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1,4-Bis(4-dimethylaminobenzyl)-2,3-diaza-1,3-butadiene

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.065
wR factor = 0.167
Data-to-parameter ratio = 16.6

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

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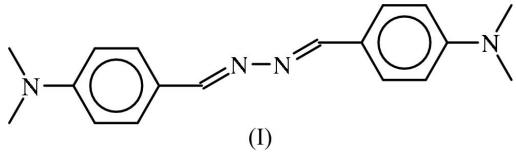
The title Schiff base compound, $C_{18}H_{22}N_4$, is derived from the condensation reaction of hydrazine and 4-(dimethylamino)benzaldehyde. There is a crystallographic centre of symmetry at the mid-point of the N—N bond.

Comment

There has been considerable interest in the study of Schiff base compounds for many years, due to their biological activities (Wetmore *et al.*, 2001; Sattari *et al.*, 1992). In this paper, we report the crystal structure of the title Schiff base compound, (I) (Fig. 1).

There is a crystallographic centre of symmetry at the mid-point of the N2—N2*a* bond [symmetry code: (*a*) $-x$, $-y$, $2-z$] and, as expected, the non-H atoms are nearly coplanar, forming an extended conjugated system. The N2—N2*a* bond distance is 1.410 (3) Å. The N2—C9—C6 angle is 123.3 (2)°, indicating the sp^2 hybridization mode of C9.



(I)

Two similar compounds, (*E*,*E*)-*p*-*N*,*N*-dimethylaminoacetophenone azine, (II), and 4,4′-bis(dimethylamino)benzophenone azine, (III), have been reported previously (Glaser *et al.*, 1995; Hunig *et al.*, 2000). Compounds (II) and (III) can be described as derivatives of (I) in which the H atom at C9 has been substituted by methyl and *p*-dimethylaminophenyl, respectively. Compounds (II) and (III) also have inversion centres at the mid-point of the N—N bond. As the size of the substituent group increases, the N—N bond distance decreases, the C=N bond lengths increase and the C6—C9—N2 bond angles are reduced [123.3 (2), 116.7 (1) and 115.4 (2)° for compounds (I), (II) and (III), respectively]. The C7—C6—

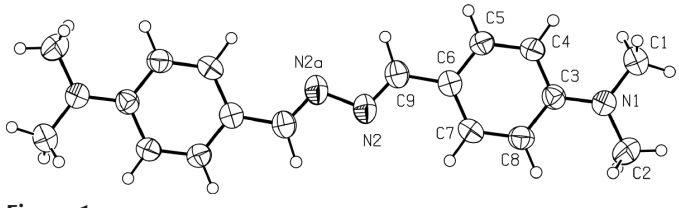


Figure 1
A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by small spheres of arbitrary radii. Unlabelled atoms are related to labelled atoms, and atom N2*a* to atom N2, by the symmetry code ($-x$, $-y$, $2-z$).

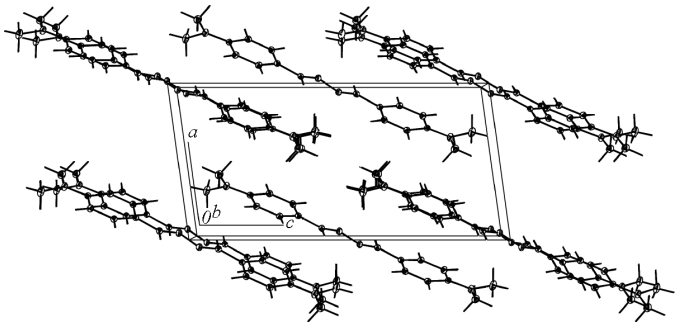


Figure 2
The molecular packing of (I), viewed along the *b* axis.

C9—N2 torsion angle also increases [4.8 (2), 14.2 (1) and 18.1 (2)° for compounds (I), (II) and III, respectively], indicating that the more bulky the substituent group, the greater the deviation from ideal planar geometry.

Experimental

The title compound was prepared by the addition of hydrazine (5 mmol) to a stirred solution of 4-(dimethylamino)benzaldehyde (10 mmol) in ethanol (50 ml). The mixture was stirred at room temperature for 24 h and then filtered; the resultant yellow crystalline solid was washed with ethanol several times and dried. Yellow crystals of (I) were grown by evaporation of a dichloromethane–dimethylformamide solution (yield 1.20 g, 82%). Spectroscopic analyisi: IR (KBr, ν, cm^{-1}): 2909, 2848, 1603, 1521, 1363, 1230, 1178, 809, 518; UV–vis (CH_2Cl_2–EtOH, 1:20): 322 (*sh*), 390 nm. Analysis calculated for $C_{18}H_{22}N_4$: C 73.44, H 7.53, N 19.03; found: C 73.16, H 7.36, N 19.43%.

Crystal data

$C_{18}H_{22}N_4$
M_r = 294.40
Monoclinic, $P2_1/c$
a = 8.232 (4) Å
b = 6.065 (3) Å
c = 16.710 (9) Å
β = 97.864 (6)°
V = 826.4 (7) Å^3
Z = 2
D_x = 1.183 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1199 reflections
θ = 2.5–27.5°
μ = 0.07 mm^{-1}
T = 292.2 K
Block, yellow
0.62 × 0.45 × 0.40 mm

Data collection

Rigaku Mercury CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SPHERE* in *CrystalClear*; Rigaku, 2002)
T_{min} = 0.910, T_{max} = 0.980
4511 measured reflections
1794 independent reflections
1192 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -7 \rightarrow 4$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.167
S = 1.10
1794 reflections
108 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 0.1316P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.13 (2)

Table 1
Selected geometric parameters (Å, °).

N1—C3	1.369 (3)	N2—C9	1.282 (3)
N1—C2	1.435 (3)	N2—N2^{i}	1.410 (3)
N1—C1	1.450 (3)	C6—C9	1.452 (3)
C3—N1—C2	122.27 (18)	C9—N2—N2^{i}	112.0 (2)
C3—N1—C1	120.58 (18)	N1—C3—C4	121.31 (18)
C2—N1—C1	116.68 (18)	N2—C9—C6	123.3 (2)

Symmetry code: (i) $-x, -y, 2-z$.

All methyl H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms [C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C)]; each group was allowed to rotate freely about its C—C bond. The other H atoms were positioned theoretically and refined in riding mode [U_{iso}(H) = 1.2U_{eq}(C)]; the C—H distances were allowed to refine.

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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