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N,N'-Bis[4-(dimethylamino)benzylidene]-butane-1,4-diamine

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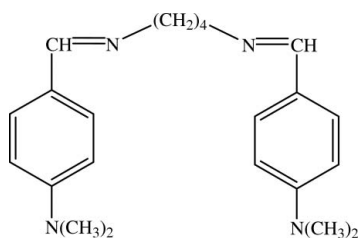
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.050; wR factor = 0.148; data-to-parameter ratio = 24.0.

The molecule of the title compound, $\text{C}_{22}\text{H}_{30}\text{N}_4$, lies across a crystallographic inversion centre and adopts an *E* configuration with respect to the azomethine $\text{C}=\text{N}$ bond. The molecular structure has a staggered conformation, with the two benzene rings parallel to each other. The dihedral angle between the benzylidene and butane groups is $77.77(7)^\circ$. In the crystal structure, molecules are arranged into columns along the *a* axis by $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For the values of bond lengths, see Allen *et al.* (1987). For related structures, see, for example, Bahron *et al.* (2007); Kassim *et al.* (2006). For information on Schiff base complexes and their pharmacological activities, see, for example, Adsule *et al.* (2006); Amirnasr *et al.* (2002); Habibi *et al.* (2007); Hou *et al.* (2001); Pal *et al.* (2005); Ren *et al.* (2002).



Experimental

Crystal data

$\text{C}_{22}\text{H}_{30}\text{N}_4$ $a = 5.2254(1)$ Å
 $M_r = 350.50$ $b = 9.5742(3)$ Å
 Monoclinic, $P2_1/c$ $c = 19.4882(5)$ Å

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$\beta = 95.512(1)^\circ$
 $V = 970.47(4)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.07$ mm⁻¹
 $T = 100.0(1)$ K
 $0.57 \times 0.31 \times 0.19$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.960$, $T_{\max} = 0.987$

34188 measured reflections
 4266 independent reflections
 3555 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.148$
 $S = 1.06$
 4266 reflections

178 parameters
 All H-atom parameters refined
 $\Delta\rho_{\max} = 0.76$ e Å⁻³
 $\Delta\rho_{\min} = -0.32$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the $\text{C4}-\text{C9}$ benzene ring.

$\text{D}-\text{H}\cdots\text{A}$	$\text{D}-\text{H}$	$\text{H}\cdots\text{A}$	$\text{D}\cdots\text{A}$	$\text{D}-\text{H}\cdots\text{A}$
$\text{C10}-\text{H10B}\cdots\text{Cg1}^{\text{i}}$	0.977 (14)	2.651 (15)	3.5273 (10)	149.1 (11)

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

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXTL (Sheldrick, 1998); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2369).

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supplementary materials

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N,N'-Bis[4-(dimethylamino)benzylidene]butane-1,4-diamine

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Comment

Schiff bases readily form stable complexes with most transition metal ions (Amirnasr *et al.*, 2002; Habibi *et al.*, 2007; Pal *et al.*, 2005) and show biological activity (Adsule *et al.*, 2006; Hou *et al.*, 2001; Ren *et al.*, 2002). In this paper, we report the crystal structure of the title Schiff base compound.

The molecule of the title compound lies across an crystallographic inversion centre and the asymmetric unit therefore contains one-half of the molecule (Fig. 1). The molecular structure of the title compound is non-planar but is in a staggered conformation about the C1–C1A bond [symmetry code (A): $1 - x, 1 - y, -z$]. The two benzene rings are parallel to each other, the dihedral angle between the planar benzylidene (C4–C9/C3/N1) and the planar bridging butane (C1/C2/C1A/C2A) [symmetry code (A): $1 - x, 1 - y, -z$] groups is $77.77(7)^\circ$. The dimethylamino group is slightly twisted respect to the attached benzene ring as indicated by torsion angles of $C10/N2/C7/C6 = -5.55(11)^\circ$ and $C11/N2/C7/C8 = 16.80(11)^\circ$. Bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and comparable to closely related structures (Bahron *et al.*, 2007; Kassim *et al.*, 2006).

In the crystal structure, Fig. 2, molecules are arranged into columns along the *a* axis by C—H \cdots π interactions (Table 1); Cg1 is the centroid of C4–C9 benzene ring.

Experimental

The title compound was prepared by mixing a solution of butane-1,4-diamine (1 mmol) dissolved in chloroform (3 ml) and 4-dimethylamino benzaldehyde (2 mmol) dissolved in chloroform (3 ml) and stirring the mixture under reflux for 8 h. Colorless single crystals suitable for *x*-ray structure determination were obtained after recrystallization by slow evaporation from an ethanol solution at room temperature after several days.

Refinement

All H atoms were located in a difference map and isotropically refined. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The final difference Fourier map showed a high peak 0.97\AA from C1.

Figures



Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. [Symmetry code: (A) $1 - x, 1 - y, -z$].

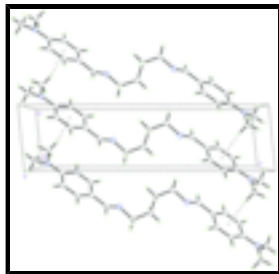


Fig. 2. The crystal packing of the title compound, viewed approximately along the *b* axis. C—H... π interactions are drawn as dashed lines.

N,N'-Bis[4-(dimethylamino)benzylidene]butane-1,4-diamine

Crystal data

$C_{22}H_{30}N_4$

$M_r = 350.50$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 5.2254$ (1) Å

$b = 9.5742$ (3) Å

$c = 19.4882$ (5) Å

$\beta = 95.512$ (1)°

$V = 970.47$ (4) Å³

$Z = 2$

$F_{000} = 380$

$D_x = 1.199$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4266 reflections

$\theta = 2.1$ – 35.0°

$\mu = 0.07$ mm⁻¹

$T = 100.0$ (1) K

Block, colorless

$0.57 \times 0.31 \times 0.19$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 8.33 pixels mm⁻¹

$T = 100.0$ (1) K

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.960$, $T_{\max} = 0.987$

34188 measured reflections

4266 independent reflections

3555 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 35.0^\circ$

$\theta_{\min} = 2.1^\circ$

$h = -8 \rightarrow 8$

$k = -15 \rightarrow 14$

$l = -31 \rightarrow 29$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.148$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0875P)^2 + 0.1308P]$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.06$	$(\Delta/\sigma)_{\max} < 0.001$
4266 reflections	$\Delta\rho_{\max} = 0.76 \text{ e } \text{\AA}^{-3}$
178 parameters	$\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.53637 (13)	0.48735 (7)	0.13556 (3)	0.02030 (14)
N2	-0.12267 (14)	0.52022 (8)	0.40171 (4)	0.02448 (15)
C1	0.62461 (15)	0.46510 (9)	0.01434 (4)	0.02237 (16)
C2	0.72103 (15)	0.51889 (9)	0.08599 (4)	0.02252 (16)
C3	0.51689 (14)	0.57613 (8)	0.18350 (4)	0.01926 (14)
C4	0.34554 (14)	0.56002 (7)	0.23809 (4)	0.01734 (13)
C5	0.17727 (13)	0.44685 (7)	0.24139 (4)	0.01744 (14)
C6	0.02178 (14)	0.43354 (8)	0.29458 (4)	0.01834 (14)
C7	0.02565 (14)	0.53483 (8)	0.34753 (4)	0.01792 (14)
C8	0.19010 (14)	0.65045 (8)	0.34311 (4)	0.01959 (14)
C9	0.34678 (14)	0.66113 (8)	0.28991 (4)	0.01941 (14)
C10	-0.30355 (16)	0.40527 (10)	0.40148 (5)	0.02695 (17)
C11	-0.16521 (18)	0.64055 (11)	0.44456 (5)	0.02906 (18)
H1A	0.760 (2)	0.4843 (13)	-0.0193 (7)	0.027 (3)*
H1B	0.589 (2)	0.3590 (15)	0.0147 (7)	0.036 (3)*
H2A	0.887 (2)	0.4716 (13)	0.1001 (7)	0.029 (3)*
H2B	0.758 (2)	0.6201 (14)	0.0852 (7)	0.031 (3)*
H3	0.620 (3)	0.6594 (15)	0.1863 (7)	0.038 (3)*
H5	0.159 (2)	0.3760 (15)	0.2047 (7)	0.035 (3)*
H6	-0.095 (2)	0.3549 (14)	0.2944 (7)	0.032 (3)*
H8	0.204 (2)	0.7260 (14)	0.3775 (7)	0.034 (3)*
H9	0.455 (2)	0.7385 (14)	0.2887 (7)	0.030 (3)*
H10A	-0.214 (2)	0.3155 (15)	0.3995 (6)	0.034 (3)*
H10B	-0.440 (3)	0.4081 (15)	0.3634 (7)	0.038 (3)*
H10C	-0.380 (3)	0.4063 (17)	0.4451 (8)	0.050 (4)*
H11A	-0.005 (3)	0.6769 (15)	0.4684 (7)	0.039 (3)*

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H11B	-0.252 (3)	0.7173 (16)	0.4176 (8)	0.043 (4)*
H11C	-0.269 (3)	0.6146 (19)	0.4806 (9)	0.059 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0218 (3)	0.0241 (3)	0.0153 (3)	-0.0016 (2)	0.0035 (2)	0.0030 (2)
N2	0.0250 (3)	0.0276 (3)	0.0222 (3)	-0.0009 (2)	0.0089 (2)	-0.0008 (2)
C1	0.0217 (3)	0.0271 (4)	0.0188 (3)	0.0026 (2)	0.0048 (2)	0.0028 (2)
C2	0.0206 (3)	0.0298 (4)	0.0174 (3)	-0.0023 (3)	0.0035 (2)	0.0037 (3)
C3	0.0199 (3)	0.0198 (3)	0.0182 (3)	-0.0007 (2)	0.0025 (2)	0.0027 (2)
C4	0.0180 (3)	0.0172 (3)	0.0168 (3)	0.0005 (2)	0.0014 (2)	0.0011 (2)
C5	0.0174 (3)	0.0177 (3)	0.0170 (3)	0.0006 (2)	0.0005 (2)	-0.0006 (2)
C6	0.0170 (3)	0.0188 (3)	0.0192 (3)	-0.0011 (2)	0.0013 (2)	0.0002 (2)
C7	0.0165 (3)	0.0205 (3)	0.0168 (3)	0.0019 (2)	0.0017 (2)	0.0008 (2)
C8	0.0210 (3)	0.0187 (3)	0.0191 (3)	0.0007 (2)	0.0018 (2)	-0.0024 (2)
C9	0.0209 (3)	0.0169 (3)	0.0205 (3)	-0.0015 (2)	0.0024 (2)	-0.0005 (2)
C10	0.0221 (4)	0.0309 (4)	0.0287 (4)	-0.0006 (3)	0.0073 (3)	0.0057 (3)
C11	0.0287 (4)	0.0371 (5)	0.0225 (4)	0.0014 (3)	0.0078 (3)	-0.0056 (3)

Geometric parameters (\AA , $^\circ$)

N1—C3	1.2744 (10)	C5—C6	1.3831 (10)
N1—C2	1.4611 (10)	C5—H5	0.983 (14)
N2—C7	1.3756 (9)	C6—C7	1.4148 (10)
N2—C10	1.4504 (11)	C6—H6	0.970 (13)
N2—C11	1.4525 (11)	C7—C8	1.4090 (10)
C1—C1 ⁱ	1.5215 (16)	C8—C9	1.3850 (10)
C1—C2	1.5275 (12)	C8—H8	0.984 (13)
C1—H1A	1.027 (12)	C9—H9	0.935 (13)
C1—H1B	1.033 (14)	C10—H10A	0.981 (14)
C2—H2A	0.994 (13)	C10—H10B	0.977 (14)
C2—H2B	0.989 (13)	C10—H10C	0.972 (16)
C3—C4	1.4629 (10)	C11—H11A	0.982 (15)
C3—H3	0.962 (14)	C11—H11B	0.988 (15)
C4—C9	1.3985 (10)	C11—H11C	0.959 (17)
C4—C5	1.4007 (10)		
C3—N1—C2	116.83 (7)	C5—C6—C7	121.01 (7)
C7—N2—C10	119.33 (7)	C5—C6—H6	119.0 (7)
C7—N2—C11	119.29 (7)	C7—C6—H6	120.0 (7)
C10—N2—C11	117.90 (7)	N2—C7—C8	121.25 (7)
C1 ⁱ —C1—C2	112.54 (8)	N2—C7—C6	121.36 (7)
C1 ⁱ —C1—H1A	107.8 (7)	C8—C7—C6	117.38 (6)
C2—C1—H1A	109.7 (7)	C9—C8—C7	120.77 (7)
C1 ⁱ —C1—H1B	106.5 (7)	C9—C8—H8	116.4 (7)
C2—C1—H1B	111.7 (7)	C7—C8—H8	122.8 (7)
H1A—C1—H1B	108.4 (10)	C8—C9—C4	121.83 (7)
N1—C2—C1	110.73 (6)	C8—C9—H9	118.8 (8)

N1—C2—H2A	109.7 (8)	C4—C9—H9	119.4 (8)
C1—C2—H2A	107.6 (7)	N2—C10—H10A	110.6 (8)
N1—C2—H2B	110.8 (7)	N2—C10—H10B	114.0 (8)
C1—C2—H2B	111.4 (8)	H10A—C10—H10B	108.0 (11)
H2A—C2—H2B	106.5 (11)	N2—C10—H10C	108.1 (9)
N1—C3—C4	124.08 (7)	H10A—C10—H10C	106.3 (12)
N1—C3—H3	120.6 (8)	H10B—C10—H10C	109.5 (12)
C4—C3—H3	115.3 (8)	N2—C11—H11A	112.5 (8)
C9—C4—C5	117.49 (6)	N2—C11—H11B	111.8 (9)
C9—C4—C3	119.44 (6)	H11A—C11—H11B	108.4 (12)
C5—C4—C3	123.07 (6)	N2—C11—H11C	110.1 (11)
C6—C5—C4	121.48 (7)	H11A—C11—H11C	105.1 (13)
C6—C5—H5	117.3 (8)	H11B—C11—H11C	108.7 (13)
C4—C5—H5	121.1 (8)		
C3—N1—C2—C1	-146.09 (7)	C10—N2—C7—C6	-5.55 (11)
C1 ⁱ —C1—C2—N1	62.55 (10)	C11—N2—C7—C6	-164.07 (8)
C2—N1—C3—C4	-178.83 (7)	C5—C6—C7—N2	-178.02 (7)
N1—C3—C4—C9	177.38 (7)	C5—C6—C7—C8	1.14 (11)
N1—C3—C4—C5	-2.61 (12)	N2—C7—C8—C9	177.21 (7)
C9—C4—C5—C6	-1.36 (11)	C6—C7—C8—C9	-1.95 (11)
C3—C4—C5—C6	178.63 (7)	C7—C8—C9—C4	1.14 (11)
C4—C5—C6—C7	0.51 (11)	C5—C4—C9—C8	0.54 (11)
C10—N2—C7—C8	175.32 (7)	C3—C4—C9—C8	-179.46 (6)
C11—N2—C7—C8	16.80 (11)		

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10—H10B \cdots Cg1 ⁱⁱ	0.977 (14)	2.651 (15)	3.5273 (10)	149.1 (11)

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

Fig. 1

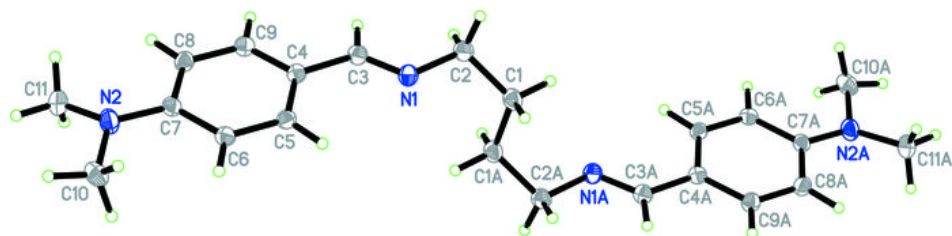


Fig. 2

