

C24	-0.3665 (2)	-0.1059 (2)	-0.0931 (4)	0.0673 (9)
C25	-0.3621 (2)	-0.2250 (2)	-0.2097 (3)	0.0683 (10)
O1W	0.42272 (15)	0.17651 (14)	0.5381 (2)	0.0772 (7)

Table 2. Selected geometric parameters (Å, °)

N1—C6	1.331 (3)	C12—C13	1.482 (3)
N1—C2	1.352 (3)	C13—N14	1.295 (3)
C2—C3	1.377 (4)	N14—N15	1.401 (3)
C3—C4	1.387 (4)	N15—C16	1.295 (3)
C4—C5	1.388 (3)	C16—C17	1.448 (4)
C5—C6	1.411 (3)	C17—C18	1.387 (3)
C5—C13	1.460 (3)	C17—C22	1.401 (3)
C6—C7	1.476 (3)	C18—C19	1.366 (4)
C7—N8	1.349 (3)	C19—C20	1.415 (3)
C7—C12	1.393 (3)	C20—N23	1.368 (3)
N8—C9	1.346 (3)	C20—C21	1.402 (3)
C9—C10	1.372 (4)	C21—C22	1.371 (4)
C10—C11	1.387 (4)	N23—C24	1.442 (4)
C11—C12	1.370 (3)	N23—C25	1.451 (4)
C6—N1—C2	114.6 (2)	C11—C12—C7	119.8 (2)
N1—C2—C3	124.6 (3)	C11—C12—C13	131.9 (2)
C2—C3—C4	120.0 (3)	C7—C12—C13	108.3 (2)
C3—C4—C5	117.2 (2)	N14—C13—C5	133.9 (2)
C4—C5—C6	118.2 (2)	N14—C13—C12	119.6 (2)
C4—C5—C13	133.2 (2)	C5—C13—C12	106.4 (2)
C6—C5—C13	108.6 (2)	C13—N14—N15	113.7 (2)
N1—C6—C5	125.4 (2)	C16—N15—N14	110.0 (2)
N1—C6—C7	126.7 (2)	N15—C16—C17	122.7 (3)
C5—C6—C7	107.9 (2)	C18—C17—C16	123.4 (2)
N8—C7—C12	124.5 (2)	C22—C17—C16	119.3 (2)
N8—C7—C6	126.8 (2)	N23—C20—C21	121.8 (2)
C12—C7—C6	108.7 (2)	N23—C20—C19	121.4 (2)
C9—N8—C7	113.9 (2)	C20—N23—C24	121.2 (2)
N8—C9—C10	125.4 (3)	C20—N23—C25	121.2 (3)
C9—C10—C11	119.6 (3)	C24—N23—C25	117.2 (3)
C12—C11—C10	116.9 (3)		

All the H atoms were located from difference Fourier maps and refined isotropically.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976), SHELXTL/PC (Sheldrick, 1990). Software used to prepare material for publication: SHELXL93. Geometric calculations: PARST (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1177). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Benzyl 3-(4-Dimethylaminobenzylidene)-dithiocarbazate

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Abstract

The title Schiff base compound, C₁₇H₁₉N₃S₂, crystallizes in the triclinic system with two molecules in the asymmetric unit. The dimethylaminophenyl and carbazate parts of the molecule lie in one plane, perpendicular to the plane of the *S*-benzyl group. One of the two molecules in the asymmetric unit displays lateral disorder of the *S*-benzyl group. The molecular packing is

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governed by N—H···S hydrogen bonds leading to the formation of dimeric pairs of the same type, *i.e.* pairs of either normal or disordered molecules.

Comment

In recent years, there has been considerable interest in the chemistry of Schiff base compounds containing N and S donors. The increased activity in this field may be because of their medicinal properties and their chelating ability with transition metal ions (Ali & Tarafdar, 1977; Ali & Bose, 1984; Davies, El-Sayed, El-Toukhy & Henary, 1990; Xu, Alyea, Ferguson & Jennings, 1991). In this paper, we report the structure of a new Schiff base ligand derived from benzyl dithiocarbazate, namely, benzyl 3-(4-dimethylaminobenzylidene)-dithiocarbazate, (I).

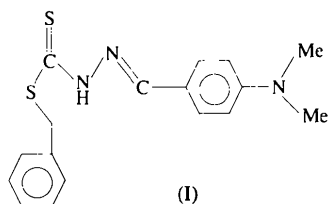


Fig. 1 shows an *ORTEP* (Johnson, 1965) plot of molecule 1 and a perspective view of molecule 2, with the respective numbering schemes. Of the two molecules in the asymmetric unit, molecule 1 is normal, whereas molecule 2 is disordered. Bond lengths and angles in these molecules are generally normal. The N1—C1 single-bond distance is shorter than usual; 1.324 (3) for molecule 1 and 1.332 (4) Å for 2. There are some differences in the C1—S2 and S2—C11 bond lengths of the disordered portion of molecule 2 (Table 2). The dimethylaminophenyl and dithiocarbazate portions of the molecule lie in one plane, due to the conjugated nature of the system. The *S*-benzyl group, however, is twisted at right angles relative to this plane. The dihedral angles between the phenyl, amino, carbazate and *S*-benzyl planes are given in Table 3.

In the crystal lattice the normal and disordered molecules exist as dimers of the same type, linked by N—H···S hydrogen bonds [H1···S1ⁱ 2.60 (2), N1···S1ⁱ 3.411 (2) Å, N1—H1···S1ⁱ 157 (2)° for molecule 1; N1A—S1Aⁱⁱ 2.56 (4), H1A···S1Aⁱⁱ 3.396 (4) Å, N1A—H1A···S1Aⁱⁱ 166 (3)° for molecule 2; symmetry codes: (i) 1 - x, -y, -z; (ii) 1 - x, 1 - y, 2 - z]. The molecules connected by these hydrogen bonds are related by a centre of symmetry. It is also observed that the molecules are stacked as (222) planar layers containing alternating normal and disordered molecules, the dihedral angle between the planes being 12.6°. An interesting feature is that the molecular orientations in these two planes are rotated by almost 90° with respect to each other, as can be seen in Fig. 2. The inversion centres of the two

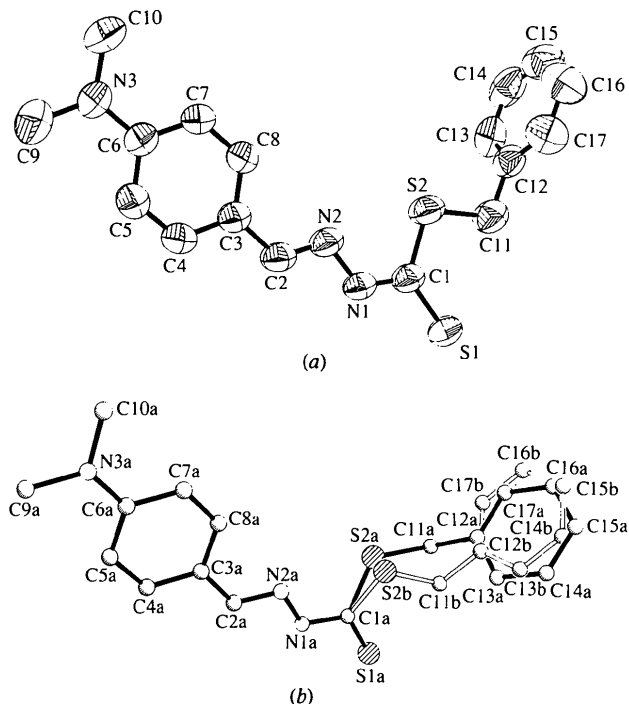


Fig. 1. (a) *ORTEP*II (Johnson, 1976) plot of molecule 1, shown with 50% displacement ellipsoids, and (b) a perspective view of molecule 2, showing the disorder.

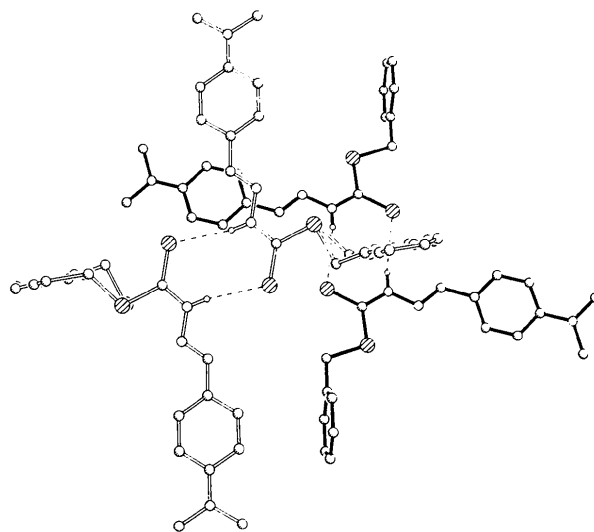


Fig. 2. Plot showing a projection of two layers of molecules. The disordered molecules are shown with open bonds and N—H···S hydrogen bonds are shown as dashed lines.

groups in Fig. 2 lie on the *b* axis, but with respect to the planar stacking, these inversion centres are related by a translation, which leads to the positioning of the *S*-benzyl group of molecule 2 sandwiched between inversion centres of molecule 1. Hence, we attribute the

disorder in the *S*-benzyl group of molecule 2 to the N—H···S hydrogen bonds surrounding it. It may also be noted that the disorder in the *S*-benzyl group is only lateral, parallel to its own plane and perpendicular to the plane formed by the N, H and S atoms which are involved in the hydrogen bonds. Since the *S*-benzyl group of molecule 1 is well away from the N and S atoms of molecule 2, it does not undergo any disorder. Among the two disordered *S*-benzyl sets, group S2B to C17B has almost the same conformation as that of the normal molecule 1, whereas its counterpart, S2A to C17A, deviates to a greater extent (relevant torsion angles are given in Table 2).

Experimental

The title compound was prepared by refluxing benzyl dithiocarbamate and dimethylaminobenzaldehyde in a 1:1 molar ratio in methanol. The product was a yellow crystalline powder. Single crystals suitable for X-ray diffraction analysis were grown from benzene at room temperature.

Crystal data

C₁₇H₁₉N₃S₂ $M_r = 329.47$

Triclinic

 $P\bar{1}$ $a = 10.431(2) \text{ \AA}$ $b = 13.477(2) \text{ \AA}$ $c = 15.016(4) \text{ \AA}$ $\alpha = 109.56(2)^\circ$ $\beta = 101.56(2)^\circ$ $\gamma = 109.14(1)^\circ$ $V = 1761.4(6) \text{ \AA}^3$ $Z = 4$ $D_x = 1.242 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation $\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

 $\theta = 8\text{--}20^\circ$ $\mu = 0.302 \text{ mm}^{-1}$ $T = 293(2) \text{ K}$

Prism

 $0.48 \times 0.46 \times 0.32 \text{ mm}$

Pale yellow

Data collection

Siemens P4 diffractometer

 $\theta/2\theta$ scans

Absorption correction:

none

6297 measured reflections

5284 independent reflections

3765 observed reflections

 $[I > 2\sigma(I)]$ $R_{\text{int}} = 0.0140$ $\theta_{\text{max}} = 24^\circ$ $h = -1 \rightarrow 11$ $k = -13 \rightarrow 13$ $l = -17 \rightarrow 17$

3 standard reflections

monitored every 100

reflections

intensity decay: < 4%

Refinement

Refinement on F^2 $R(F) = 0.0464$ $wR(F^2) = 0.1382$ $S = 1.049$

5284 reflections

446 parameters

Only H-atom U 's refined $w = 1/[\sigma^2(F_o^2) + (0.0729P)^2 + 0.2318P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.323 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.378 \text{ e \AA}^{-3}$

Atomic scattering factors

from *International Tables*for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
S1	0.45849 (9)	0.13434 (8)	0.10013 (5)	0.0860 (3)
N1	0.6102 (2)	0.0281 (2)	0.1481 (2)	0.0696 (6)
N2	0.7086 (2)	0.0112 (2)	0.21178 (15)	0.0668 (6)
N3	1.0959 (3)	-0.2328 (2)	0.3579 (2)	0.0859 (7)
C1	0.5805 (3)	0.1195 (2)	0.1793 (2)	0.0653 (7)
C2	0.7239 (3)	-0.0812 (2)	0.1692 (2)	0.0675 (7)
C3	0.8200 (3)	-0.1175 (2)	0.2202 (2)	0.0609 (6)
C4	0.8374 (3)	-0.2143 (2)	0.1651 (2)	0.0735 (7)
C5	0.9278 (3)	-0.2525 (2)	0.2092 (2)	0.0756 (8)
C6	1.0048 (3)	-0.1958 (2)	0.3125 (2)	0.0654 (7)
C7	0.9864 (3)	-0.0981 (2)	0.3684 (2)	0.0676 (7)
C8	0.8970 (3)	-0.0608 (2)	0.3231 (2)	0.0655 (7)
C9	1.1074 (4)	-0.3371 (3)	0.2998 (3)	0.1114 (12)
C10	1.1600 (4)	-0.1823 (3)	0.4662 (2)	0.0962 (10)
S2	0.67764 (8)	0.21738 (6)	0.30542 (5)	0.0772 (2)
C11	0.6108 (4)	0.3286 (3)	0.3209 (2)	0.0918 (9)
C12	0.6910 (3)	0.4214 (2)	0.4268 (2)	0.0728 (7)
C13	0.8261 (4)	0.5084 (3)	0.4548 (3)	0.0958 (10)
C14	0.8979 (4)	0.5926 (3)	0.5555 (4)	0.1112 (13)
C15	0.8345 (6)	0.5878 (4)	0.6245 (3)	0.1150 (14)
C16	0.7033 (6)	0.5048 (4)	0.5982 (3)	0.1201 (13)
C17	0.6327 (4)	0.4226 (3)	0.5003 (3)	0.0991 (10)
S1A	0.62739 (13)	0.44610 (8)	1.09140 (7)	0.1125 (4)
N1A	0.5321 (3)	0.3690 (2)	0.8953 (2)	0.0863 (7)
N2A	0.5211 (3)	0.3051 (2)	0.7983 (2)	0.0806 (7)
N3A	0.2900 (3)	0.0614 (2)	0.3121 (2)	0.0878 (7)
C1A	0.6157 (3)	0.3689 (3)	0.9750 (2)	0.0865 (9)
C2A	0.4311 (3)	0.3099 (2)	0.7301 (2)	0.0774 (8)
C3A	0.3995 (3)	0.2465 (2)	0.6236 (2)	0.0693 (7)
C4A	0.3020 (3)	0.2582 (3)	0.5540 (2)	0.0760 (7)
C5A	0.2658 (3)	0.1987 (3)	0.4527 (2)	0.0768 (8)
C6A	0.3245 (3)	0.1219 (2)	0.4137 (2)	0.0693 (7)
C7A	0.4228 (3)	0.1099 (2)	0.4837 (2)	0.0764 (8)
C8A	0.4587 (3)	0.1711 (2)	0.5849 (2)	0.0763 (8)
C9A	0.1878 (4)	0.0747 (4)	0.2411 (2)	0.1093 (12)
C10A	0.3329 (4)	-0.0309 (3)	0.2730 (2)	0.1045 (11)
S2A	0.6822 (3)	0.2626 (3)	0.9388 (4)	0.1076 (14)
C11A	0.7795 (7)	0.2535 (8)	1.0510 (6)	0.097 (2)
C12A	0.9309 (6)	0.2756 (9)	1.0649 (8)	0.061 (3)
C13A	1.0263 (12)	0.3866 (8)	1.0841 (8)	0.103 (3)
C14A	1.1731 (10)	0.4148 (9)	1.1047 (7)	0.161 (7)
C15A	1.2244 (6)	0.3322 (14)	1.1062 (10)	0.169 (6)
C16A	1.1290 (12)	0.2212 (12)	1.0870 (9)	0.147 (4)
C17A	0.9822 (10)	0.1929 (7)	1.0664 (6)	0.098 (3)
S2B	0.7342 (3)	0.3066 (3)	0.9481 (3)	0.0943 (10)
C11B	0.8305 (8)	0.3472 (7)	1.0789 (4)	0.096 (2)
C12B	0.9471 (7)	0.3089 (9)	1.0779 (9)	0.067 (3)
C13B	1.0876 (10)	0.3844 (7)	1.0962 (8)	0.095 (3)
C14B	1.1959 (6)	0.3456 (9)	1.0994 (10)	0.146 (5)
C15B	1.1636 (10)	0.2313 (11)	1.0843 (10)	0.144 (4)
C16B	1.0231 (12)	0.1559 (7)	1.0661 (6)	0.125 (4)
C17B	0.9149 (7)	0.1946 (8)	1.0629 (6)	0.094 (3)

Table 2. Selected geometric parameters (\AA , $^\circ$)

S1—C1	1.671 (3)	N1A—N2A	1.381 (3)
N1—C1	1.324 (3)	N2A—C2A	1.277 (4)
N1—N2	1.389 (3)	N3A—C6A	1.370 (4)
N2—C2	1.274 (3)	N3A—C10A	1.440 (4)
N3—C6	1.377 (3)	N3A—C9A	1.451 (4)
N3—C10	1.441 (4)	C1A—S2B	1.746 (4)
N3—C9	1.443 (4)	C1A—S2A	1.763 (4)
C1—S2	1.742 (3)	C2A—C3A	1.444 (4)
C2—C3	1.448 (4)	S2A—C11A	1.850 (8)
S2—C11	1.817 (3)	C11A—C12A	1.465 (8)
C11—C12	1.498 (4)	S2B—C11B	1.814 (7)
S1A—C1A	1.664 (3)	C11B—C12B	1.470 (7)
N1A—C1A	1.332 (4)		

N2—N1—C1—S2	1.3 (3)
N1—C1—S2—C11	179.9 (2)
S1—C1—S2—C11	-0.1 (2)
C1—S2—C11—C12	-178.0 (2)
S2—C11—C12—C13	82.4 (3)
S2—C11—C12—C17	-97.1 (3)
N2A—N1A—C1A—S2A	-9.4 (4)
N1A—C1A—S2A—C11A	-173.4 (3)
S1A—C1A—S2A—C11A	-3.5 (4)
C1A—S2A—C11A—C12A	-121.6 (7)
S2A—C11A—C12A—C13A	64.9 (8)
S2A—C11A—C12A—C17A	-119.6 (8)
N2A—N1A—C1A—S2B	12.1 (4)
N1A—C1A—S2B—C11B	172.8 (4)
S1A—C1A—S2B—C11B	5.1 (4)
C1A—S2B—C11B—C12B	-176.8 (6)
S2B—C11B—C12B—C13B	97.1 (7)
S2B—C11B—C12B—C17B	-86.0 (8)

Table 3. Dihedral angles ($^{\circ}$) between the planes of various molecular groups

	Molecule 1	Molecule 1A	Molecule 2B
Phenyl/amino	9.8 (2)	10.1 (3)	
Phenyl/carbazate	6.9 (1)	5.1 (1)	8.9 (1)
Phenyl/S-benzyl	82.2 (1)	101.6 (3)	100.4 (3)
Carbazate/S-benzyl	83.5 (1)	98.8 (3)	96.7 (2)

The structure of molecule 1 was solved by direct methods. For the second molecule, the complete structure was not solved by direct methods, due to the disorder, but was completed by locating the missing atoms from successive Fourier maps. Initially, the disorder in molecule 2 led to non-convergence of refinement and unreliable bond lengths and angles. Subsequently, the *S*-benzyl group was split into two sets of atoms and their occupancies were refined at the beginning and fixed at 0.5 (the refined value), resulting in satisfactory convergence and geometric parameters. During refinement, the phenyl rings of both the disordered components of the *S*-benzyl group were restrained as regular hexagons with C—C distances of 1.39 Å. All H atoms were fixed geometrically and not refined, but were allowed to ride on those atoms to which they are attached with a displacement parameter 1.2 times that of the parent atom.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990a). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976), SHELXTL/PC (Sheldrick, 1990b). Software used to prepare material for publication: SHELXL93. Geometric calculations: PARST (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1178). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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14-Methyl-7,14-dihydrodibenzo[*a,j*]acridine

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Abstract

14-Methyl-7,14-dihydrodibenzo[*a,j*]acridine, C₂₂H₁₇N, has butterfly-like mirror symmetry. The central ring of the acridine moiety adopts a flattened-boat conformation. The crystal structure is stabilized by van der Waals forces.

Comment

In recent years, cavity-shaped azaarenes have invaded the field of molecular recognition because of their unique structural features that help to hold the planar

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