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N,N′-Bis(2-tosylaminobenzylidene)-1,2-ethanediamine

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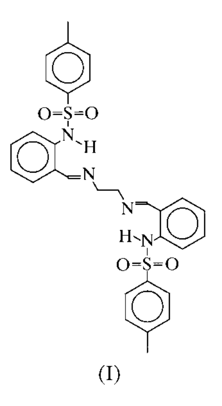
The structure of the title compound, $C_{30}H_{30}N_4O_4S_2$, is highly conditioned in the solid state by two strong N—H···N interactions. The values of the distances between the aminic N atoms [5.865 (3) Å] and the iminic N atoms [2.930 (4) Å], in conjunction with the long distance between the S atoms of the two tosyl groups [7.673 (1) Å], suggest that when the molecule acts as a ligand it will provide a single cavity for N_4 coordination to the metal centre.

Comment

The development of Schiff base ligands has received considerable interest since Zhang *et al.* (1990) and Irie *et al.* (1990) had success in the asymmetric epoxidation of unfunctionalized olefins with Mn^{III} complexes containing salen-type ligands as catalysts. These ligands yield complexes which seem rather promising for other one-O-atom transfer processes (epoxidation of enolates, oxidation of sulfide to sulfoxide, catalase reaction, water photolysis, *etc.*) and other catalytic asymmetric reactions such as azidination of olefins, Diels–Alder cycloaddition, enantioselective cyclopropanation of styrenes and ring opening of epoxides. During our search for new metal catalysts containing salen-type ligands, we have determined the crystal structure of the title compound, (I).



(I)

Previously, some non-planar N_4 Schiff bases similar to (I) have been reported (Mahía, Maestro, Vázquez, Bermejo, Sanmartín & Maneiro, 1999; Bailey *et al.*, 1974; Bailey & McKenzie, 1980). As previously seen for *N,N′*-bis-(2-tosylaminobenzylidene)-1,3-propanediamine (PTS) (Mahía, Maestro, Vázquez, Bermejo, Sanmartín & Maneiro, 1999), the structure of (I) is highly conditioned by two strong N—H···N interactions, N1—H1*A*···N2 and N4—H4*A*···N3 (Fig. 1). Due to these, the atoms H1*A*, N1, C8, C13, C14 and N2, and H4*A*, N4, C23, C18, C17 and N3 form two six-membered rings, with r.m.s. deviations of 0.0296 and 0.0354 Å, respectively. This interaction also involves the opening of the bond angles C8—N1—S1 and C23—N4—S2 to 123.9 (2) and 124.2 (2)°, respectively, close to the expected value for an sp^2 central atom. There are also two weak intermolecular C—H···O interactions, namely C10—H10*A*···O1^{i} and C17—H17*A*···O1^{ii} [symmetry codes: (i) $x - 1, y, z$; (ii) $x + 1, y, z$].

The angle between the least-squares planes of the C8–C13 and C18–C23 rings is 82.76 (8)°, and the value of the C14—N2—C15—C16 torsion angle is −109.1 (3)°. However, the two members of the methylene chain are coplanar with the conjugated system formed by the C18–C23 ring and its imino group, as the C17—N3—C16—C15 torsion angle confirms [179.3 (3)°].

The bond angles C5—S1—N1 and C24—S2—N4 and the torsion angles C8—N1—S1—C5 and C23—N4—S2—C24 indicate that both tosyl groups adopt a similar conformation in the molecule. They appear in opposite positions, probably to minimize the steric repulsion.

Finally, there are some structural differences between the molecules of (I) and PTS. The spatial disposition of the tosyl groups with respect to their benzylidene rings is similar for both ligands, but the conformation of the tosyl groups with

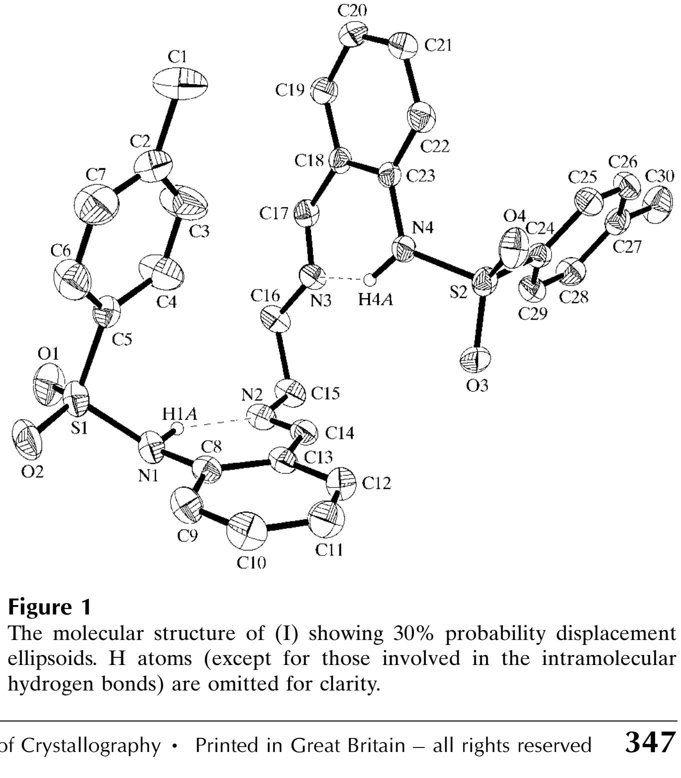


Figure 1
The molecular structure of (I) showing 30% probability displacement ellipsoids. H atoms (except for those involved in the intramolecular hydrogen bonds) are omitted for clarity.

respect to each other is completely different for the two compounds. Both aromatic rings of the tosyl groups are practically perpendicular in PTS, forming an angle of 82.5 (1)°, while the same aromatic rings adopt a semi-parallel conformation in (I) [17.1 (2)°]. The distance between the iminic N atoms N2···N3 is longer in PTS [4.413 (3) Å] than in (I) [2.930 (4) Å]. Additionally, the distance between the aminic N atoms N1···N4 is shorter in (I) [5.865 (3) Å] than in PTS [7.724 (3) Å]. Consequently, the distance between the S atoms is longer in the PTS molecule [9.007 (1), *versus* 7.673 (1) Å in (I)]. These distances suggest that when the molecule acts as a ligand it will do so in a monocompartmental fashion, providing a single cavity for N_4 coordination to the metal centre. Clearly, rotation about the C15—C16 bond to give a near eclipsed or *gauche* conformation would achieve quasi square-planar coordination environments.

Experimental

The title Schiff base was synthesized by condensation of 2-tosylaminobenzaldehyde (Chernova *et al.*, 1971; Mahía, Maestro, Vázquez, Bermejo, González & Maneiro, 1999) and 1,2-ethanediamine. To a solution of 2-tosylaminobenzaldehyde (1 g, 3.58 mmol) in chloroform (150 ml) was added 1,2-ethanediamine (0.12 ml, 1.79 mmol). The mixture was heated to 343 K and stirred for 3 h. The resultant yellow solution was filtered and then concentrated. Yellow crystals of (I) were formed after one night of slow evaporation at room temperature (yield: 0.8 g, 77.7%; b.p. 480–487 K). Elemental analysis for $C_{30}H_{30}N_4O_4S_2$, calculated (found): C 62.72 (62.65), H 5.05 (5.14), N 9.75 (9.59), S 10.76 (10.38)%; mass spectroscopy, M^+, calculated (found): 574.8 (575.3); ^{1}H NMR ($CDCl_3$, p.p.m.): 13.0 (*s*, 2H), 8.5 (*s*, 2H), 7.5–6.7 (*m*, 16H), 4.1 (*s*, 4H), 2.1 (*s*, 6H); IR spectroscopy (KBr, cm^{-1}): ν(C═N) 1635 (*s*), ν(C—N) 1334 (*s*), $\nu_{asy}(SO_2)$ 1284 (*s*), $\nu_{sym}(SO_2)$ 1156 (*s*).

Crystal data

$C_{30}H_{30}N_4O_4S_2$
$M_r = 574.70$
Monoclinic, $C2/c$
$a = 8.5144$ (1) Å
$b = 22.7900$ (3) Å
$c = 29.9046$ (3) Å
$\beta = 96.600$ (1)°
$V = 5764.33$ (12) Å^3
$Z = 8$
$D_x = 1.324$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7195 reflections
$\theta = 3$–28°
$\mu = 0.227$ mm^{-1}
$T = 298$ (2) K
Plate, yellow
0.30 × 0.20 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.715$, $T_{max} = 1.000$
11 944 measured reflections
5078 independent reflections
3207 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 25.03°$
$h = -10 \rightarrow 9$
$k = -26 \rightarrow 26$
$l = -25 \rightarrow 35$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.127$
$S = 1.041$
5078 reflections
365 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 4.8618P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N2—C14	1.268 (4)	C15—C16	1.514 (4)
N3—C17	1.257 (4)		
N1—S1—C5	106.50 (14)	C8—N1—S1	123.9 (2)
N4—S2—C24	106.47 (13)	C23—N4—S2	124.2 (2)
C5—S1—N1—C8	57.8 (3)	C14—N2—C15—C16	−109.1 (3)
C24—S2—N4—C23	−57.5 (3)	C17—N3—C16—C15	179.3 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N2	0.96	1.84	2.705 (3)	147
N4—H4*A*···N3	1.02	1.80	2.681 (3)	142
C10—H10*A*···O1^{i}	0.93	2.50	3.159 (4)	128
C17—H17*A*···O4^{ii}	0.93	2.54	3.394 (3)	153

Symmetry codes: (i) $x - 1, y, z$; (ii) $1 + x, y, z$.

The positions of all H atoms were calculated geometrically and a riding model was used in their refinement, except for those involved in the intramolecular hydrogen bonds (H1*A* and H4*A*), which were found in a difference electron-density map and then refined with their coordinates riding on the corresponding carrier heteroatom.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1378). Services for accessing these data are described at the back of the journal.

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