0.20 \times 0.19 \times 0.11 \text{ mm}$
$Z = 8$	

Data collection

Bruker Proteum <i>M</i> diffractometer	$R_{\text{int}} = 0.050$
ω scans	$\theta_{\text{max}} = 27.2^\circ$
18 512 measured reflections	$h = -10 \rightarrow 11$
6011 independent reflections	$k = -29 \rightarrow 28$
4101 reflections with $I > 2\sigma(I)$	$l = -17 \rightarrow 15$

Refinement

Refinement on F^2	Only coordinates of H atoms refined
$R[F^2 > 2\sigma(F^2)] = 0.038$	$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2]$
$wR(F^2) = 0.078$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.90$	$(\Delta/\sigma)_{\text{max}} = 0.001$
6011 reflections	$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
475 parameters	$\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

C1–N1	1.483 (2)	C16–N3	1.475 (2)
C3–S1	1.7561 (18)	C18–S2	1.7606 (19)
C7–O5	1.417 (2)	C22–O10	1.413 (2)
C14–N2	1.319 (2)	C29–N4	1.322 (2)
C15–N2	1.368 (2)	C30–N4	1.368 (2)
S1–O4	1.4242 (13)	S2–O9	1.4208 (14)
S1–O3	1.4270 (13)	S2–O8	1.4278 (14)
S1–O5	1.5909 (13)	S2–O10	1.5938 (13)
N1–O2	1.221 (2)	N3–O6	1.223 (2)
N1–O1	1.223 (2)	N3–O7	1.228 (2)
O4–S1–O3	119.43 (8)	O9–S2–O8	119.27 (9)
O4–S1–O5	108.26 (8)	O9–S2–O10	108.09 (8)
O3–S1–O5	108.56 (7)	O8–S2–O10	108.40 (8)
O4–S1–C3	108.84 (9)	O9–S2–C18	108.59 (9)
O3–S1–C3	109.91 (8)	O8–S2–C18	110.24 (9)
O5–S1–C3	100.10 (8)	O10–S2–C18	100.62 (8)
O2–N1–O1	124.52 (17)	O6–N3–O7	124.29 (17)
O2–N1–C1	117.70 (17)	O6–N3–C16	118.16 (17)
O1–N1–C1	117.78 (18)	O7–N3–C16	117.55 (16)
C14–N2–C15	116.65 (17)	C29–N4–C30	116.77 (17)
C7–O5–S1	117.13 (11)	C22–O10–S2	118.15 (11)
C3–S1–O5–C7	138.04 (13)	C18–S2–O10–C22	–135.79 (13)

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
C2–H2 \cdots N2	0.949 (19)	2.51 (2)	3.281 (3)	138.2 (15)
C2–H2 \cdots O2	0.949 (19)	2.379 (18)	2.691 (2)	98.7 (13)
C2–H2 \cdots O3	0.949 (19)	2.808 (18)	3.023 (2)	93.8 (12)
C4–H4 \cdots O4	0.960 (19)	2.568 (19)	2.926 (2)	102.3 (13)
C6–H6 \cdots O1	0.98 (2)	2.427 (19)	2.734 (2)	97.6 (13)
C17–H17 \cdots N4	0.953 (19)	2.49 (2)	3.336 (3)	147.6 (15)
C17–H17 \cdots O7	0.953 (19)	2.438 (18)	2.704 (2)	95.7 (13)
C17–H17 \cdots O10	0.953 (19)	2.833 (18)	3.056 (2)	94.3 (12)
C19–H19 \cdots O9	0.926 (19)	2.526 (19)	2.911 (2)	105.3 (14)
C21–H21 \cdots O6	0.93 (2)	2.45 (2)	2.726 (2)	96.6 (14)
C25–H25 \cdots O3	0.95 (2)	2.968 (19)	3.382 (2)	107.9 (14)
C27–H27 \cdots O3	0.95 (2)	2.810 (19)	3.249 (2)	109.2 (14)
C4–H4 \cdots O8 ⁱ	0.960 (19)	2.977 (19)	3.665 (2)	129.7 (14)
C5–H5 \cdots O9 ⁱ	0.96 (2)	2.96 (2)	3.749 (3)	140.2 (15)
C6–H6 \cdots N2 ⁱⁱ	0.98 (2)	2.68 (2)	3.377 (3)	128.6 (15)
C9–H9 \cdots O6 ⁱⁱⁱ	0.960 (19)	2.661 (19)	3.344 (2)	128.4 (15)
C8–H8 \cdots N3 ⁱⁱⁱ	0.952 (19)	2.970 (19)	3.807 (2)	147.4 (14)
C8–H8 \cdots O6 ⁱⁱⁱ	0.952 (19)	2.761 (19)	3.380 (2)	123.4 (14)
C8–H8 \cdots O7 ⁱⁱⁱ	0.952 (19)	2.506 (19)	3.444 (2)	168.3 (16)
C29–H29 \cdots O4 ⁱⁱⁱ	0.96 (2)	2.495 (19)	3.305 (2)	141.8 (16)
C9–H9 \cdots O10 ^{iv}	0.960 (19)	2.82 (2)	3.566 (2)	134.9 (15)
C10–H10 \cdots N4 ^{iv}	0.963 (19)	2.67 (2)	3.346 (2)	127.7 (15)
C10–H10 \cdots O8 ^v	0.963 (19)	2.99 (2)	3.391 (2)	106.5 (13)
C12–H12 \cdots O8 ^v	0.94 (2)	2.94 (2)	3.313 (2)	105.3 (14)
C12–H12 \cdots O4 ^{vi}	0.94 (2)	2.71 (2)	3.547 (2)	147.9 (16)
C13–H13 \cdots O3 ^{vi}	0.98 (2)	2.79 (2)	3.509 (2)	130.6 (15)
C24–H24 \cdots O1 ^{vii}	0.96 (2)	2.78 (2)	3.517 (3)	133.8 (15)
C14–H14 \cdots O9 ^{vii}	0.99 (2)	2.491 (19)	3.217 (2)	129.9 (15)
C13–H13 \cdots O9 ^{vii}	0.98 (2)	2.77 (2)	3.336 (2)	117.5 (14)
C23–H23 \cdots O2 ^{vii}	0.993 (19)	2.479 (19)	3.412 (2)	156.3 (15)
C19–H19 \cdots O3 ^{viii}	0.926 (19)	2.76 (2)	3.435 (2)	130.6 (15)
C21–H21 \cdots O1 ^{ix}	0.93 (2)	2.87 (2)	3.694 (3)	147.2 (16)
C24–H24 \cdots O5 ^x	0.96 (2)	2.81 (2)	3.651 (2)	146.8 (16)
C28–H28 \cdots O8 ^{xi}	0.91 (2)	2.78 (2)	3.387 (2)	125.9 (16)
C25–H25 \cdots O7 ^{xi}	0.95 (2)	2.68 (2)	3.543 (3)	151.7 (16)

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

All H atoms were located from difference Fourier maps and their positional parameters were refined. Their isotropic displacement parameters were set at values 20% greater than those of their bonded partners. The C–H bond lengths are in the range 0.90 (2)–0.99 (2) \AA . Reflections were measured to $\theta_{\text{max}} = 27.16^\circ$ with 97% completeness, but the data are 100% complete to 25° .

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*.

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

References

- Alford, R. L., Honda, S., Lawrence, C. B. & Belmont, J. W. (1991). *Virology*, **183**, 611–619.
- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N. (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.
- Jiang, F. N., Jiang, S., Liu, D., Richter, A. & Levy, J. G. (1990). *J. Immunol. Methods* **134**, 139–149.
- Narayanan, C. S. & Krakow, J. S. (1983). *Nucleic Acids Res.* **11**, 2701–2716.
- Sheldrick, G. M. (1998). *SHELXTL*. University of Göttingen, Germany.
- Spek, A. L. (1998). *PLATON*. University of Utrecht, The Netherlands.
- Spungin, B., Levinshal, T., Rubenstein, S. & Breitbart, H. (1992). *FEBS Lett.* **311**, 155–160.
- Tharakan, J., Highsmith, F., Clark, D. & Drohns, W. (1992). *J. Chromatogr.* **595**, 103–111.
- Vembu, N., Nallu, M., Garrison, J., Hindi, K. & Youngs, W. J. (2003). *Acta Cryst. E59*, o830–o832.
- Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003a). *Acta Cryst. E59*, o378–o380.
- Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003b). *Acta Cryst. E59*, o503–o505.
- Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003c). *Acta Cryst. E59*, o776–o779.
- Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003d). *Acta Cryst. E59*, o913–o916.
- Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003e). *Acta Cryst. E59*, o936–o938.
- Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003f). *Acta Cryst. E59*, o939–o941.
- Vembu, N., Nallu, M., Garrison, J. & Youngs, W. J. (2003g). *Acta Cryst. E59*, o1019–o1021.
- Vembu, N., Nallu, M., Spencer, E. C. & Howard, J. A. K. (2003a). *Acta Cryst. E59*, o1009–o1011.
- Vembu, N., Nallu, M., Spencer, E. C. & Howard, J. A. K. (2003b). *Acta Cryst. E59*, o1033–o1035.
- Vembu, N., Nallu, M., Spencer, E. C. & Howard, J. A. K. (2003c). *Acta Cryst. E59*, o1036–o1038.
- Vembu, N., Nallu, M., Spencer, E. C. & Howard, J. A. K. (2003d). *Acta Cryst. E59*, o1213–o1215.
- Vembu, N., Nallu, M., Spencer, E. C. & Howard, J. A. K. (2003e). *Acta Cryst. E59*, o1216–o1219.
- Yachi, K., Sugiyama, Y., Sawada, Y., Iga, T., Ikeda, Y., Toda, G. & Hanano, M. (1989). *Biochim. Biophys. Acta*, **978**, 1–7.