

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

Hoong-Kun Fun\* and Reza Kia‡

 X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia  
 Correspondence e-mail: hkfun@usm.my

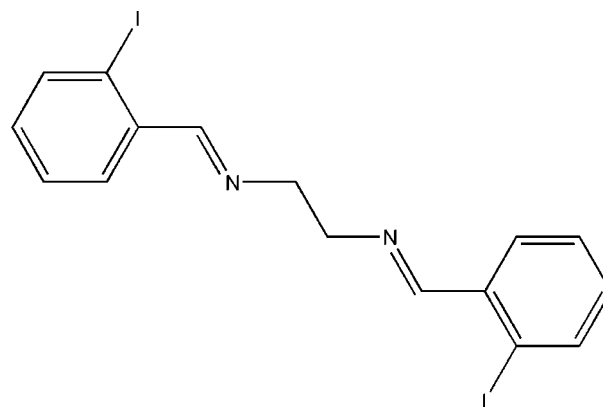
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 Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.031;  $wR$  factor = 0.074; data-to-parameter ratio = 36.8.

The molecule of the title Schiff base compound,  $\text{C}_{16}\text{H}_{14}\text{I}_2\text{N}_2$ , lies across a crystallographic inversion centre. An intramolecular  $\text{C}-\text{H}\cdots\text{I}$  hydrogen bond forms a five-membered ring, producing an  $S(5)$  ring motif. The  $\text{C}=\text{N}$  bond is coplanar with the benzene ring and adopts a *trans* configuration. Within the molecule, the planar units are parallel, but extend in opposite directions from the dimethylene bridge. An interesting feature of the crystal structure is the short  $\text{I}\cdots\text{N}$  [3.2096 (15) Å] interaction, which is significantly shorter than the sum of the van der Waals radii of these atoms. In the crystal structure, molecules are linked into one-dimensional extended chains along the  $c$  axis and also into one-dimensional extended chains along the  $b$  axis through short intermolecular  $\text{I}\cdots\text{N}$  interactions, forming two-dimensional networks parallel to the  $bc$  plane.

### Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the hydrogen bond capability of halogens, see: Brammer *et al.* (2001). For halogen–electronegative atom interactions, see: Lommerse *et al.* (1996). For related structures, see, for example: Fun, Kia & Kargar (2008); Fun, Kargar & Kia (2008); Fun, Mirkhani *et al.* (2008); Calligaris & Randaccio, (1987). For information on Schiff base ligands, their complexes and their applications, see, for example: Pal *et al.* (2005); Hou *et al.* (2001); Ren *et al.* (2002).



### Experimental

#### Crystal data

$\text{C}_{16}\text{H}_{14}\text{I}_2\text{N}_2$	$V = 813.44$ (4) Å <sup>3</sup>
$M_r = 488.09$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 12.1820$ (4) Å	$\mu = 3.86$ mm <sup>-1</sup>
$b = 4.5978$ (1) Å	$T = 100.0$ (1) K
$c = 14.5664$ (4) Å	$0.51 \times 0.14 \times 0.02$ mm
$\beta = 94.424$ (2)°	

#### Data collection

Bruker SMART APEXII CCD area-detector diffractometer	24819 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	4235 independent reflections
$T_{\min} = 0.244$ , $T_{\max} = 0.916$	3466 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.044$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	115 parameters
$wR(F^2) = 0.074$	All H-atom parameters refined
$S = 1.16$	$\Delta\rho_{\text{max}} = 1.89$ e Å <sup>-3</sup>
4235 reflections	$\Delta\rho_{\text{min}} = -1.74$ e Å <sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C7}-\text{H7}\cdots\text{I1}$	0.93 (3)	2.87 (3)	3.3880 (18)	116 (2)

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: AT2623).

‡ Additional correspondence author, e-mail: zsrkk@yahoo.com.

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**supplementary materials**

*Acta Cryst.* (2008). E64, o1870-o1871 [ doi:10.1107/S1600536808027608 ]

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

H.-K. Fun and R. Kia

### 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 (Pal *et al.*, 2005; Hou *et al.*, 2001; Ren *et al.*, 2002). As an extension of our work (Fun, Kia & Kargar 2008; Fun, Kargar & Kia 2008; Fun, Mirkhani *et al.* 2008) 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). An intramolecular C—H...I hydrogen bond (Brammer *et al.* 2001) forms a five-membered ring, producing an *S*(5) ring motif (Bernstein *et al.*, 1995) (Table 1). The asymmetric unit of the compound is composed of one-half of the molecule. The C=N bond is coplanar with the benzene ring and adopts a *trans* configuration. 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 short I...N [3.2096 (15) Å] interactions (Lommerse *et al.* 1996), which is significantly shorter than the sum of the van der Waals radii of the relevant atoms. In the crystal structure, molecules are linked into 1-D extended chains along the *c* axis and are also into 1-D extended chains along the *b* axis through short intermolecular I...N interactions forming 2-D networks (Fig. 2 & 3) which are parallel to the *bc* plane.

### Experimental

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

### Refinement

All of the H atoms were located from the difference Fourier map and freely refined. The highest peak is located 0.61 Å from C5 and the deepest hole is located 0.63 Å from I1.

### Figures

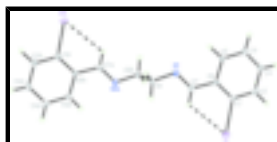


Fig. 1. The molecular structure of (I) with atom labels and 50% probability ellipsoids for non-H atoms [symmetry code for A:  $-x, 1 - y, -z$ ].

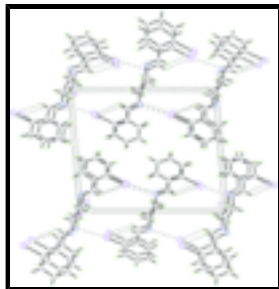


Fig. 2. The crystal packing of (I), viewed down the *b* axis, showing 1-D extended chains along the *c* axis. Intra- and intermolecular interactions are shown as dashed lines.

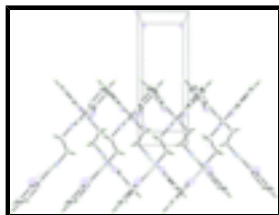


Fig. 3. The crystal packing of (I), viewed down the *c*-axis showing 1-D extended chains along the *b*-axis. Intra and intermolecular interactions are shown as dashed lines.

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

#### *Crystal data*

$C_{16}H_{14}I_2N_2$

$M_r = 488.09$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.1820$  (4) Å

$b = 4.59780$  (10) Å

$c = 14.5664$  (4) Å

$\beta = 94.424$  (2)°

$V = 813.44$  (4) Å<sup>3</sup>

$Z = 2$

$F_{000} = 460$

$D_x = 1.993$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 7125 reflections

$\theta = 2.8$ – $38.9$ °

$\mu = 3.86$  mm<sup>-1</sup>

$T = 100.0$  (1) K

Plate, colourless

$0.51 \times 0.14 \times 0.02$  mm

#### *Data collection*

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 100.0$ (1) K

$\varphi$  and  $\omega$  scans

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

$T_{\min} = 0.244$ ,  $T_{\max} = 0.917$

24819 measured reflections

4235 independent reflections

3466 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.044$

$\theta_{\text{max}} = 37.5$ °

$\theta_{\text{min}} = 1.7$ °

$h = -19 \rightarrow 20$

$k = -7 \rightarrow 7$

$l = -24 \rightarrow 24$

#### *Refinement*

Refinement on  $F^2$

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.031$$

$$wR(F^2) = 0.074$$

$$S = 1.16$$

4235 reflections

115 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2 + 0.1458P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 1.89 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.74 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

### Special details

**Experimental.** The low-temperature data was collected with the Oxford Cryosystem 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
II	0.219874 (11)	0.02241 (3)	0.328844 (8)	0.01748 (5)
N1	0.14038 (12)	0.3597 (4)	0.03053 (10)	0.0158 (3)
C1	0.29294 (15)	-0.0875 (4)	0.20654 (12)	0.0146 (3)
C2	0.37697 (15)	-0.2923 (4)	0.21484 (12)	0.0167 (3)
C3	0.43327 (16)	-0.3648 (5)	0.13879 (12)	0.0178 (3)
C4	0.40419 (16)	-0.2345 (5)	0.05442 (13)	0.0185 (4)
C5	0.32010 (17)	-0.0341 (4)	0.04599 (13)	0.0160 (3)
H5	0.2813	-0.0060	-0.0162	0.019*
C6	0.26222 (16)	0.0456 (4)	0.12178 (13)	0.0135 (3)
C7	0.17398 (15)	0.2630 (4)	0.10940 (12)	0.0147 (3)
C8	0.05021 (16)	0.5681 (4)	0.02623 (13)	0.0160 (3)
H8B	0.0723 (18)	0.725 (5)	-0.0044 (15)	0.016 (6)*
H4	0.4465 (18)	-0.278 (5)	0.0004 (15)	0.017 (6)*
H8A	0.0293 (19)	0.629 (6)	0.0866 (16)	0.021 (6)*
H2	0.3998 (18)	-0.387 (6)	0.2766 (16)	0.015 (6)*
H7	0.144 (2)	0.323 (7)	0.1634 (18)	0.034 (7)*
H3	0.488 (3)	-0.507 (5)	0.148 (2)	0.031 (9)*

## supplementary materials

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
II	0.02070 (7)	0.01995 (7)	0.01214 (6)	0.00044 (4)	0.00353 (4)	0.00024 (4)
N1	0.0157 (7)	0.0154 (8)	0.0163 (6)	0.0034 (6)	0.0008 (5)	0.0003 (5)
C1	0.0162 (8)	0.0149 (8)	0.0127 (7)	-0.0002 (6)	0.0015 (6)	-0.0012 (6)
C2	0.0184 (8)	0.0151 (8)	0.0160 (7)	0.0003 (7)	-0.0026 (6)	0.0001 (6)
C3	0.0155 (8)	0.0175 (9)	0.0201 (8)	0.0046 (7)	-0.0003 (6)	-0.0004 (7)
C4	0.0192 (8)	0.0187 (9)	0.0178 (8)	0.0049 (7)	0.0033 (6)	-0.0019 (6)
C5	0.0188 (8)	0.0161 (8)	0.0132 (7)	0.0018 (6)	0.0028 (6)	-0.0030 (6)
C6	0.0151 (8)	0.0132 (8)	0.0123 (7)	0.0001 (6)	0.0011 (6)	-0.0011 (6)
C7	0.0147 (7)	0.0129 (8)	0.0168 (7)	0.0008 (6)	0.0021 (6)	-0.0011 (6)
C8	0.0151 (8)	0.0149 (8)	0.0178 (8)	0.0038 (6)	0.0009 (6)	0.0002 (6)

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

II—C1	2.1133 (17)	C4—C5	1.376 (3)
N1—C7	1.270 (2)	C4—H4	0.99 (2)
N1—C8	1.455 (2)	C5—C6	1.404 (3)
C1—C2	1.389 (3)	C5—H5	0.9975
C1—C6	1.403 (3)	C6—C7	1.469 (3)
C2—C3	1.388 (3)	C7—H7	0.93 (3)
C2—H2	1.02 (2)	C8—C8 <sup>i</sup>	1.526 (4)
C3—C4	1.388 (3)	C8—H8B	0.90 (2)
C3—H3	0.94 (3)	C8—H8A	0.98 (2)
C7—N1—C8	117.33 (16)	C4—C5—H5	117.7
C2—C1—C6	121.13 (17)	C6—C5—H5	116.7
C2—C1—II	116.36 (13)	C1—C6—C5	117.54 (18)
C6—C1—II	122.46 (14)	C1—C6—C7	123.23 (17)
C3—C2—C1	119.97 (17)	C5—C6—C7	119.23 (17)
C3—C2—H2	118.9 (13)	N1—C7—C6	122.09 (17)
C1—C2—H2	121.1 (13)	N1—C7—H7	122.8 (17)
C2—C3—C4	119.67 (18)	C6—C7—H7	115.1 (17)
C2—C3—H3	116 (2)	N1—C8—C8 <sup>i</sup>	108.9 (2)
C4—C3—H3	124 (2)	N1—C8—H8B	107.1 (14)
C5—C4—C3	120.34 (18)	C8 <sup>i</sup> —C8—H8B	109.9 (14)
C5—C4—H4	119.5 (13)	N1—C8—H8A	113.6 (15)
C3—C4—H4	120.1 (13)	C8 <sup>i</sup> —C8—H8A	108.3 (14)
C4—C5—C6	121.35 (18)	H8B—C8—H8A	109 (2)
C6—C1—C2—C3	1.1 (3)	II—C1—C6—C7	-2.6 (3)
II—C1—C2—C3	-176.50 (15)	C4—C5—C6—C1	-0.2 (3)
C1—C2—C3—C4	-0.9 (3)	C4—C5—C6—C7	179.31 (18)
C2—C3—C4—C5	0.1 (3)	C8—N1—C7—C6	178.18 (17)
C3—C4—C5—C6	0.5 (3)	C1—C6—C7—N1	-173.04 (19)
C2—C1—C6—C5	-0.5 (3)	C5—C6—C7—N1	7.4 (3)
II—C1—C6—C5	176.90 (13)	C7—N1—C8—C8 <sup>i</sup>	-114.6 (2)

C2—C1—C6—C7 179.94 (17)

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

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

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C7—H7 $\cdots$ I1	0.93 (3)	2.87 (3)	3.3880 (18)	116 (2)



Fig. 1

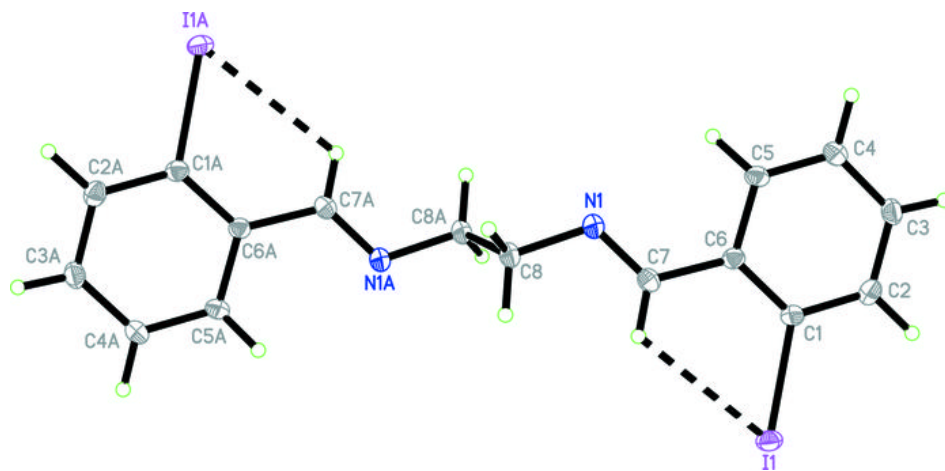


Fig. 2

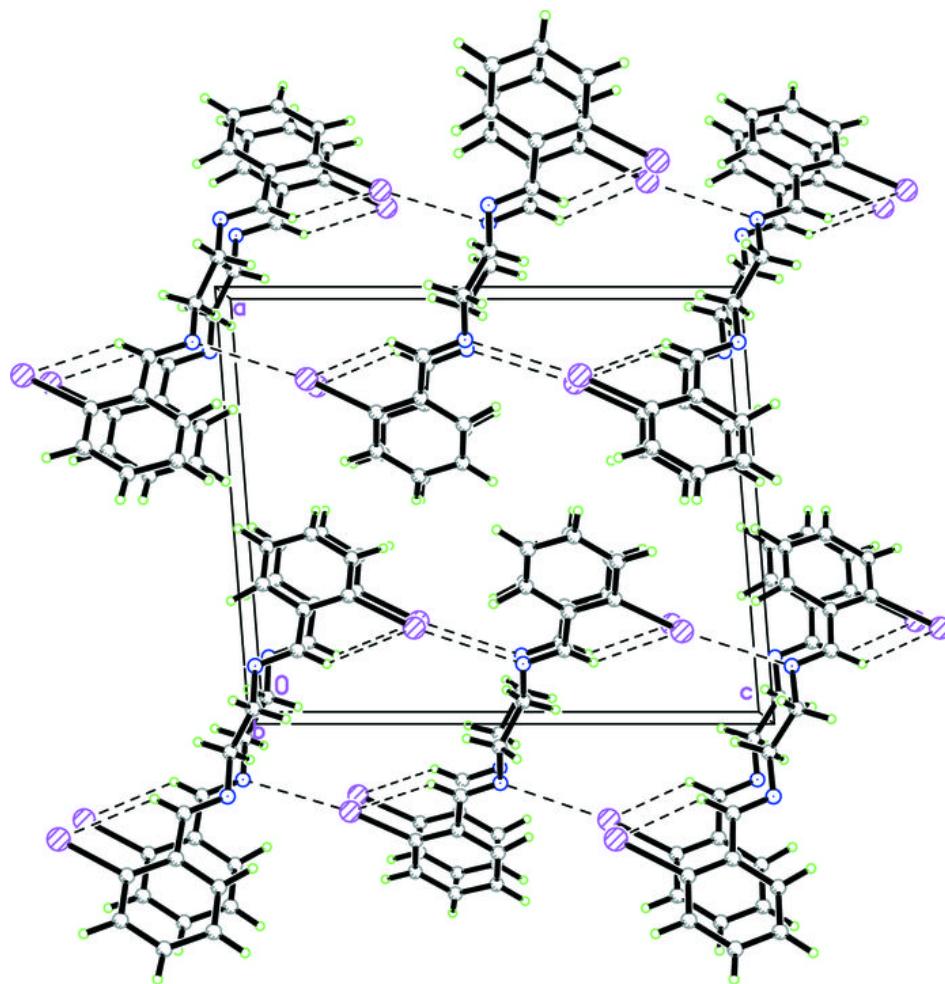


Fig. 3

