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## Structure Reports

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## *N,N'*-Bis(4-bromobenzylidene)biphenyl-2,2'-diamine

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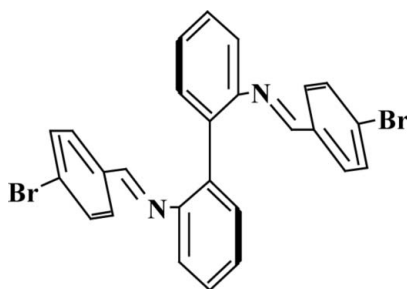
Received 7 January 2009; accepted 8 January 2009

Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.034;  $wR$  factor = 0.116; data-to-parameter ratio = 18.7.

The complete molecule of the title Schiff base,  $\text{C}_{26}\text{H}_{18}\text{Br}_2\text{N}_2$ , is generated by crystallographic twofold symmetry. The aromatic rings of the biphenylene portion of the molecule are twisted, as shown by the dihedral of  $61.8$  ( $1^\circ$ ) formed between them.

### Related literature

There are relatively few crystallographic reports of Schiff bases formed by condensing biphenyl-2,2'-diamine with aldehydes or ketones. See: Alajarín *et al.* (2007); Coxall *et al.* (2003); Cunningham *et al.* (2004); FINDER *et al.* (1973); Pruszyński *et al.* (1992).



### Experimental

#### Crystal data

 $\text{C}_{26}\text{H}_{18}\text{Br}_2\text{N}_2$  $M_r = 518.24$ Orthorhombic,  $Aba2$  $a = 15.9691$  (10) Å $b = 8.3482$  (5) Å $c = 16.7767$  (11) Å $V = 2236.6$  (2) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 3.64$  mm<sup>-1</sup> $T = 295$  (2) K $0.28 \times 0.25 \times 0.19$  mm

#### Data collection

Rigaku R-Axis RAPID diffractometer

Absorption correction: multi-scan (ABSCOR; Higashi, 1995)

 $T_{\min} = 0.429$ ,  $T_{\max} = 0.545$  (expected range = 0.394–0.501)

10424 measured reflections

2542 independent reflections

1333 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.040$ 

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$  $wR(F^2) = 0.116$  $S = 0.98$ 

2542 reflections

136 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.28$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.36$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

1209 Friedel pairs

Flack parameter:  $-0.013$  (15)

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2356).

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**supplementary materials**

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***N,N'*-Bis(4-bromobenzylidene)biphenyl-2,2'-diamine**

**S. Dehghanpour, S. Asadizadeh, S. Gao and S. W. Ng**

**Comment**

(type here to add)

**Experimental**

Biphenyl-2,2'-diamine (5 mmol) and 4-bromobenzaldehyde (10 mmol) were dissolved in ethanol (50 ml). The solution was heated for 5 h; the solid that separated from the cooled solution was collected and recrystallized from chloroform; a second recrystallization was effected with ethanol. The yield as 90%. Analysis found: C 60.20, H 3.54, N 5.43; C<sub>26</sub>H<sub>18</sub>Br<sub>2</sub>N<sub>2</sub> requires: C 60.26, H 3.50, N 5.41.

**Refinement**

Carbon-bound H atoms were placed in calculated positions [C—H 0.93 Å and  $U_{\text{iso}}(\text{H})$  1.2–1.5 $U_{\text{eq}}(\text{C})$ ] and were included in the refinement in the riding-model approximation.

**Figures**

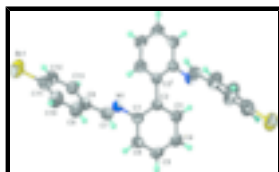


Fig. 1. Thermal ellipsoid plot (Barbour, 2001); displacement ellipsoids are drawn at the 50% probability level, and H atoms as spheres of arbitrary radius. (Symmetry code:  $i = 2 - x, 3 - y, z$ ).

***N,N'*-Bis(4-bromobenzylidene)biphenyl-2,2'-diamine**

*Crystal data*

C<sub>26</sub>H<sub>18</sub>Br<sub>2</sub>N<sub>2</sub>

$M_r = 518.24$

Orthorhombic, *Ab*a2

Hall symbol: A 2 -2ac

$a = 15.9691$  (10) Å

$b = 8.3482$  (5) Å

$c = 16.7767$  (11) Å

$V = 2236.6$  (2) Å<sup>3</sup>

$Z = 4$

$F_{000} = 1032$

$D_x = 1.539$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 5898 reflections

$\theta = 3.0$ – $27.4^\circ$

$\mu = 3.64$  mm<sup>-1</sup>

$T = 295$  (2) K

Cuboid, light yellow

$0.28 \times 0.25 \times 0.19$  mm

# supplementary materials

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## Data collection

Rigaku R-Axis RAPID diffractometer	2542 independent reflections
Radiation source: fine-focus sealed tube	1333 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.040$
Detector resolution: 10.000 pixels $\text{mm}^{-1}$	$\theta_{\text{max}} = 27.4^\circ$
$T = 295(2)$ K	$\theta_{\text{min}} = 3.0^\circ$
$\omega$ scans	$h = -18 \rightarrow 20$
Absorption correction: Multi-scan (ABSCOR; Higashi, 1995)	$k = -10 \rightarrow 10$
$T_{\text{min}} = 0.429$ , $T_{\text{max}} = 0.545$	$l = -21 \rightarrow 21$
10424 measured reflections	

## Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.034$	$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2]$
$wR(F^2) = 0.116$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2542 reflections	$\Delta\rho_{\text{max}} = 0.28 \text{ e } \text{\AA}^{-3}$
136 parameters	$\Delta\rho_{\text{min}} = -0.36 \text{ e } \text{\AA}^{-3}$
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1209 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: $-0.013$ (15)

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.89090 (4)	0.63217 (6)	0.50003 (6)	0.1058 (3)
N1	0.8979 (2)	1.3119 (4)	0.7320 (3)	0.0604 (9)
C1	0.8877 (2)	1.4642 (5)	0.7689 (3)	0.0555 (10)
C2	0.9576 (2)	1.5394 (4)	0.8036 (2)	0.0537 (9)
C3	0.9461 (3)	1.6846 (5)	0.8415 (3)	0.0650 (11)
H3	0.9920	1.7371	0.8636	0.078*
C4	0.8672 (3)	1.7534 (6)	0.8471 (4)	0.0676 (13)
H4	0.8605	1.8514	0.8727	0.081*
C5	0.7989 (3)	1.6768 (5)	0.8150 (3)	0.0685 (12)
H5	0.7459	1.7214	0.8205	0.082*
C6	0.8087 (2)	1.5347 (5)	0.7748 (3)	0.0656 (12)
H6	0.7626	1.4854	0.7514	0.079*
C7	0.8654 (3)	1.2832 (7)	0.6649 (3)	0.0633 (12)
H7	0.8381	1.3663	0.6387	0.076*

C8	0.8685 (3)	1.1275 (5)	0.6264 (3)	0.0597 (11)
C9	0.8443 (3)	1.1094 (5)	0.5480 (3)	0.0818 (14)
H9	0.8236	1.1973	0.5202	0.098*
C10	0.8505 (3)	0.9629 (6)	0.5101 (4)	0.0891 (14)
H10	0.8346	0.9518	0.4570	0.107*
C11	0.8805 (3)	0.8339 (6)	0.5523 (3)	0.0714 (13)
C12	0.9015 (3)	0.8464 (5)	0.6305 (3)	0.0703 (13)
H12	0.9195	0.7566	0.6585	0.084*
C13	0.8961 (2)	0.9926 (5)	0.6683 (3)	0.0646 (11)
H13	0.9108	1.0016	0.7217	0.078*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.1445 (6)	0.0704 (3)	0.1026 (5)	-0.0098 (2)	0.0284 (5)	-0.0131 (4)
N1	0.058 (2)	0.0512 (18)	0.072 (3)	-0.0048 (15)	0.0000 (19)	-0.0029 (19)
C1	0.050 (3)	0.058 (2)	0.058 (3)	0.0000 (18)	0.0025 (18)	0.009 (2)
C2	0.054 (2)	0.052 (2)	0.056 (2)	-0.0007 (16)	0.0007 (19)	0.0069 (19)
C3	0.063 (3)	0.064 (2)	0.067 (3)	-0.001 (2)	-0.005 (2)	-0.004 (2)
C4	0.081 (4)	0.057 (3)	0.065 (3)	0.004 (2)	-0.002 (3)	-0.002 (2)
C5	0.059 (3)	0.064 (2)	0.082 (3)	0.017 (2)	0.008 (2)	0.008 (2)
C6	0.052 (3)	0.065 (3)	0.080 (3)	-0.0025 (19)	0.000 (2)	0.014 (2)
C7	0.066 (3)	0.066 (3)	0.058 (3)	0.000 (2)	-0.007 (2)	0.010 (2)
C8	0.062 (2)	0.065 (3)	0.052 (3)	-0.0084 (18)	-0.002 (2)	0.001 (2)
C9	0.111 (4)	0.068 (3)	0.066 (3)	0.004 (3)	-0.020 (3)	0.002 (2)
C10	0.127 (4)	0.075 (3)	0.066 (3)	-0.001 (3)	-0.024 (4)	0.010 (3)
C11	0.065 (3)	0.084 (3)	0.065 (3)	-0.006 (2)	0.008 (2)	0.004 (3)
C12	0.070 (3)	0.058 (2)	0.083 (4)	-0.0047 (19)	-0.004 (3)	0.015 (2)
C13	0.074 (3)	0.060 (3)	0.059 (3)	0.001 (2)	-0.008 (2)	0.006 (2)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Br1—C11	1.906 (5)	C6—H6	0.9300
N1—C7	1.262 (6)	C7—C8	1.452 (7)
N1—C1	1.424 (6)	C7—H7	0.9300
C1—C6	1.395 (5)	C8—C9	1.378 (7)
C1—C2	1.407 (6)	C8—C13	1.399 (6)
C2—C3	1.381 (6)	C9—C10	1.383 (6)
C2—C2 <sup>i</sup>	1.506 (7)	C9—H9	0.9300
C3—C4	1.389 (6)	C10—C11	1.376 (7)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.373 (7)	C11—C12	1.358 (8)
C4—H4	0.9300	C12—C13	1.378 (6)
C5—C6	1.374 (6)	C12—H12	0.9300
C5—H5	0.9300	C13—H13	0.9300
C7—N1—C1	120.7 (4)	N1—C7—H7	118.2
C6—C1—C2	120.0 (4)	C8—C7—H7	118.2
C6—C1—N1	120.8 (4)	C9—C8—C13	118.6 (4)

## supplementary materials

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C2—C1—N1	119.2 (3)	C9—C8—C7	120.9 (4)
C3—C2—C1	118.5 (4)	C13—C8—C7	120.5 (5)
C3—C2—C2 <sup>i</sup>	120.2 (4)	C8—C9—C10	121.1 (4)
C1—C2—C2 <sup>i</sup>	121.2 (4)	C8—C9—H9	119.5
C2—C3—C4	121.0 (4)	C10—C9—H9	119.5
C2—C3—H3	119.5	C9—C10—C11	118.7 (5)
C4—C3—H3	119.5	C9—C10—H10	120.7
C5—C4—C3	120.1 (4)	C11—C10—H10	120.7
C5—C4—H4	120.0	C12—C11—C10	121.6 (5)
C3—C4—H4	120.0	C12—C11—Br1	119.4 (4)
C6—C5—C4	120.3 (4)	C10—C11—Br1	119.0 (4)
C6—C5—H5	119.8	C11—C12—C13	119.8 (5)
C4—C5—H5	119.8	C11—C12—H12	120.1
C5—C6—C1	120.1 (4)	C13—C12—H12	120.1
C5—C6—H6	119.9	C12—C13—C8	120.1 (5)
C1—C6—H6	119.9	C12—C13—H13	119.9
N1—C7—C8	123.6 (5)	C8—C13—H13	119.9
C7—N1—C1—C6	48.5 (6)	C1—N1—C7—C8	-175.6 (4)
C7—N1—C1—C2	-135.0 (5)	N1—C7—C8—C9	-169.2 (5)
C6—C1—C2—C3	-1.4 (6)	N1—C7—C8—C13	10.8 (7)
N1—C1—C2—C3	-177.9 (4)	C13—C8—C9—C10	-2.8 (8)
C6—C1—C2—C2 <sup>i</sup>	175.4 (3)	C7—C8—C9—C10	177.2 (5)
N1—C1—C2—C2 <sup>i</sup>	-1.1 (5)	C8—C9—C10—C11	0.6 (8)
C1—C2—C3—C4	1.6 (6)	C9—C10—C11—C12	2.1 (7)
C2 <sup>i</sup> —C2—C3—C4	-175.3 (4)	C9—C10—C11—Br1	-179.0 (4)
C2—C3—C4—C5	0.2 (8)	C10—C11—C12—C13	-2.7 (7)
C3—C4—C5—C6	-2.2 (8)	Br1—C11—C12—C13	178.5 (3)
C4—C5—C6—C1	2.4 (7)	C11—C12—C13—C8	0.4 (7)
C2—C1—C6—C5	-0.6 (7)	C9—C8—C13—C12	2.2 (7)
N1—C1—C6—C5	175.9 (4)	C7—C8—C13—C12	-177.8 (4)

Symmetry codes: (i)  $-x+2, -y+3, z$ .

Fig. 1

