Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-Bis(4-methylbenzylidene)benzene-1,4-diamine

Haoyang Li, Yonggang Xiang and Hongfei Han*

Department of Chemistry, Taiyuan Normal University, Taiyuan 030031, People's Republic of China
Correspondence e-mail: hhf_2222@yahoo.com.cn

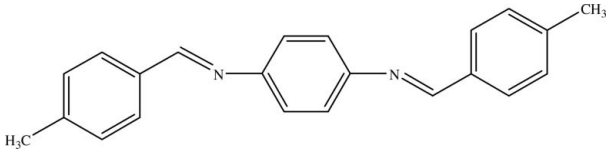
Received 8 October 2011; accepted 10 October 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.145; data-to-parameter ratio = 13.8.

The centrosymmetric title compound, $C_{22}H_{20}N_2$, crystallizes with one half-molecule in the asymmetric unit. The dihedral angle between the central and outer benzene rings is 46.2 (2)°.

Related literature

For the use of Schiff bases as ligands in metal complexes, see: Chen *et al.* (2008); May *et al.* (2004).



Experimental

Crystal data

$C_{22}H_{20}N_2$
M_r = 312.40
Monoclinic, $P2_1/c$
a = 6.4750 (6) Å
b = 7.1561 (8) Å
c = 19.594 (2) Å
β = 107.555 (1)°
V = 865.61 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 293 K
0.46 × 0.40 × 0.37 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.968, T_{max} = 0.974
4151 measured reflections
1532 independent reflections
844 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.145
S = 1.04
1532 reflections
111 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was carried out under the sponsorship of the ShanXi scientific technology project (20110321044).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5670).

References

Bruker (2007). *APEX2* and *SAINT* Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, Z. H., Morimoto, H., Matsunaga, S. & Shibasaki, M. (2008). *J. Am. Chem. Soc.* **130**, 2170–2171.
May, J. P., Ting, R., Lermer, L., Thomas, J. M., Roupioz, Y. & Perrin, D. M. (2004). *J. Am. Chem. Soc.* **126**, 4145–4156.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o2953 [doi:10.1107/S1600536811041675]

N,*N*′-Bis(4-methylbenzylidene)benzene-1,4-diamine

Haoyang Li, Yonggang Xiang and Hongfei Han

S1. Comment

Schiff bases containing the C═N bond have been receiving considerable attention for many years, primarily due to their importance as ligands in metal complexes with special biological (May *et al.*, 2004), and catalytic properties (Chen *et al.*, 2008).

As a part of our studies on synthesis and structural peculiarities of Schiff bases derived from 1,4-benzenediamine and 4-methyl benzaldehyde, we determined the structure of the title compound (Fig. 1). The molecule includes two C═ N bonds, which are coplanar. The distance between the C atom and the N atom in the C═N bond is 1.266 (2) Å. In the structure the dihedral angle between adjacent benzene rings planes is 46.2 (2)°.

S2. Experimental

1,4-benzenediamine (0.324 g, 3 mmol) was added dropwise with stirring at 273K to a solution of 4-methyl benzaldehyde (0.721 g, 6 mmol) in ethanol. The mixture were warmed to room temperature and stirred for 2 h. The reaction mixture was filtered and the filter cake was recrystallized from ethanol (yield 75%). Crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of a tetrahydrofuran solution.

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.96 Å), and refined as riding with U_{iso}(H) = 1.2U_{eq} or 1.5U_{eq} (methyl H atoms).

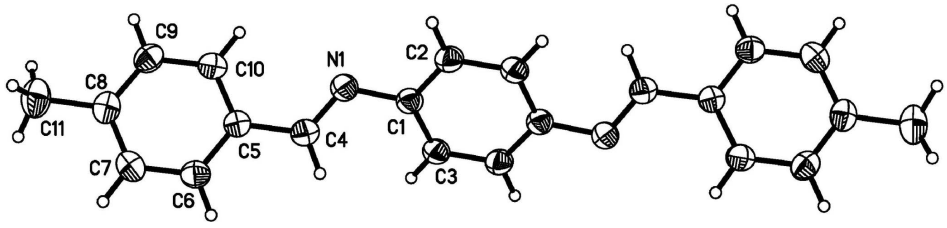


Figure 1

The molecular structure, showing the atom–numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

N,*N*′-Bis(4-methylbenzylidene)benzene-1,4-diamine

Crystal data

$C_{22}H_{20}N_2$
M_r = 312.40
Monoclinic, $P2_1/c$
a = 6.4750 (6) Å
b = 7.1561 (8) Å
c = 19.594 (2) Å
β = 107.555 (1)°
V = 865.61 (16) Å^3

$Z = 2$
$F(000) = 332$
$D_x = 1.199$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 954 reflections

$\theta = 2.9$–$23.3°$
$\mu = 0.07$ mm^{-1}
$T = 293$ K
Block, colorless
0.46 × 0.40 × 0.37 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.968$, $T_{max} = 0.974$

4151 measured reflections
1532 independent reflections
844 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.1°$
$h = -7 \rightarrow 7$
$k = -8 \rightarrow 6$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.145$
$S = 1.04$
1532 reflections
111 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.1422P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.069 (8)

Special details

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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.3167 (3)	0.4579 (2)	0.61261 (9)	0.0604 (6)
C1	0.4131 (3)	0.4803 (3)	0.55707 (11)	0.0543 (6)
C2	0.2944 (3)	0.5650 (3)	0.49390 (11)	0.0604 (6)
H2	0.1554	0.6084	0.4891	0.072*
C3	0.6190 (3)	0.4146 (3)	0.56201 (11)	0.0602 (6)
H3	0.7002	0.3558	0.6038	0.072*
C4	0.4309 (4)	0.4832 (3)	0.67687 (11)	0.0558 (6)
H4	0.5734	0.5226	0.6857	0.067*
C5	0.3491 (3)	0.4533 (3)	0.73774 (11)	0.0504 (5)
C6	0.4766 (4)	0.4939 (3)	0.80622 (11)	0.0609 (6)

H6	0.6167	0.5381	0.8135	0.073*
C7	0.3996 (4)	0.4699 (3)	0.86443 (12)	0.0678 (7)
H7	0.4889	0.4982	0.9101	0.081*
C8	0.1925 (4)	0.4049 (3)	0.85571 (12)	0.0621 (6)
C9	0.0668 (4)	0.3596 (3)	0.78732 (13)	0.0645 (6)
H9	−0.0723	0.3134	0.7802	0.077*
C10	0.1436 (3)	0.3814 (3)	0.72947 (12)	0.0609 (6)
H10	0.0564	0.3476	0.6841	0.073*
C11	0.1042 (5)	0.3887 (4)	0.91853 (13)	0.0913 (9)
H11A	0.2211	0.3657	0.9614	0.137*
H11B	0.0028	0.2872	0.9105	0.137*
H11C	0.0326	0.5030	0.9236	0.137*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0561 (12)	0.0671 (13)	0.0566 (11)	0.0008 (9)	0.0150 (10)	0.0079 (9)
C1	0.0510 (14)	0.0562 (13)	0.0538 (13)	−0.0039 (10)	0.0131 (11)	0.0040 (10)
C2	0.0483 (13)	0.0689 (15)	0.0620 (14)	0.0065 (10)	0.0137 (11)	0.0091 (11)
C3	0.0513 (14)	0.0707 (15)	0.0540 (13)	0.0037 (11)	0.0090 (11)	0.0118 (11)
C4	0.0535 (14)	0.0499 (13)	0.0631 (14)	−0.0004 (10)	0.0162 (12)	0.0037 (10)
C5	0.0527 (13)	0.0424 (11)	0.0553 (13)	0.0031 (9)	0.0153 (11)	0.0031 (9)
C6	0.0574 (14)	0.0571 (14)	0.0659 (15)	−0.0033 (10)	0.0151 (12)	−0.0020 (11)
C7	0.0766 (17)	0.0665 (15)	0.0560 (14)	0.0031 (12)	0.0137 (13)	−0.0026 (11)
C8	0.0741 (16)	0.0499 (13)	0.0678 (15)	0.0103 (12)	0.0299 (13)	0.0089 (11)
C9	0.0597 (15)	0.0599 (14)	0.0774 (16)	−0.0007 (11)	0.0261 (13)	0.0070 (12)
C10	0.0555 (14)	0.0619 (14)	0.0609 (14)	−0.0018 (11)	0.0113 (11)	0.0006 (11)
C11	0.114 (2)	0.0928 (19)	0.0806 (18)	0.0107 (17)	0.0488 (17)	0.0172 (14)

Geometric parameters (Å, º)

N1—C4	1.266 (2)	C6—C7	1.387 (3)
N1—C1	1.418 (3)	C6—H6	0.9300
C1—C2	1.384 (3)	C7—C8	1.380 (3)
C1—C3	1.389 (3)	C7—H7	0.9300
C2—C3^{i}	1.381 (3)	C8—C9	1.380 (3)
C2—H2	0.9300	C8—C11	1.510 (3)
C3—C2^{i}	1.381 (3)	C9—C10	1.377 (3)
C3—H3	0.9300	C9—H9	0.9300
C4—C5	1.459 (3)	C10—H10	0.9300
C4—H4	0.9300	C11—H11A	0.9600
C5—C6	1.378 (3)	C11—H11B	0.9600
C5—C10	1.390 (3)	C11—H11C	0.9600
C4—N1—C1	119.14 (19)	C8—C7—C6	121.1 (2)
C2—C1—C3	118.22 (19)	C8—C7—H7	119.4
C2—C1—N1	118.79 (19)	C6—C7—H7	119.4
C3—C1—N1	122.94 (19)	C7—C8—C9	117.8 (2)

C3^{i}—C2—C1	120.5 (2)	C7—C8—C11	121.2 (2)
C3^{i}—C2—H2	119.8	C9—C8—C11	121.1 (2)
C1—C2—H2	119.8	C10—C9—C8	121.3 (2)
C2^{i}—C3—C1	121.3 (2)	C10—C9—H9	119.4
C2^{i}—C3—H3	119.3	C8—C9—H9	119.4
C1—C3—H3	119.3	C9—C10—C5	121.1 (2)
N1—C4—C5	123.0 (2)	C9—C10—H10	119.4
N1—C4—H4	118.5	C5—C10—H10	119.4
C5—C4—H4	118.5	C8—C11—H11A	109.5
C6—C5—C10	117.6 (2)	C8—C11—H11B	109.5
C6—C5—C4	120.4 (2)	H11A—C11—H11B	109.5
C10—C5—C4	122.0 (2)	C8—C11—H11C	109.5
C5—C6—C7	121.1 (2)	H11A—C11—H11C	109.5
C5—C6—H6	119.5	H11B—C11—H11C	109.5
C7—C6—H6	119.5		

C4—N1—C1—C2	−140.7 (2)	C4—C5—C6—C7	178.63 (19)
C4—N1—C1—C3	41.9 (3)	C5—C6—C7—C8	−0.1 (3)
C3—C1—C2—C3^{i}	−0.9 (3)	C6—C7—C8—C9	1.8 (3)
N1—C1—C2—C3^{i}	−178.49 (19)	C6—C7—C8—C11	−176.6 (2)
C2—C1—C3—C2^{i}	0.9 (4)	C7—C8—C9—C10	−1.1 (3)
N1—C1—C3—C2^{i}	178.39 (19)	C11—C8—C9—C10	177.3 (2)
C1—N1—C4—C5	−176.51 (17)	C8—C9—C10—C5	−1.2 (3)
N1—C4—C5—C6	−176.06 (19)	C6—C5—C10—C9	2.8 (3)
N1—C4—C5—C10	4.8 (3)	C4—C5—C10—C9	−178.00 (19)
C10—C5—C6—C7	−2.2 (3)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.