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N,N'-Bis(3-methoxybenzylidene)ethane-1,2-diamine

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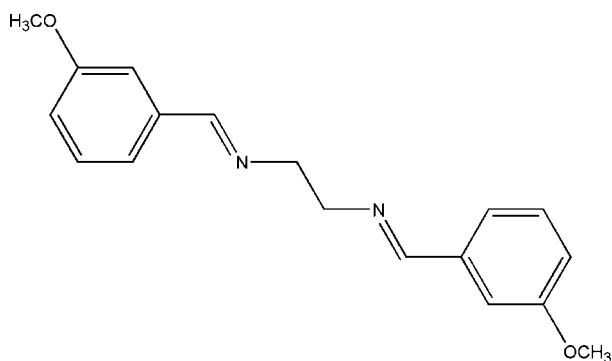
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.041; wR factor = 0.109; data-to-parameter ratio = 20.3.

The molecule of the title bidentate Schiff base ligand, $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2$, has twofold crystallographic rotation symmetry, giving one half-molecule per asymmetric unit. It adopts a twisted *E* configuration with respect to the azomethine $\text{C}=\text{N}$ bond. The imino group is coplanar with the aromatic ring. The dihedral angle between the two benzene rings is 69.52 (5)°. The methoxy group is coplanar with the benzene ring, as indicated by the $\text{C}-\text{O}-\text{C}-\text{C}$ torsion angle of -179.56 (8)°. In the unit cell, molecules are linked together by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming chains along the *a* axis; these chains are further stacked down the *b* axis by both intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For related structures see: Fun *et al.* (2008*a,b,c,d*); Calligaris & Randaccio, (1987). For information on Schiff base complexes and their applications, see: Kia *et al.* (2007*a,b*); Pal *et al.* (2005); Hou *et al.* (2001)


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Experimental

Crystal data

 $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2$
 $M_r = 296.36$
 Monoclinic, $C2/c$
 $a = 22.7076$ (3) Å
 $b = 6.0374$ (1) Å
 $c = 11.6789$ (2) Å
 $\beta = 100.235$ (1)°

 $V = 1575.64$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 100.0$ (1) K
 $0.49 \times 0.33 \times 0.22$ mm

Data collection

 Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.886$, $T_{\max} = 0.982$

 11683 measured reflections
 2298 independent reflections
 1879 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.108$
 $S = 1.10$
 2298 reflections
 113 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.35$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C9}-\text{H9A}\cdots\text{O1}^{\text{i}}$	0.96	2.50	3.3809 (13)	153
$\text{C8}-\text{H8B}\cdots\text{Cg1}^{\text{ii}}$	0.984 (13)	2.822 (13)	3.6221 (12)	138.9 (9)
$\text{C9}-\text{H9C}\cdots\text{Cg1}^{\text{iii}}$	0.96	2.75	3.5636 (12)	143

 Symmetry codes: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, y + 1, z$; (iii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$. Cg1 is the centroid of the C1–C6 benzene ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); 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: FL2216).

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supporting information

Acta Cryst. (2008). E64, o1803–o1804 [doi:10.1107/S1600536808026652]

N,N'*-Bis(3-methoxybenzylidene)ethane-1,2-diamine*Hoong-Kun Fun, Valiollah Mirkhani and Akbar Rostami Vartooni****S1. Comment**

Schiff bases are one of most prevalent mixed-donor ligands found in the field of coordination chemistry. There has been growing interest in Schiff base ligands, mainly because of their wide applications in the fields of biochemistry, synthesis, and catalysis (Kia *et al.*, 2007*a,b*; Pal *et al.*, 2005; Hou *et al.*, 2001). Many Schiff base complexes have been structurally characterized, but in comparison only a relatively small number of free Schiff bases have been described (Calligaris & Randaccio, 1987). As an extension of our work (Fun *et al.*, 2008*a*, 2008*b*, 2008*c*, 2008*d*) on the structural characterization of Schiff base compounds, the title compound (I), (Fig. 1), is reported here.

(I) has twofold crystallographic rotation symmetry to give 1/2 molecule per asymmetric unit and it adopts a twisted *E* configuration with respect to the azomethine C=N bond. Bond lengths and angles are within normal ranges. The imino group is coplanar with the aromatic ring. The dihedral angle between two phenyl rings is 69.52 (5)°. The methoxy group is coplanar with the benzene ring as indicated by the C9–O1–C2–C1 torsion of -179.56 (8)°. In the unit cell, (Fig. 2), neighbouring molecules are linked together by intermolecular C—H···O hydrogen bonds to form chains along the *a*-axis and these chains are further stacked down the *b*-axis by both intermolecular C—H···O and C—H··· π interactions (Table 1).

S2. Experimental

The overall synthetic method has been described earlier (Fun *et al.*, 2008*a*), except that ethylenediamine (1 mmol, 60 mg) and 3-methoxybenzaldehyde (2 mmol, 137 mg) were used as starting materials. Single crystals suitable for *X*-ray diffraction were obtained by evaporation of an ethanol solution at room temperature.

S3. Refinement

H atoms bound to C7 and C8 were located from the difference Fourier map and freely refined. The rest of the hydrogen atoms were positioned geometrically with C—H = 0.93–0.96 Å and refined in riding mode with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$. A rotating-group model was used for the methyl group.

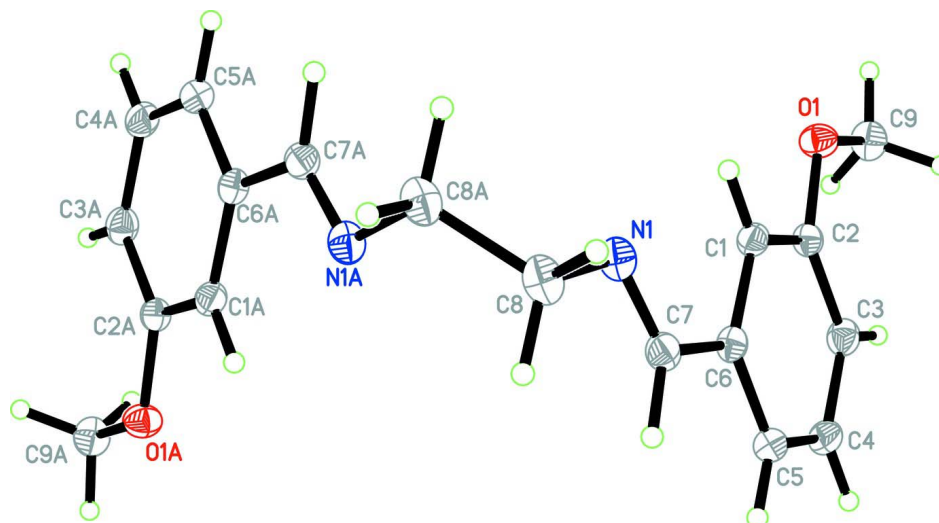


Figure 1

The molecular structure of (I) with atom labels and 50% probability ellipsoids for non-H atoms [symmetry code for A: $-x + 1, Y, 0.5 - Z$].

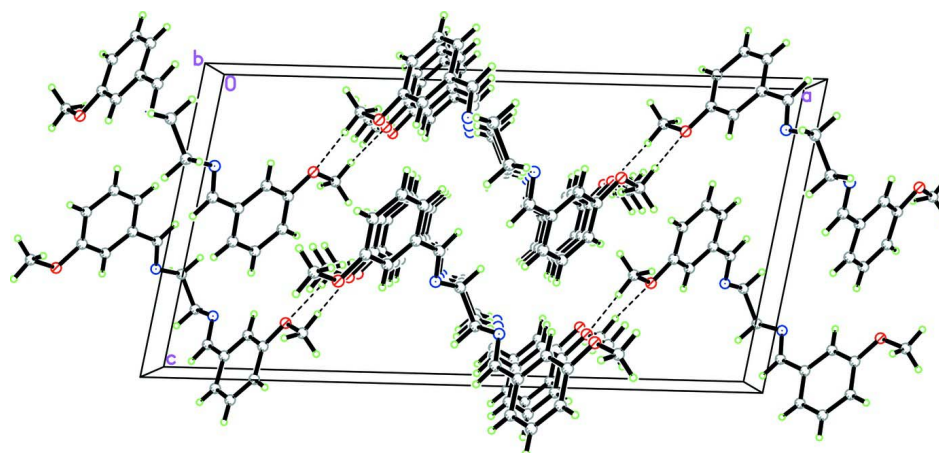


Figure 2

The crystal packing of (I), viewed down the b axis, showing chains along the a axis and stacking of these chains along the b axis. Intermolecular interactions are shown as dashed lines.

N,N'-Bis(3-methoxybenzylidene)ethane-1,2-diamine

Crystal data

$C_{18}H_{20}N_2O_2$

$M_r = 296.36$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 22.7076 (3) \text{ \AA}$

$b = 6.0374 (1) \text{ \AA}$

$c = 11.6789 (2) \text{ \AA}$

$\beta = 100.235 (1)^\circ$

$V = 1575.64 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 632$

$D_x = 1.249 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3509 reflections

$\theta = 3.6\text{--}33.9^\circ$

$\mu = 0.08 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Block, colourless

$0.49 \times 0.33 \times 0.22 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2005)
 $T_{\min} = 0.886$, $T_{\max} = 0.982$

11683 measured reflections
2298 independent reflections
1879 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\text{max}} = 30.0^\circ$, $\theta_{\text{min}} = 3.5^\circ$
 $h = -31 \rightarrow 31$
 $k = -8 \rightarrow 8$
 $l = -14 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.108$
 $S = 1.11$
2298 reflections
113 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 atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.7792P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.35 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.21 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.29856 (3)	0.12810 (12)	0.16829 (6)	0.02029 (18)
N1	0.44163 (4)	0.77578 (14)	0.17093 (8)	0.0197 (2)
C1	0.36408 (4)	0.39310 (16)	0.11788 (8)	0.0166 (2)
H1A	0.3629	0.4625	0.1885	0.020*
C2	0.33032 (4)	0.20304 (16)	0.08715 (8)	0.0167 (2)
C3	0.33099 (4)	0.10022 (17)	-0.01966 (9)	0.0196 (2)
H3A	0.3080	-0.0257	-0.0407	0.024*
C4	0.36637 (4)	0.18805 (18)	-0.09415 (9)	0.0216 (2)
H4A	0.3671	0.1197	-0.1653	0.026*
C5	0.40051 (4)	0.37574 (18)	-0.06388 (9)	0.0203 (2)
H5A	0.4240	0.4328	-0.1145	0.024*
C6	0.39970 (4)	0.47980 (17)	0.04289 (8)	0.0172 (2)
C7	0.43794 (4)	0.67504 (17)	0.07480 (9)	0.0184 (2)
C8	0.48159 (5)	0.96685 (17)	0.18898 (10)	0.0215 (2)

C9	0.26352 (5)	-0.06749 (17)	0.14119 (10)	0.0225 (2)
H9A	0.2443	-0.1047	0.2055	0.034*
H9B	0.2890	-0.1874	0.1267	0.034*
H9C	0.2337	-0.0419	0.0731	0.034*
H7A	0.4614 (6)	0.722 (2)	0.0143 (11)	0.027 (3)*
H8B	0.4562 (6)	1.100 (2)	0.1792 (11)	0.025 (3)*
H8A	0.5085 (6)	0.971 (2)	0.1293 (12)	0.026 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0226 (4)	0.0197 (4)	0.0195 (4)	-0.0036 (3)	0.0063 (3)	-0.0004 (3)
N1	0.0174 (4)	0.0181 (4)	0.0228 (4)	-0.0004 (3)	0.0018 (3)	0.0020 (3)
C1	0.0170 (4)	0.0178 (4)	0.0144 (4)	0.0026 (3)	0.0015 (3)	-0.0002 (3)
C2	0.0152 (4)	0.0179 (4)	0.0167 (5)	0.0027 (3)	0.0020 (3)	0.0019 (3)
C3	0.0190 (5)	0.0191 (5)	0.0196 (5)	0.0007 (4)	0.0006 (4)	-0.0027 (4)
C4	0.0202 (5)	0.0281 (5)	0.0157 (5)	0.0031 (4)	0.0014 (4)	-0.0039 (4)
C5	0.0177 (5)	0.0271 (5)	0.0161 (5)	0.0012 (4)	0.0031 (4)	0.0016 (4)
C6	0.0153 (4)	0.0192 (5)	0.0163 (4)	0.0022 (3)	0.0003 (3)	0.0022 (4)
C7	0.0164 (4)	0.0197 (5)	0.0189 (5)	0.0008 (4)	0.0026 (4)	0.0056 (4)
C8	0.0186 (5)	0.0162 (5)	0.0291 (6)	-0.0010 (4)	0.0027 (4)	0.0027 (4)
C9	0.0226 (5)	0.0189 (5)	0.0255 (5)	-0.0033 (4)	0.0025 (4)	0.0019 (4)

Geometric parameters (Å, °)

O1—C2	1.3659 (12)	C4—H4A	0.9300
O1—C9	1.4277 (12)	C5—C6	1.3993 (14)
N1—C7	1.2665 (14)	C5—H5A	0.9300
N1—C8	1.4597 (13)	C6—C7	1.4723 (14)
C1—C2	1.3912 (14)	C7—H7A	0.999 (13)
C1—C6	1.3954 (13)	C8—C8 ⁱ	1.519 (2)
C1—H1A	0.9300	C8—H8B	0.984 (13)
C2—C3	1.3958 (14)	C8—H8A	1.005 (13)
C3—C4	1.3899 (14)	C9—H9A	0.9600
C3—H3A	0.9300	C9—H9B	0.9600
C4—C5	1.3830 (15)	C9—H9C	0.9600
C2—O1—C9	117.53 (8)	C1—C6—C7	121.54 (9)
C7—N1—C8	116.68 (9)	C5—C6—C7	118.98 (9)
C2—C1—C6	120.12 (9)	N1—C7—C6	123.50 (9)
C2—C1—H1A	119.9	N1—C7—H7A	122.1 (8)
C6—C1—H1A	119.9	C6—C7—H7A	114.4 (8)
O1—C2—C1	115.39 (8)	N1—C8—C8 ⁱ	111.10 (7)
O1—C2—C3	124.31 (9)	N1—C8—H8B	106.9 (8)
C1—C2—C3	120.29 (9)	C8 ⁱ —C8—H8B	108.8 (8)
C4—C3—C2	119.28 (9)	N1—C8—H8A	111.0 (8)
C4—C3—H3A	120.4	C8 ⁱ —C8—H8A	110.5 (7)
C2—C3—H3A	120.4	H8B—C8—H8A	108.3 (11)

C5—C4—C3	120.84 (9)	O1—C9—H9A	109.5
C5—C4—H4A	119.6	O1—C9—H9B	109.5
C3—C4—H4A	119.6	H9A—C9—H9B	109.5
C4—C5—C6	120.01 (9)	O1—C9—H9C	109.5
C4—C5—H5A	120.0	H9A—C9—H9C	109.5
C6—C5—H5A	120.0	H9B—C9—H9C	109.5
C1—C6—C5	119.46 (9)		
C9—O1—C2—C1	-179.56 (8)	C2—C1—C6—C5	0.84 (14)
C9—O1—C2—C3	-0.45 (14)	C2—C1—C6—C7	-177.36 (8)
C6—C1—C2—O1	177.98 (8)	C4—C5—C6—C1	-0.24 (15)
C6—C1—C2—C3	-1.17 (14)	C4—C5—C6—C7	178.01 (9)
O1—C2—C3—C4	-178.19 (9)	C8—N1—C7—C6	-179.92 (9)
C1—C2—C3—C4	0.88 (15)	C1—C6—C7—N1	0.51 (15)
C2—C3—C4—C5	-0.27 (15)	C5—C6—C7—N1	-177.70 (10)
C3—C4—C5—C6	-0.04 (15)	C7—N1—C8—C8 ⁱ	-136.92 (11)

Symmetry code: (i) $-x+1, y, -z+1/2$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9A \cdots O1 ⁱⁱ	0.96	2.50	3.3809 (13)	153
C8—H8B \cdots Cg1 ⁱⁱⁱ	0.984 (13)	2.822 (13)	3.6221 (12)	138.9 (9)
C9—H9C \cdots Cg1 ^{iv}	0.96	2.75	3.5636 (12)	143

Symmetry codes: (ii) $-x+1/2, y-1/2, -z+1/2$; (iii) $x, y+1, z$; (iv) $-x+1/2, -y+1/2, -z$.