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2-(Benzylsulfanyl)benzaldehyde azine

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Key indicators

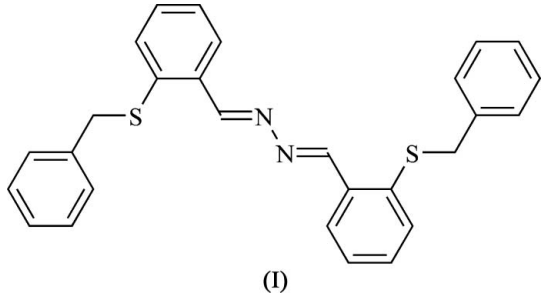
Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.101
Data-to-parameter ratio = 18.8

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

The molecule of the title compound, $C_{28}H_{24}N_2S_2$, has a crystallographically imposed center of symmetry at the mid-point of the N—N bond. It is not planar, due to the steric repulsion between S and H atoms.

Comment

Schiff bases are important ligands in coordination chemistry. There are many examples of imine-based ligands, but fewer examples of azine ligands. Salicylaldehyde azine has been used extensively as an anionic bidentate ligand for the preparation of mononuclear (Trivedi *et al.*, 2004) and binuclear complexes (Pal & Pal, 2001; Singh *et al.*, 2004). Phenyl 2-pyridyl ketone azine has been used as a neutral, multidentate ligand in mononuclear and binuclear complexes (Amadei *et al.*, 1998; Tuna *et al.*, 2003). Our group is interested in the synthesis and coordination chemistry of sulfur-containing Schiff base ligands (Hamaker & Halbach, 2006). As a part of our ongoing studies, we have synthesized some sulfur-containing ligands for the preparation of binuclear complexes. Here we report the crystal structure of the title compound, (I).



(I)

The molecule of (I) (Fig. 1) possesses a crystallographically imposed center of symmetry at the mid-point of the N—N bond, as is observed in many symmetric azines with an *E*,*E* configuration (Mijanuddin *et al.*, 2004; Zheng *et al.*, 2005; Wang *et al.*, 2005). The bond lengths and angles (Table 1) are typical of other azines (Mijanuddin *et al.*, 2004; Zheng *et al.*, 2005; Wang *et al.*, 2005) and thioether Schiff bases (Hamaker *et al.*, 2006). The core of the molecule deviates slightly from planarity due to the steric repulsion between atoms S and H16(C16), with a C14—C15—C16—N torsion angle of 12.7 (2)°.

Experimental

2-(Benzylsulfanyl)benzaldehyde (1.078 g, 4.46 mmol) was added to a solution of hydrazine monohydrate (2.05 mmol, 100 ml) in ethanol (20 ml) at 273 K. The reaction mixture was refluxed for 3 h and

cooled to 233 K. The solid was collected by suction filtration and washed with cold ethanol (yield 0.545 g, 58.7%; m.p. 384–386 K). Crystals were obtained by slow evaporation of a dichloromethane solution. ^{1}H NMR (CD_2Cl_2, 400 MHz): δ 9.04 (*s*, 2H, N═CH), 8.08 (*dd*, 2H, aromatic), 7.45 (*dd*, 2H, aromatic), 7.33 (*m*, 6H, aromatic), 7.24 (*m*, 8H, aromatic), 4.07 (*s*, 4H, SCH_2Ph). IR (KBr, ν, cm^{-1}): 1614 (C═N).

Crystal data

$C_{28}H_{24}N_2S_2$
M_r = 452.61
Triclinic, $P\overline{1}$
a = 7.5712 (5) Å
b = 7.8467 (6) Å
c = 11.3069 (10) Å
α = 91.882 (8)°
β = 92.124 (7)°
γ = 117.234 (6)°
V = 595.92 (9) Å^3
Z = 1
D_x = 1.261 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 297 (2) K
Block, yellow
0.59 × 0.41 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
non–profiled $\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.* (1968)'
T_{min} = 0.907, T_{max} = 0.938
2873 measured reflections
2733 independent reflections
2134 reflections with $I > 2\sigma(I)$
R_{int} = 0.008
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.101
S = 1.05
2733 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.0834P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C16—N	1.2662 (18)	N—N^i	1.416 (2)
N—C16—C15	121.71 (12)	C16—N—N^i	112.24 (14)

Symmetry code: (i) $-x+1, -y+1, -z$.

H atoms were positioned geometrically, with C—H = 0.93 and 0.97 Å for aromatic and methylene H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

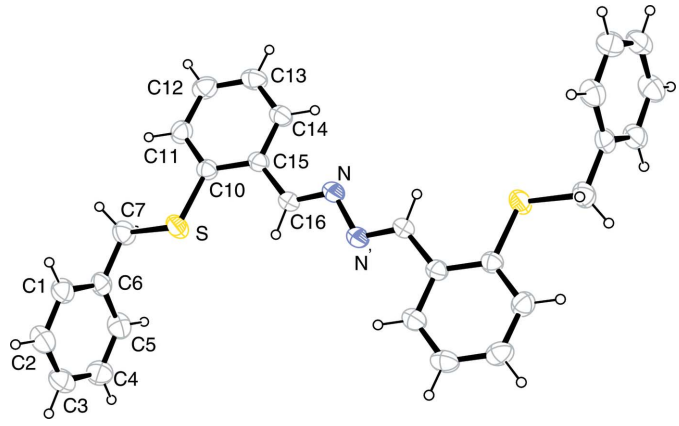


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of the title molecular structure with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabeled atoms are related to labeled atoms by $1-x, 1-y, -z$.

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References

Amadei, E., Carcelli, M., Ianelli, S., Cozzini, P., Pelagatti, P. & Pelizzi, C. (1998). *J. Chem. Soc. Dalton Trans.* pp. 1025–1029.
Beurskens, P. T., Beurskens, G., de Gelder, R., Garciía-Granda, S., Gould, R. O., Israel, R. & Smits, J. M. M. (1999). *The DIRDIF99 Program System.* Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
Enraf–Nonius (1994). *CAD-4 EXPRESS Software*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hamaker, C. G. & Halbach, D. P. (2006). *Inorg. Chim. Acta*, **359**, 846–852.
Hamaker, C. G., Maryashina, O. & Halbach, D. P. (2006). *Acta Cryst.* E**62**, o802–o803.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Mijanuddin, M., Sheldrick, W. S., Mayer-Figge, H., Ali, M. & Chattopadhyay, N. (2004). *J. Mol. Struct.* **693**, 161–165.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Pal, S. & Pal, S. (2001). *Inorg. Chem.* **40**, 4807–4810.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Singh, S. K., Chandra, M. & Pandey, D. S. (2004). *J. Organomet. Chem.* **689**, 2073–2079.
Trivedi, M., Chandra, M., Pandey, D. S., Puerta, M. C. & Valerga, P. (2004). *J. Organomet. Chem.* **689**, 879–882.
Tuna, F., Clarkson, G., Alcock, N. W. & Hannon, M. J. (2003). *Dalton Trans.* pp. 2149–2155.
Wang, Q.-H., Guo, G.-C. & Wang, M.-S. (2005). *Acta Cryst.* E**61**, o1156–o1157.
Zheng, P.-W., Wang, W. & Duan, X.-M. (2005). *Acta Cryst.* E**61**, o3020–o3021.