Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N-[2-(Benzylsulfanyl)benzylidene]-2-(methylsulfanyl)aniline

Christopher G. Hamaker,* Oksana Maryashina and Dominic P. Halbach

Department of Chemistry, Illinois State University, Campus Box 4160, Normal, IL 61790-4160 USA

Correspondence e-mail: chamake@ilstu.edu

Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.149
Data-to-parameter ratio = 19.1

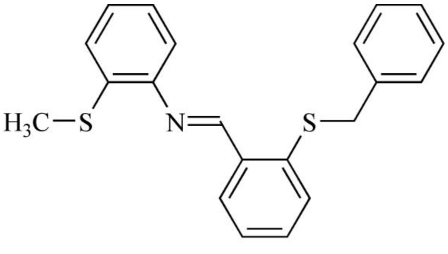
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 19 January 2006
Accepted 23 January 2006

The title molecule, $C_{21}H_{19}NS_2$, is non-planar with a dihedral angle between the two benzene rings bonded to the N=CH group of 51.33 (8)°.

Comment

Schiff base ligands have played an important role in the development of coordination chemistry due to their ease of preparation (Che & Huang, 2003). Schiff base ligands are also readily modified, both sterically and electronically. Our group is interested in the synthesis and utility of sulfur-containing Schiff base ligands (Hamaker & Halbach, 2006; Hamaker & Corgliano, 2006). As part of our ongoing studies, we report the synthesis and crystal structure of the title compound, (I).



(I)

The ArN=CHAr moiety in (I) (Fig. 1) is non-planar, with a dihedral angle of 51.33 (8)° between the arene rings. The N=CH group is nearly coplanar with the C11–C16 arene ring, with an N—C10—C11—C16 torsion angle of 172.4 (2)°. The C2–C7 arene ring is twisted away from coplanarity to relieve the steric repulsion between H12 and the SCH_3 group, with a C10—N—C7—C2 torsion angle of 140.7 (2)°. The N=C10 double-bond length is 1.274 (3) Å, similar to that in related molecules (Hamaker & Corgliano, 2006; Ainscough *et al.*, 2000; Özbey *et al.*, 1998).

In the crystal structure, the molecules stack along the *c* axis; the crystal packing is shown in Fig. 2.

Experimental

To a solution of 2-(methylsulfanyl)aniline (0.960 g, 6.90 mmol) in absolute ethanol (50 ml) in a 100 ml round-bottomed flask equipped with a reflux condensor was added 2-(benzylsulfanyl)benzaldehyde (1.529 g, 6.70 mmol). The mixture was heated under reflux for 48 h, cooled to room temperature and placed in a freezer at 233 K for 14 h. The reaction product was filtered off, washed with cold ethanol and dried *in vacuo* to yield 1.490 g (63.7%) of a yellow solid. Crystals (m.p. 267–269 K) were obtained by diffusion of heptane into a 1,2-dichloroethane solution of (I). ^1H NMR ($CDCl_3$, 400 MHz): δ 8.95 (*s*, 1H, N=CH), 8.30 (*m*, 1H, aromatic), 7.49 (*m*, 1H, aromatic), 7.41 (*m*,

doi:10.1107/S1600536806002819

2H, aromatic), 7.27 (*m*, 8H, aromatic), 6.93 (*d*, 1H, aromatic), 4.13 (*s*, 2H, SCH_2Ph), 2.51 (*s*, 3H, SCH_3). IR (Nujol, ν, cm^{-1}): 1602 (C═N). Analysis calculated (found) for $C_{21}H_{19}NS_2$: C 72.16 (72.24), H 5.48 (5.46), N 4.01% (4.00%).

Crystal data

$C_{21}H_{19}NS_2$
$M_r = 349.49$
Monoclinic, $P2_1/c$
$a = 9.7960$ (7) Å
$b = 17.1416$ (16) Å
$c = 11.2536$ (14) Å
$\beta = 106.699$ (8)°
$V = 1810.0$ (3) $Å^3$
$Z = 4$

$D_x = 1.283$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 5.6$–13.7°
$\mu = 0.30$ mm^{-1}
$T = 297$ (2) K
Plate, yellow
0.50 × 0.50 × 0.23 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
non–profiled $\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.784$, $T_{max} = 0.931$
4362 measured reflections

4154 independent reflections
2629 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.5°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.149$
$S = 1.10$
4154 reflections
217 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0823P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e $Å^{-3}$
$\Delta\rho_{min} = -0.32$ e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

N—C10	1.274 (3)	N—C7	1.420 (3)
C16—S2—C20	104.34 (11)	C10—N—C7	118.00 (19)
C2—S1—C1	102.87 (13)	N—C10—C11	122.8 (2)

H atoms were treated as riding, with C—H distances of 0.93–0.97 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$ for the aromatic and methylene H atoms and $U_{iso}(H) = 1.5U_{eq}(C)$ for the methyl H atoms.

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).

CGH thanks Illinois State University for partial financial support.

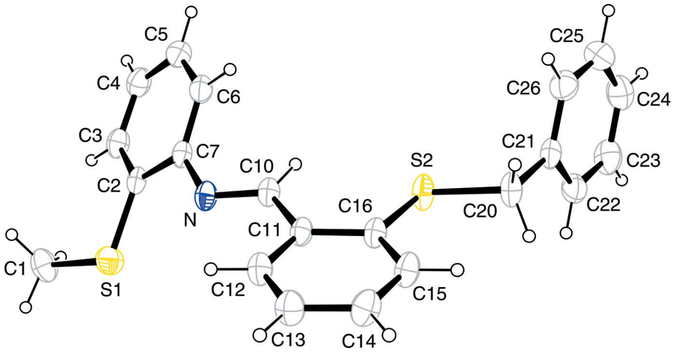


Figure 1
View of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

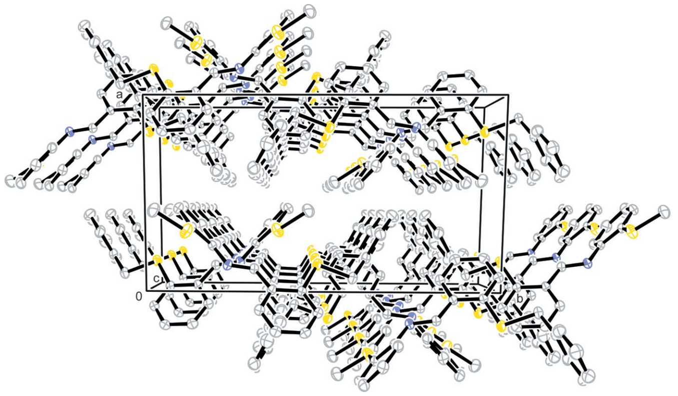


Figure 2
The crystal packing of (I), viewed along the *c* axis. H atoms have been omitted for clarity.

References

Ainscough, E. W., Brodie, A. M., Burrell, A. K., Xiaohong, F., Halstead, M. J. R., Kennedy, S. M. F. & Waters, J. M. (2000). *Polyhedron*, **19**, 2585–2592.
Beurskens, P. T., Beurskens, G., de Gelder, R., García-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.
Che, C.-M. & Huang, J.-S. (2003). *Coord. Chem. Rev.* **242**, 97–113.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. 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. & Corgliano, D. M. (2006). *Acta Cryst.* E**62**, o68–o69.
Hamaker, C. G. & Halbach, D. P. (2006). *Inorg. Chim. Acta*, **359**, 846–852.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Özbey, S., Temel, A., Ancin, N., Öztas, S. G. & Tüzün, M. (1998). *Z. Kristallogr. New Cryst. Struct.* **213**, 207–208.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.