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## Structure Reports

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# *N,N'*-Bis(3-bromobenzylidene)ethane-1,2-diamine

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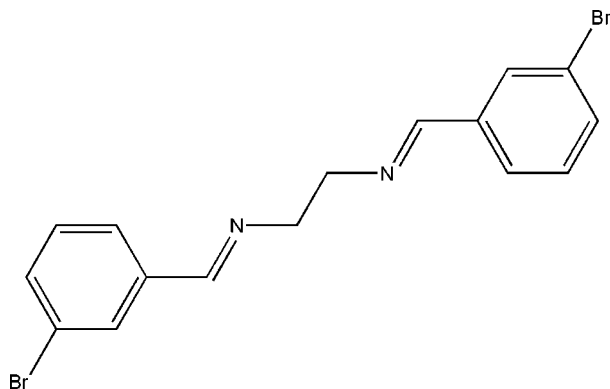
Received 30 June 2008; accepted 8 July 2008

 Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.029;  $wR$  factor = 0.066; data-to-parameter ratio = 32.1.

The molecule of the title Schiff base compound,  $\text{C}_{16}\text{H}_{14}\text{Br}_2\text{N}_2$ , lies across a crystallographic inversion centre. The  $\text{C}=\text{N}$  bond adopts a *trans* configuration. The imino group is coplanar with the benzene ring. Within the molecule, the planar units are parallel, but extend in opposite directions from the dimethylene bridge. The interesting feature of the structure is the weak  $\text{Br}\cdots\text{Br}$  interaction [ $3.7501(2)$  Å] linking the molecules into chains along the  $c$  axis. These chains are stacked along the  $b$  axis.

## Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see, for example: Fun, Kargar & Kia (2008); Fun, Kia & Kargar (2008); Fun, Mirkhani *et al.* (2008); Calligaris & Randaccio, (1987). For information on Schiff base complexes and their applications, see, for example: Kia, Mirkhani, Harkema & van Hummel (2007); Kia, Mirkhani, Kalman & Deak (2007); Pal *et al.* (2005); Hou *et al.* (2001); Ren *et al.* (2002).


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## Experimental

## Crystal data

$\text{C}_{16}\text{H}_{14}\text{Br}_2\text{N}_2$	$V = 736.32(2)$ Å <sup>3</sup>
$M_r = 394.11$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.2578(1)$ Å	$\mu = 5.50$ mm <sup>-1</sup>
$b = 4.6549(1)$ Å	$T = 100.0(1)$ K
$c = 25.3272(5)$ Å	$0.57 \times 0.22 \times 0.17$ mm
$\beta = 93.592(1)^\circ$	

## Data collection

Bruker SMART APEXII CCD area-detector diffractometer	24876 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	3822 independent reflections
$T_{\min} = 0.125$ , $T_{\max} = 0.393$	2975 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.033$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.066$	
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.69$ e Å <sup>-3</sup>
3822 reflections	$\Delta\rho_{\text{min}} = -0.61$ e Å <sup>-3</sup>
119 parameters	

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: AT2587).

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- Bruker (2005). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Calligaris, M. & Randaccio, L. (1987). *Comprehensive Coordination Chemistry*, Vol. 2, edited by G. Wilkinson, pp. 715–738. London: Pergamon.
- Fun, H.-K., Kargar, H. & Kia, R. (2008). *Acta Cryst.* **E64**, o1308.
- Fun, H.-K., Kia, R. & Kargar, H. (2008). *Acta Cryst.* **E64**, o1335.
- Fun, H.-K., Mirkhani, V., Kia, R. & Vartooni, A. R. (2008). *Acta Cryst.* **E64**, o1374–o1375.
- Hou, B., Friedman, N., Ruhman, S., Sheves, M. & Ottolenghi, M. (2001). *J. Phys. Chem. B*, **105**, 7042–7048.
- Kia, R., Mirkhani, V., Harkema, S. & van Hummel, G. J. (2007). *Inorg. Chim. Acta*, **360**, 3369–3375.
- Kia, R., Mirkhani, V., Kalman, A. & Deak, A. (2007). *Polyhedron*, **26**, 1711–1716.
- Pal, S., Barik, A. K., Gupta, S., Hazra, A., Kar, S. K., Peng, S.-M., Lee, G.-H., Butcher, R. J., El Fallah, M. S. & Ribas, J. (2005). *Inorg. Chem.* **44**, 3880–3889.
- Ren, S., Wang, R., Komatsu, K., Bonaz-Krause, P., Zyrianov, Y., McKenna, C. E., Csipke, C., Tokes, Z. A. & Lien, E. J. (2002). *J. Med. Chem.* **45**, 410–419.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

## supporting information

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

Schiff bases are one of most prevalent mixed-donor ligands in the field of coordination chemistry. Schiff bases have been used widely as ligands in the formation of transition metal complexes. Many such complexes have been structurally characterized, but only a relatively small number of free Schiff base ligands have been characterized (Calligaris & Randaccio, 1987). There has been growing interest in Schiff base ligands, mainly because of their wide application in the field of biochemistry, synthesis, and catalysis (Kia *et al.*, 2007a,b; Pal *et al.*, 2005; Hou *et al.*, 2001; Ren *et al.*, 2002). As an extension of our work (Fun *et al.*, 2008a,b,c) on the structural characterization of Schiff base compounds, the title compound (I), is reported here.

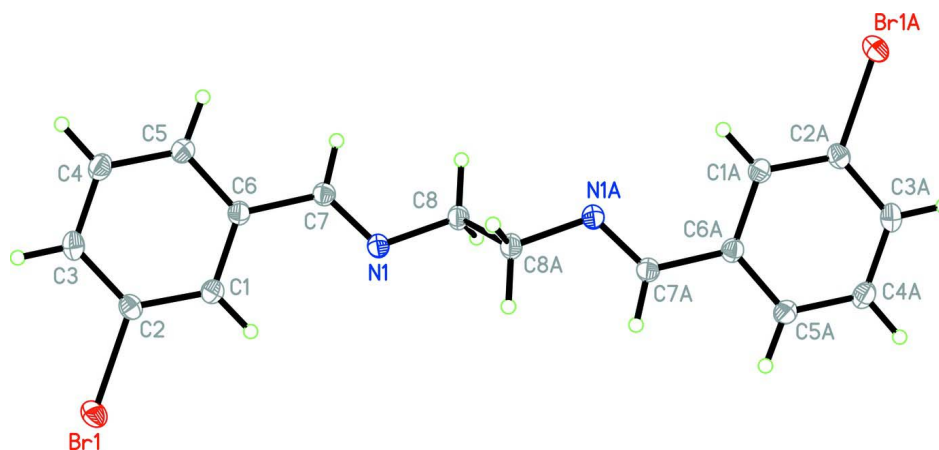
The molecule of the title compound, (I), (Fig. 1), lies across a crystallographic inversion centre. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The asymmetric unit is composed of one-half of the molecule. The C=N bond adopts a *trans* configuration. The imino group is coplanar with the benzene ring. Within the molecule, the planar units are parallel, but extend in opposite directions from the methylene bridge. The interesting feature of the crystal structure is the weak Br...Br [symmetry code: 1 - *x*, -1/2 + *y*, 1/2 - *z*] interactions with distance 3.7501 (2) Å linking the molecules into chains along the *c* axis. These chains are stacked along the *b* axis (Fig. 2).

**S2. Experimental**

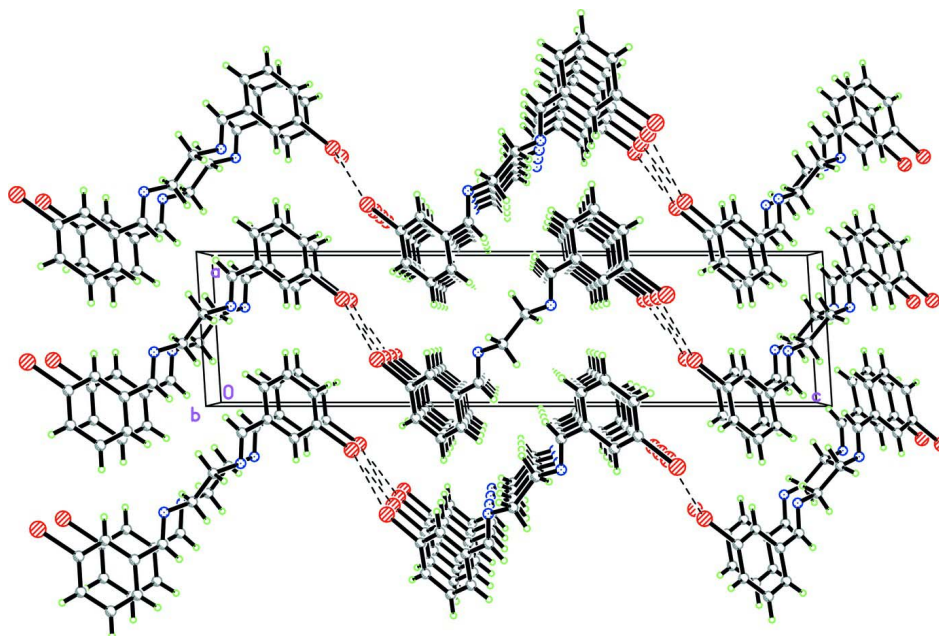
The synthetic method has been described earlier (Fun *et al.*, 2008a,b,c). Single crystals suitable for *X*-ray diffraction were obtained by evaporation of an ethanol solution at room temperature.

**S3. Refinement**

All of the H atoms were located from the difference Fourier map and freely refined.

**Figure 1**

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

**Figure 2**

The crystal packing of (I), viewed down the  $b$  axis, showing chains along the  $c$  axis and stacking of these chains along the  $b$ -axis. The Br $\cdots$ Br contacts are shown as dashed lines.

### *N,N'*-Bis(3-bromobenzylidene)ethane-1,2-diamine

#### Crystal data

$C_{16}H_{14}Br_2N_2$

$M_r = 394.11$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2ybc$

$a = 6.2578$  (1) Å

$b = 4.6549$  (1) Å

$c = 25.3272$  (5) Å

$\beta = 93.592$  (1) $^\circ$

$V = 736.32$  (2) Å $^3$

$Z = 2$

$F(000) = 388$

$D_x = 1.778$  Mg m $^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 8650 reflections

$\theta = 3.2\text{--}38.3^\circ$   
 $\mu = 5.50 \text{ mm}^{-1}$   
 $T = 100 \text{ K}$

Block, colourless  
 $0.57 \times 0.22 \times 0.17 \text{ mm}$

*Data collection*

Bruker SMART APEXII CCD area-detector  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2005)  
 $T_{\min} = 0.125$ ,  $T_{\max} = 0.393$

24876 measured reflections  
 3822 independent reflections  
 2975 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$   
 $\theta_{\max} = 37.5^\circ$ ,  $\theta_{\min} = 3.2^\circ$   
 $h = -10 \rightarrow 10$   
 $k = -7 \rightarrow 7$   
 $l = -43 \rightarrow 43$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.066$   
 $S = 1.07$   
 3822 reflections  
 119 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0244P)^2 + 0.509P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.69 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.62 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.30202 (2)	0.03394 (3)	0.278341 (6)	0.02085 (4)
N1	0.35517 (18)	0.8207 (3)	0.44274 (5)	0.0165 (2)
C1	0.2106 (2)	0.3996 (3)	0.36391 (5)	0.0158 (2)
C2	0.1288 (2)	0.1862 (3)	0.33044 (5)	0.0165 (2)
C3	-0.0766 (2)	0.0762 (3)	0.33433 (6)	0.0188 (3)
C4	-0.2013 (2)	0.1822 (3)	0.37319 (6)	0.0194 (3)
C5	-0.1226 (2)	0.3970 (3)	0.40730 (6)	0.0181 (2)
C6	0.0838 (2)	0.5062 (3)	0.40303 (5)	0.0158 (2)
C7	0.1634 (2)	0.7313 (3)	0.43991 (6)	0.0162 (2)
C8	0.4077 (2)	1.0477 (3)	0.48093 (6)	0.0171 (2)
H1	0.348 (3)	0.476 (4)	0.3617 (9)	0.021 (5)*

H3	-0.128 (3)	-0.067 (5)	0.3113 (9)	0.026 (6)*
H4	-0.344 (3)	0.115 (5)	0.3752 (8)	0.025 (5)*
H5	-0.207 (4)	0.474 (5)	0.4344 (10)	0.030 (6)*
H7	0.064 (3)	0.805 (5)	0.4623 (8)	0.019 (5)*
H8A	0.448 (3)	1.212 (5)	0.4612 (8)	0.024 (5)*
H8B	0.283 (3)	1.109 (4)	0.5004 (7)	0.014 (4)*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.02520 (7)	0.02173 (8)	0.01584 (6)	0.00015 (5)	0.00296 (4)	-0.00294 (6)
N1	0.0157 (5)	0.0168 (6)	0.0168 (5)	-0.0003 (4)	0.0000 (4)	-0.0027 (4)
C1	0.0152 (5)	0.0160 (6)	0.0160 (5)	0.0002 (4)	-0.0002 (4)	0.0008 (5)
C2	0.0188 (5)	0.0167 (6)	0.0137 (5)	0.0010 (4)	-0.0007 (4)	-0.0002 (4)
C3	0.0206 (6)	0.0168 (7)	0.0183 (6)	-0.0013 (5)	-0.0037 (4)	-0.0018 (5)
C4	0.0153 (5)	0.0196 (7)	0.0232 (7)	-0.0019 (4)	-0.0014 (5)	-0.0012 (5)
C5	0.0154 (5)	0.0184 (7)	0.0205 (6)	-0.0008 (4)	0.0012 (4)	-0.0019 (5)
C6	0.0151 (5)	0.0147 (6)	0.0173 (5)	0.0000 (4)	-0.0003 (4)	-0.0010 (4)
C7	0.0154 (5)	0.0162 (6)	0.0170 (6)	0.0007 (4)	0.0001 (4)	-0.0015 (5)
C8	0.0160 (5)	0.0170 (6)	0.0179 (6)	0.0001 (4)	-0.0018 (4)	-0.0030 (5)

*Geometric parameters (Å, °)*

Br1—C2	1.8967 (14)	C4—C5	1.391 (2)
N1—C7	1.2676 (17)	C4—H4	0.95 (2)
N1—C8	1.4564 (19)	C5—C6	1.3985 (19)
C1—C2	1.383 (2)	C5—H5	0.96 (2)
C1—C6	1.399 (2)	C6—C7	1.470 (2)
C1—H1	0.93 (2)	C7—H7	0.93 (2)
C2—C3	1.393 (2)	C8—C8 <sup>i</sup>	1.525 (3)
C3—C4	1.385 (2)	C8—H8A	0.96 (2)
C3—H3	0.93 (2)	C8—H8B	0.991 (19)
C7—N1—C8	116.70 (12)	C4—C5—H5	121.6 (14)
C2—C1—C6	118.96 (12)	C6—C5—H5	118.0 (14)
C2—C1—H1	122.9 (13)	C5—C6—C1	119.58 (13)
C6—C1—H1	118.1 (13)	C5—C6—C7	119.22 (13)
C1—C2—C3	121.90 (13)	C1—C6—C7	121.20 (12)
C1—C2—Br1	119.28 (10)	N1—C7—C6	123.50 (13)
C3—C2—Br1	118.81 (11)	N1—C7—H7	120.7 (12)
C4—C3—C2	118.88 (13)	C6—C7—H7	115.8 (12)
C4—C3—H3	121.0 (14)	N1—C8—C8 <sup>i</sup>	109.87 (15)
C2—C3—H3	120.1 (13)	N1—C8—H8A	106.8 (12)
C3—C4—C5	120.27 (13)	C8 <sup>i</sup> —C8—H8A	110.5 (12)
C3—C4—H4	119.5 (13)	N1—C8—H8B	112.9 (11)
C5—C4—H4	120.1 (13)	C8 <sup>i</sup> —C8—H8B	110.9 (11)
C4—C5—C6	120.41 (14)	H8A—C8—H8B	105.7 (17)

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C6—C1—C2—C3	0.5 (2)	C4—C5—C6—C7	-179.45 (14)
C6—C1—C2—Br1	-178.55 (10)	C2—C1—C6—C5	-0.4 (2)
C1—C2—C3—C4	-0.6 (2)	C2—C1—C6—C7	179.52 (13)
Br1—C2—C3—C4	178.42 (11)	C8—N1—C7—C6	179.20 (13)
C2—C3—C4—C5	0.7 (2)	C5—C6—C7—N1	172.14 (14)
C3—C4—C5—C6	-0.6 (2)	C1—C6—C7—N1	-7.8 (2)
C4—C5—C6—C1	0.5 (2)	C7—N1—C8—C8 <sup>i</sup>	123.51 (16)

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