Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N,*N′*-*m*-Phenylenebis(4-nitrobenzenesulfonamide)

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Received 8 December 2000
Accepted 25 January 2001
Online 13 February 2001

In the title compound, $C_{18}H_{14}N_4O_8S_2$, the conformation observed across the two C—S—N—C linkages is synclinal. In the crystal, a network of intermolecular N—H···O hydrogen bonds and weak C—H···O hydrogen bonds is formed.

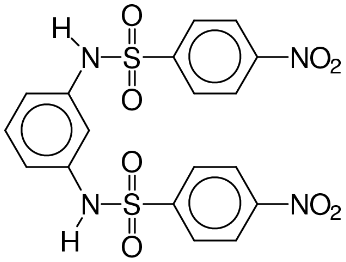
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.100
Data-to-parameter ratio = 15.1

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

Comment

Bis(4-nitrophenylsulfonyl)-1,4-phenylenediamine and its clathrates with cyclopentanone, cyclohexanone, tetrahydrofuran, *N*,*N*-dimethylformamide and pyridine are well documented (Bock *et al.*, 1999). In the structure of bis(4-nitrophenylsulfonyl)-1,3-phenylenediamine, (I), the conformation of the attachment of the 4-nitrophenylsulfonyl substituents to the phenylenediamine moiety is better described by the torsion angles C11—S1—N1—C1 [61.3 (2)°] and C21—S2—N2—C3 [−47.5 (2)°] and they show a (±)-synclinal conformation (Fig. 1). As a result, both the nitrophenyl rings are folded into the same side of the central phenylenediamine moiety, in contrast to the case in bis(4-nitrophenylsulfonyl)-1,4-phenylenediamine (Bock *et al.*, 1999), in which they are arranged on either side to give an extended structure. The distances and angles of (I) are comparable to those observed in the 1,4-phenylenediamine derivative.



(I)

In the crystal, each molecule of (I) takes part in eight hydrogen bonds (four independent H bonds, each as acceptor and donor, see Table 2) to five neighboring molecules with a hydrogen-bonded network as a result (Fig. 2). Both amino groups (N1—H1N and N2—H2N), one sulfonyl (O1—S1—O2) and one nitro (O7—N4—O8) group are involved in the hydrogen bonding. Atom O7 exhibits a bifurcated hydrogen bond.

A weak phenyl–phenyl ring interaction is observed between ring C11–C26 and its inversion related pair at ($-1-x$, $1-y$, $2-z$), with a centroid-to-centroid distance of 3.885 (1) Å. The interacting phenyl rings can be arranged in any one of the

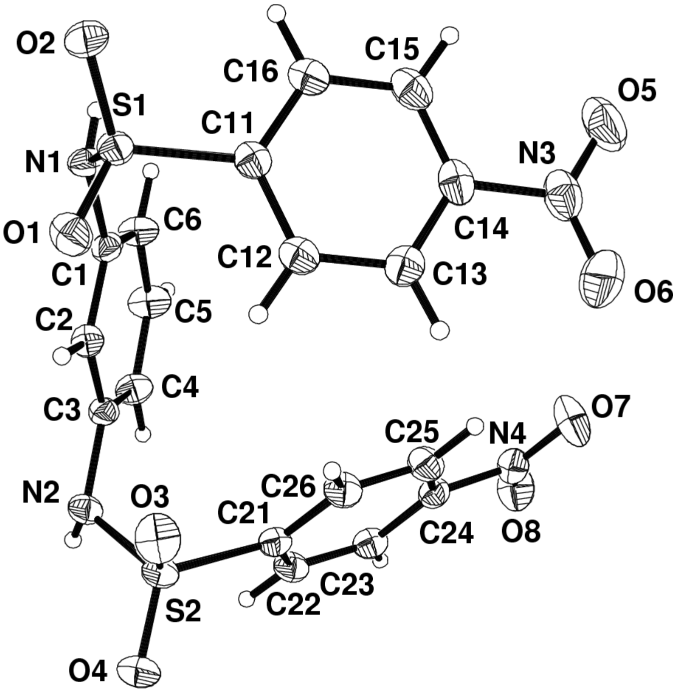


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

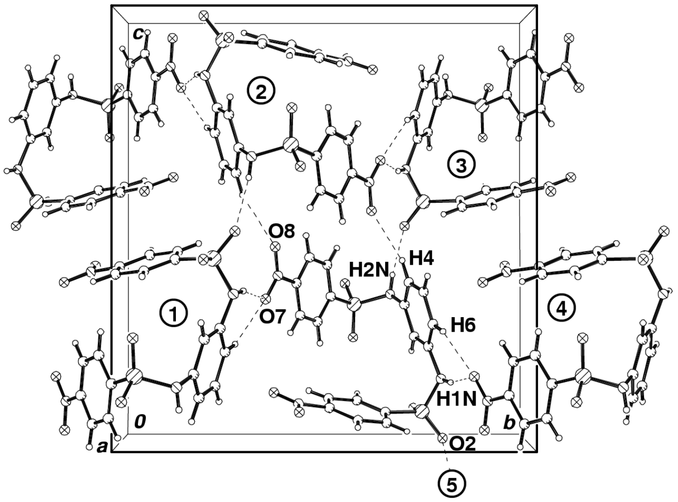


Figure 2
The crystal packing of (I) viewed along the *a* axis. Hydrogen bonds are indicated by dashed lines with selected labeled centers for one molecule interacting to five neighboring units (four are shown).

following ways: (i) parallel-sandwich, (ii) parallel-displaced, (iii) T-shaped and (iv) displaced T-shaped form (Hobza *et al.*, 1994). In this structure, a parallel-displaced geometry is observed for the ring interaction. Using *ab initio* methods, Hobza *et al.* (1994) found that the parallel-displaced structure was more stable in the benzene dimer. For that dimer, they obtained a minimum interaction energy of -9.53 kJ mol^{-1} when the centroid–centroid distance was 3.85 Å and the parallel displacement was 1.6 Å.

Experimental

Bis(4-nitrophenylsulfonyl)-1,3-phenylenediamine was prepared by a modified synthesis (Shirley *et al.*, 1952) as follows: to 1,3-phenylenediamine (3.24 g, 30 mmol), dissolved in 80 ml hot pyridine, at room temperature, a solution of 1,4-nitrobenzensulfonylchloride (13.3 g, 60 mmol) in 200 ml pyridine was added over 45 min with stirring. After 3 h additional stirring, the reaction mixture was poured into a cold mixture of 1350 ml water and 150 ml concentrated hydrochloric acid. The precipitate was washed with water and *n*-hexane. Drying in vacuum yielded dark-red (I)·3pyridine (19.6 g, 87%). Dissolving this in acetone and precipitating with *n*-hexane yielded brown (I)·2pyridine. Red crystals of (I) without pyridine were obtained by vapor diffusion of *n*-hexane into a solution of (I)·2 pyridine in ethanol.

Crystal data

$C_{18}H_{14}N_4O_8S_2$
$M_r = 478.45$
Monoclinic, $P2_1/n$
$a = 6.8331$ (10) Å
$b = 16.4997$ (14) Å
$c = 17.3290$ (13) Å
$\beta = 90.216$ (8)°
$V = 1953.7$ (4) Å^3
$Z = 4$
$D_x = 1.627$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 84 reflections
$\theta = 10.0$–16.5°
$\mu = 0.33$ mm^{-1}
$T = 150$ (2) K
Block, red
0.35 × 0.25 × 0.20 mm

Data collection

Siemens *P*4 four-circle diffractometer
ω scans
4837 measured reflections
4475 independent reflections
3740 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$
$\theta_{\rm max} = 28.0$°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 21$
$l = -22 \rightarrow 22$
4 standard reflections every 100 reflections intensity decay: <5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.100$
$S = 1.05$
4475 reflections
297 parameters
H atoms treated by a mixture of independent and constrained refinement
$w\ 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 1.4125P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.37$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—N1	1.621 (2)	N2—C3	1.431 (2)
S2—N2	1.627 (2)	N3—C14	1.471 (3)
N1—C1	1.431 (2)	N4—C24	1.464 (2)
C11—S1—N1—C1	61.3 (2)	S2—N2—C3—C4	106.8 (2)
C21—S2—N2—C3	−47.5 (2)	N1—S1—C11—C12	−97.7 (2)
S1—N1—C1—C6	−138.9 (2)	N2—S2—C21—C22	−65.6 (2)

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

$D-\mathrm{H}\cdots A$	$D-\mathrm{H}$	$\mathrm{H}\cdots A$	$D\cdots A$	$D-\mathrm{H}\cdots A$
$\mathrm{N2-H2N}\cdots\mathrm{O2}^{\mathrm{i}}$	0.84 (2)	2.14 (3)	2.974 (2)	169 (2)
$\mathrm{N1-H1N}\cdots\mathrm{O7}^{\mathrm{ii}}$	0.81 (2)	2.27 (2)	3.060 (2)	163 (2)
$\mathrm{C6-H6}\cdots\mathrm{O7}^{\mathrm{ii}}$	0.95	2.43	3.253 (2)	145
$\mathrm{C4-H4}\cdots\mathrm{O8}^{\mathrm{iii}}$	0.95	2.49	3.359 (2)	152

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

All H atoms were located from the difference map; the two amino H atoms were freely refined, whereas the others were placed on idealized positions ($\mathrm{C-H_{phenyl}} = 0.95$ Å) and refined using a riding model.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL*97.

This project was supported by the State of Hesse.

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