organic papers

N,N'-(o-Phenylene)dibenzenesulfonamide

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Key indicators

Single-crystal X-ray study

T = 200 K

Mean σ(C–C) = 0.003 Å

R factor = 0.033

wR factor = 0.090

Data-to-parameter ratio = 17.4

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

The N atoms of the title compound, $C_{18}H_{16}N_2O_4S_2$, are not preorganized for metal cation binding. The N—H groups form hydrogen bonds with symmetry-equivalent sulfonamide groups.

Comment

The title compound, (I), was prepared as part of a new investigation of its ability to act as an ion-exchange extractant for Pb[II]. The compound was selected because of its facile synthesis (Amundsen, 1937), its solubility in less polar solvents, and its acidic sulfonamide H atoms (Dauphin & Kergomard, 1961). The structure of (I) has now been determined, in order to provide a basis for comparison with future structures of lead complexes.

(I)
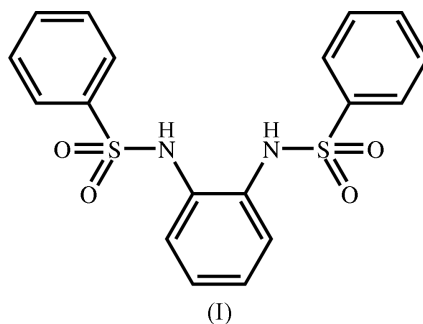
Because much of the molecule of (I) is made up of rigid benzene groups, its conformation can be described by the torsion angles for the six single bonds that connect the arene rings (Table 1). After deprotonation, binding of a metal cation is expected to take place at the two N atoms. It is apparent from Figs. 1 and 2 that some ligand reorganization will be required, as the two N—H vectors are oriented in roughly opposite directions.
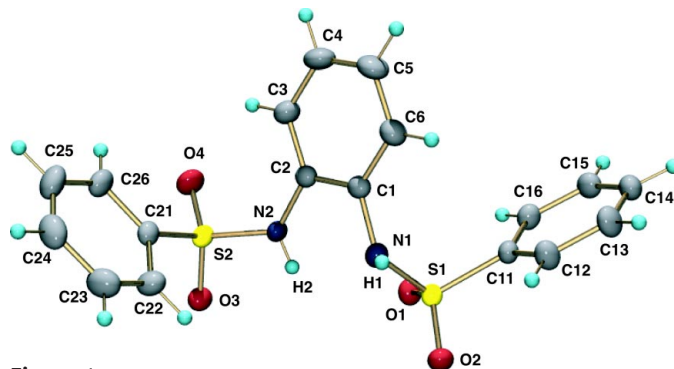

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

The observed conformation of (I) may, at least in part, be due to intermolecular hydrogen bonding (Table 2). As illustrated in Fig. 2, eight-membered rings are formed between symmetry-equivalent sulfonamide groups. Each disulfonamide molecule binds to two other disulfonamide molecules, forming one-dimensional chains. Similar hydrogen-bonding interactions were reported for closely related N,N'-diphenyl-1,3-benzenedisulfonylamide structures (Eagle *et al.*, 2002).

No π-stacking is observed in this structure, but one edge-to-face arene interaction is evident. Geometrical parameters for the best C—H$\cdots\pi$ interaction are given in Table 2.

Experimental

The title compound was prepared as described previously by Amundsen (1937), and was crystallized from dimethylsulfoxide/water (1:2).

Crystal data

$C_{18}H_{16}N_2O_4S_2$	$D_x = 1.480$ Mg m^{-3}
$M_r = 388.4$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25
$a = 9.5311$ (8) Å	reflections
$b = 12.5662$ (10) Å	$\theta = 10.1–16.0°$
$c = 14.9862$ (12) Å	$\mu = 0.33$ mm^{-1}
$\beta = 103.774$ (7)°	$T = 200$ K
$V = 1743.3$ (3) Å^3	Prism, colorless
$Z = 4$	$0.44 \times 0.34 \times 0.29$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 28.0°$
ω scans	$h = -12 \rightarrow 0$
8691 measured reflections	$k = -16 \rightarrow 16$
4198 independent reflections	$l = -19 \rightarrow 19$
3371 reflections with $I > 2\sigma(I)$	3 standard reflections
$R_{int} = 0.036$	frequency: 120 min
	intensity decay: 4%

Refinement

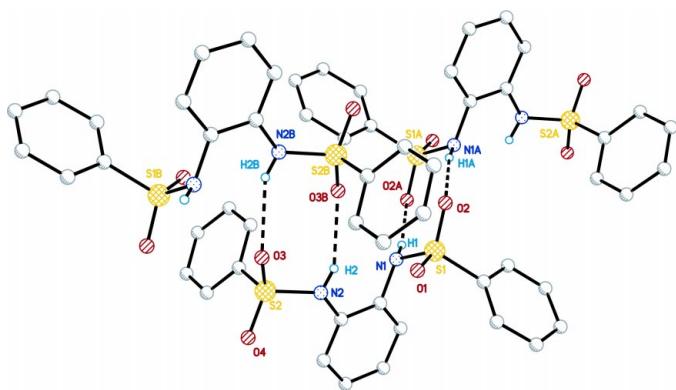|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$+ 0.6951P]$
$wR(F^2) = 0.090$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4198 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
241 parameters	$\Delta\rho_{min} = -0.41$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Figure 2
The hydrogen-bonding interactions (dashed lines) of (I). Arene H atoms have been omitted for clarity. [Symmetry codes: (A) $2 - x, 1 - y, -z$; (B) $1 - x, 1 - y, -z$.]

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4287 (12)	S2—O3	1.4340 (12)
S1—O2	1.4405 (12)	S2—O4	1.4298 (13)
S1—N1	1.6376 (14)	S2—N2	1.6333 (15)
O1—S1—O2	120.19 (7)	O3—S2—N2	103.79 (7)
O1—S1—N1	107.64 (7)	O3—S2—C21	108.59 (7)
O1—S1—C11	107.99 (7)	O4—S2—N2	109.40 (7)
O2—S1—N1	104.86 (7)	O4—S2—C21	108.19 (8)
O2—S1—C11	107.80 (7)	N2—S2—C21	107.21 (7)
N1—S1—C11	107.80 (7)	S1—N1—C1	120.37 (11)
O3—S2—O4	119.07 (8)	S2—N2—C2	126.37 (11)
C11—S1—N1—C1	61.36 (14)	N2—S2—C21—C22	−60.78 (15)
N1—S1—C11—C12	72.33 (14)	S1—N1—C1—C2	91.94 (16)
C21—S2—N2—C2	−65.23 (15)	S2—N2—C2—C1	151.82 (12)

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

$Cg3$ is the centroid of the C21–26 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O2^i	0.79 (2)	2.15 (2)	2.9310 (19)	169 (2)
N2—H2$\cdots$O3^{ii}	0.814 (19)	2.275 (19)	3.0318 (18)	154.8 (19)
C16—H16$\cdots Cg3^{iii}$	0.95	2.73	3.573 (2)	148

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

The sulfonamide H atoms were located in a difference map and their coordinates were refined, although the N—H bond lengths were restrained to be equal to within 0.02 Å. All other H atoms were placed in calculated positions, with C—H distances of 0.95 Å, and refined using a riding model. For all H atoms, $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(N)$.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC Software*; data reduction: *XCAD4* (Harms, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* for Windows (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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