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## Structure Reports

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**(1*E*,2*E*)-1,2-Bis[1-(3-chlorophenyl)ethylidene]hydrazine**Hoong-Kun Fun,<sup>a,\*</sup> Patcharaporn Jansrisewangwong,<sup>b</sup> Chatchanok Karalai<sup>b</sup> and Suchada Chantrapromma<sup>b,§</sup><sup>a</sup>X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and <sup>b</sup>Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

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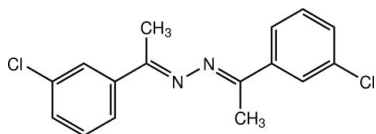
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Key indicators: single-crystal X-ray study;  $T = 297$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.051;  $wR$  factor = 0.180; data-to-parameter ratio = 21.4.

The title molecule,  $\text{C}_{16}\text{H}_{14}\text{Cl}_2\text{N}_2$ , lies on an inversion center. The dihedral angle between the symmetry-related benzene rings is  $0.02$  ( $11^\circ$ ). The mean plane of the central  $\text{C}(\text{methyl})-\text{C}=\text{N}-\text{N}=\text{C}(\text{methyl})$  unit forms a dihedral angle of  $5.57$  ( $12^\circ$ ) with the symmetry-unique benzene ring.

## Related literature

For background to the biological activity and fluorescent properties of hydrazones, see: Li *et al.* (2009); Qin *et al.* (2009). For related structures see: Chantrapromma *et al.* (2010); Fun *et al.* (2010, 2011); Jansrisewangwong *et al.* (2010); Nilwanna *et al.* (2011). For standard bond-length data, see: Allen *et al.* (1987).



## Experimental

## Crystal data

$\text{C}_{16}\text{H}_{14}\text{Cl}_2\text{N}_2$   
 $M_r = 305.19$   
 Monoclinic,  $P2_1/c$   
 $a = 10.7796$  (18) Å

$b = 5.2725$  (9) Å  
 $c = 15.3427$  (18) Å  
 $\beta = 121.540$  (8) $^\circ$   
 $V = 743.2$  (2) Å<sup>3</sup>

$Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.43$  mm<sup>-1</sup>

$T = 297$  K  
 $0.31 \times 0.15 \times 0.11$  mm

## Data collection

Bruker APEX DUO CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2009)  
 $T_{\min} = 0.880$ ,  $T_{\max} = 0.957$

7616 measured reflections  
 1970 independent reflections  
 1469 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$   
 $wR(F^2) = 0.180$   
 $S = 1.09$   
 1970 reflections

92 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.46$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.41$  e Å<sup>-3</sup>

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; 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, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5380).

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## supporting information

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**(1*E*,2*E*)-1,2-Bis[1-(3-chlorophenyl)ethylidene]hydrazine**

**Hoong-Kun Fun, Patcharaporn Jansrisewangwong, Chatchanok Karalai and Suchada Chantrapromma**

**S1. Comment**

Due to the interesting applications of hydrazones with respect to their antibacterial, antiviral and antioxidant (Li *et al.*, 2009) as well as fluorescent properties (Qin *et al.*, 2009), we have synthesized a series of hydrazones in order to study these activities and have reported some of these crystal structures (Chantrapromma *et al.*, 2010; Fun *et al.*, 2010,2011; Jansrisewangwong *et al.*, 2010; Nilwanna *et al.*, 2011). As part of our on-going research on the medicinal chemistry of hydrazones, the title compound (I) was synthesized and its biological activities will be reported elsewhere. However, it does not possess fluorescent property.

The molecular structure of (I) is shown in Fig. 1. The asymmetric unit contains half a molecule and the complete molecule is generated by a crystallographic inversion center at  $-x, 1-y, 2-z$ . The molecule exists in an *E,E* configuration with respect to the two ethylidene C=N bonds [1.279 (3) Å] and the torsion angle N1A–N1–C7–C1 = 179.8 (2)°. The molecule is essentially planar with the dihedral angle between the two benzene rings of 0.02 (11)°. The diethylidenehydrazine moiety (C7/C8/N1/N1A/C7A/C8A) is planar with the *r.m.s* of 0.0015 (2) Å. This central C(methyl)—C=N—N=C—C(methyl) mean plane makes the dihedral angle of 5.57 (12)° with the adjacent benzene rings. The bond distances are within the normal range (Allen *et al.*, 1987) and are comparable with the related structures (Chantrapromma *et al.*, 2010; Fun *et al.*, 2010; 2011; Jansrisewangwong *et al.*, 2010; Nilwanna *et al.*, 2011).

Although no classical hydrogen bonds or weak interactions were observed in the crystal structure, the crystal packing is shown in Fig. 2.

**S2. Experimental**

The title compound (I) was synthesized by mixing a solution (1:2 molar ratio) of hydrazine hydrate (0.10 ml, 2 mmol) and 3-chloroacetophenone (0.50 ml, 4 mmol) in ethanol (20 ml). The resulting solution was refluxed for 7 h, yielding the yellow crystalline solid. The resultant solid was filtered off and washed with methanol. Yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystallized from acetone by slow evaporation of the solvent at room temperature over several days, Mp. 356–358 K.

**S3. Refinement**

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with  $d(\text{C-H}) = 0.93$  Å for aromatic and 0.96 Å for CH<sub>3</sub> atoms. The  $U_{\text{iso}}$  values were constrained to be  $1.5U_{\text{eq}}$  of the carrier atom for methyl H atoms and  $1.2U_{\text{eq}}$  for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 1.92 Å from H8B and the deepest hole is located at 0.70 Å from C11.

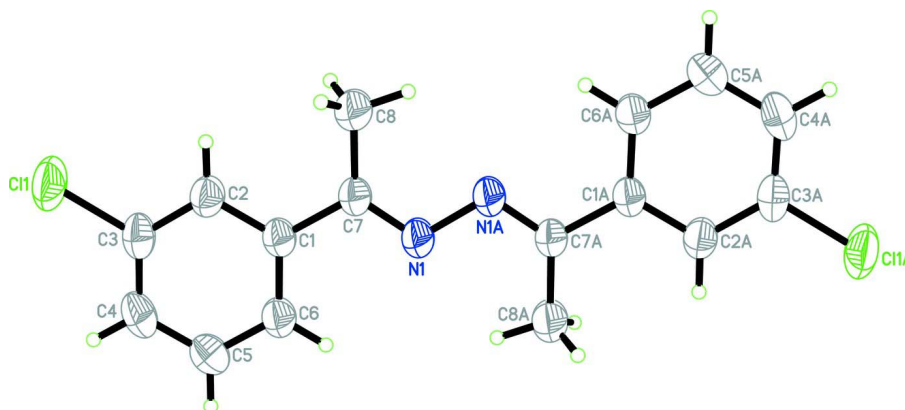


Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids. Atoms with suffix A were generated by symmetry code  $-x, 1-y, 2-z$ .

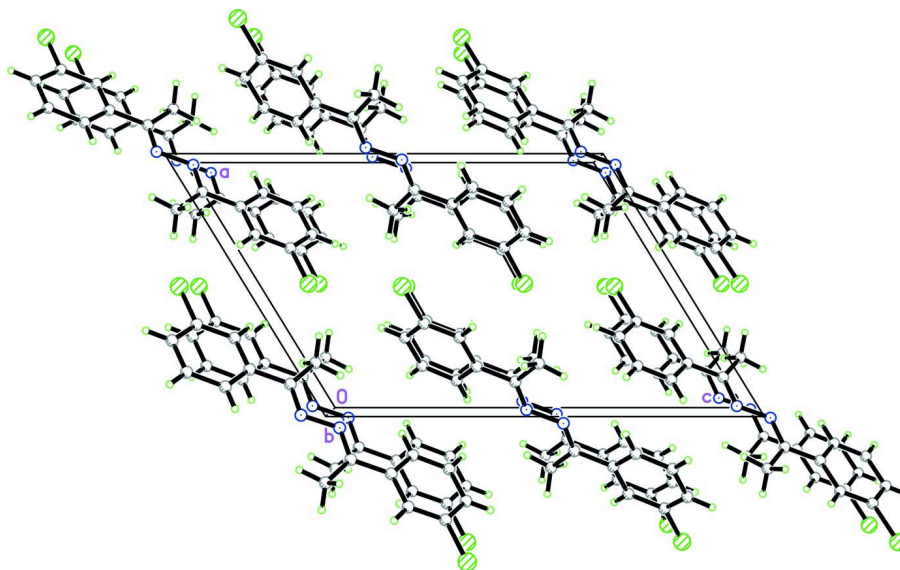


Figure 2

The crystal packing of (I). No classical hydrogen bonds nor weak interactions are observed in the crystal structure

### (1*E*,2*E*)-1,2-Bis[1-(3-chlorophenyl)ethylidene]hydrazine

#### Crystal data

$C_{16}H_{14}Cl_2N_2$

$M_r = 305.19$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P 2_1/c$

$a = 10.7796$  (18) Å

$b = 5.2725$  (9) Å

$c = 15.3427$  (18) Å

$\beta = 121.540$  (8)°

$V = 743.2$  (2) Å<sup>3</sup>

$Z = 2$

$F(000) = 316$

$D_x = 1.364$  Mg m<sup>-3</sup>

Melting point = 356–358 K

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

Cell parameters from 1970 reflections

$\theta = 2.2$ – $29.0$ °

$\mu = 0.43$  mm<sup>-1</sup>

$T = 297$  K

Block, yellow

$0.31 \times 0.15 \times 0.11$  mm

*Data collection*

Bruker APEX DUO CCD area-detector  
diffractometer  
Radiation source: sealed tube  
Graphite monochromator  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan  
(*SADABS*; Bruker, 2009)  
 $T_{\min} = 0.880$ ,  $T_{\max} = 0.957$

7616 measured reflections  
1970 independent reflections  
1469 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$   
 $\theta_{\max} = 29.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$   
 $h = -14 \rightarrow 14$   
 $k = -7 \rightarrow 6$   
 $l = -20 \rightarrow 20$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.051$   
 $wR(F^2) = 0.180$   
 $S = 1.09$   
1970 reflections  
92 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0951P)^2 + 0.2508P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.46 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.41 \text{ e } \text{\AA}^{-3}$

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|--------------|----------------------------------|
| Cl1 | 0.49474 (7) | 0.23824 (14) | 0.85934 (6)  | 0.0708 (3)                       |
| N1  | 0.0247 (2)  | 0.5365 (4)   | 0.96753 (14) | 0.0543 (5)                       |
| C1  | 0.1907 (2)  | 0.4809 (4)   | 0.91400 (14) | 0.0413 (4)                       |
| C2  | 0.3036 (2)  | 0.3431 (4)   | 0.91724 (16) | 0.0460 (5)                       |
| H2A | 0.3438      | 0.2045       | 0.9607       | 0.055*                           |
| C3  | 0.3550 (2)  | 0.4148 (4)   | 0.85517 (16) | 0.0478 (5)                       |
| C4  | 0.2988 (2)  | 0.6198 (5)   | 0.78983 (17) | 0.0527 (6)                       |
| H4A | 0.3353      | 0.6655       | 0.7490       | 0.063*                           |
| C5  | 0.1869 (3)  | 0.7552 (4)   | 0.78659 (19) | 0.0535 (6)                       |
| H5A | 0.1476      | 0.8939       | 0.7430       | 0.064*                           |
| C6  | 0.1324 (2)  | 0.6875 (4)   | 0.84733 (16) | 0.0468 (5)                       |
| H6A | 0.0565      | 0.7801       | 0.8438       | 0.056*                           |
| C7  | 0.1350 (2)  | 0.4107 (4)   | 0.98120 (15) | 0.0426 (4)                       |
| C8  | 0.2087 (3)  | 0.2056 (6)   | 1.0585 (2)   | 0.0711 (8)                       |
| H8A | 0.1613      | 0.1850       | 1.0964       | 0.107*                           |
| H8B | 0.3088      | 0.2501       | 1.1045       | 0.107*                           |

H8C                    0.2034                    0.0496                    1.0245                    0.107\*

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

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C11 | 0.0653 (4)  | 0.0831 (5)  | 0.0902 (5)  | 0.0089 (3)   | 0.0590 (4)  | -0.0068 (3)  |
| N1  | 0.0601 (11) | 0.0663 (12) | 0.0567 (10) | 0.0214 (9)   | 0.0445 (9)  | 0.0206 (9)   |
| C1  | 0.0438 (10) | 0.0463 (10) | 0.0414 (9)  | 0.0004 (8)   | 0.0276 (8)  | -0.0015 (8)  |
| C2  | 0.0470 (10) | 0.0503 (11) | 0.0487 (10) | 0.0038 (9)   | 0.0306 (9)  | -0.0020 (9)  |
| C3  | 0.0459 (10) | 0.0571 (13) | 0.0518 (11) | -0.0052 (9)  | 0.0335 (9)  | -0.0125 (9)  |
| C4  | 0.0611 (13) | 0.0609 (14) | 0.0530 (11) | -0.0129 (11) | 0.0415 (11) | -0.0081 (10) |
| C5  | 0.0604 (13) | 0.0577 (14) | 0.0507 (12) | 0.0002 (10)  | 0.0347 (11) | 0.0068 (9)   |
| C6  | 0.0466 (10) | 0.0547 (12) | 0.0473 (10) | 0.0046 (9)   | 0.0302 (9)  | 0.0048 (9)   |
| C7  | 0.0471 (10) | 0.0470 (11) | 0.0435 (9)  | 0.0054 (8)   | 0.0305 (8)  | 0.0023 (8)   |
| C8  | 0.0766 (17) | 0.0858 (19) | 0.0761 (16) | 0.0374 (15)  | 0.0574 (15) | 0.0370 (15)  |

*Geometric parameters (Å, °)*

|                       |              |                           |             |
|-----------------------|--------------|---------------------------|-------------|
| C11—C3                | 1.743 (2)    | C4—C5                     | 1.380 (3)   |
| N1—C7                 | 1.279 (3)    | C4—H4A                    | 0.9300      |
| N1—N1 <sup>i</sup>    | 1.406 (3)    | C5—C6                     | 1.383 (3)   |
| C1—C2                 | 1.395 (3)    | C5—H5A                    | 0.9300      |
| C1—C6                 | 1.399 (3)    | C6—H6A                    | 0.9300      |
| C1—C7                 | 1.486 (3)    | C7—C8                     | 1.491 (3)   |
| C2—C3                 | 1.382 (3)    | C8—H8A                    | 0.9600      |
| C2—H2A                | 0.9300       | C8—H8B                    | 0.9600      |
| C3—C4                 | 1.380 (3)    | C8—H8C                    | 0.9600      |
| C7—N1—N1 <sup>i</sup> | 113.9 (2)    | C4—C5—H5A                 | 119.6       |
| C2—C1—C6              | 118.78 (18)  | C6—C5—H5A                 | 119.6       |
| C2—C1—C7              | 120.47 (19)  | C5—C6—C1                  | 120.5 (2)   |
| C6—C1—C7              | 120.74 (18)  | C5—C6—H6A                 | 119.8       |
| C3—C2—C1              | 119.3 (2)    | C1—C6—H6A                 | 119.8       |
| C3—C2—H2A             | 120.3        | N1—C7—C1                  | 115.82 (18) |
| C1—C2—H2A             | 120.3        | N1—C7—C8                  | 124.68 (19) |
| C4—C3—C2              | 122.2 (2)    | C1—C7—C8                  | 119.49 (18) |
| C4—C3—C11             | 119.20 (16)  | C7—C8—H8A                 | 109.5       |
| C2—C3—C11             | 118.63 (18)  | C7—C8—H8B                 | 109.5       |
| C5—C4—C3              | 118.4 (2)    | H8A—C8—H8B                | 109.5       |
| C5—C4—H4A             | 120.8        | C7—C8—H8C                 | 109.5       |
| C3—C4—H4A             | 120.8        | H8A—C8—H8C                | 109.5       |
| C4—C5—C6              | 120.9 (2)    | H8B—C8—H8C                | 109.5       |
| C6—C1—C2—C3           | 0.3 (3)      | C2—C1—C6—C5               | -0.6 (3)    |
| C7—C1—C2—C3           | -178.95 (19) | C7—C1—C6—C5               | 178.6 (2)   |
| C1—C2—C3—C4           | 0.2 (3)      | N1 <sup>i</sup> —N1—C7—C1 | 179.8 (2)   |
| C1—C2—C3—C11          | -179.32 (16) | N1 <sup>i</sup> —N1—C7—C8 | -0.5 (4)    |
| C2—C3—C4—C5           | -0.4 (3)     | C2—C1—C7—N1               | -175.2 (2)  |

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|              |             |             |            |
|--------------|-------------|-------------|------------|
| C11—C3—C4—C5 | 179.15 (17) | C6—C1—C7—N1 | 5.6 (3)    |
| C3—C4—C5—C6  | 0.0 (4)     | C2—C1—C7—C8 | 5.1 (3)    |
| C4—C5—C6—C1  | 0.5 (4)     | C6—C1—C7—C8 | -174.1 (2) |

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