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## Structure Reports

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## 1-(2,4-Dichlorobenzylidene)-4-ethylthiosemicarbazide

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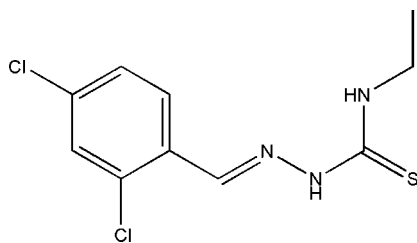
Received 31 August 2010; accepted 6 September 2010

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;  $R$  factor = 0.060;  $wR$  factor = 0.187; data-to-parameter ratio = 18.7.

The title compound,  $\text{C}_{10}\text{H}_{11}\text{Cl}_2\text{N}_3\text{S}$ , was prepared by the reaction of 4-ethylthiosemicarbazide and 2,4-dichlorobenzaldehyde. It is approximately planar, the dihedral angle between the benzene ring and the thiourea unit being  $8.43(18)^\circ$ . In the crystal, inversion dimers linked by pairs of  $\text{N}-\text{H}\cdots\text{S}$  hydrogen bonds generate  $R_2^2(8)$  loops.

## Related literature

For background to Schiff bases, see: Casas *et al.* (2000). For a related structure, see: Li & Jian (2010).



## Experimental

## Crystal data

$\text{C}_{10}\text{H}_{11}\text{Cl}_2\text{N}_3\text{S}$   
 $M_r = 276.18$   
Monoclinic,  $P2_1/n$   
 $a = 5.4339(11)$  Å  
 $b = 20.526(4)$  Å  
 $c = 11.313(2)$  Å  
 $\beta = 101.97(3)^\circ$   
 $V = 1234.4(4)$  Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.67$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.22 \times 0.20 \times 0.18$  mm

## Data collection

Bruker SMART CCD diffractometer  
10913 measured reflections  
2707 independent reflections  
1416 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.109$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.060$   
 $wR(F^2) = 0.187$   
 $S = 0.92$   
2707 reflections  
145 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.37$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.35$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2A}\cdots\text{S1}^i$	0.86	2.56	3.409 (5)	168

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5632).

## References

- Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.  
Casas, J. S., Garcia-T, M. S. & Sordo, J. (2000). *Coord. Chem. Rev.* **209**, 197–261.  
Li, Y.-F. & Jian, F.-F. (2010). *Acta Cryst.* **E66**, o1399.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

## supporting information

*Acta Cryst.* (2010). E66, o2550 [doi:10.1107/S1600536810035671]

**1-(2,4-Dichlorobenzylidene)-4-ethylthiosemicarbazide****Yu-Feng Li****S1. Comment**

Schiff-base have attracted much attention because they can be utilized as effective ligands to be coordination compounds in coordination chemistry. (Casas *et al.*, 2000). As part of our research for new Schiff-base compounds we synthesized the title compound (I), and describe its structure here. In the molecule structure, the dihedral angle between the benzene ring and the thiourea unit is [8.43 (18)°].

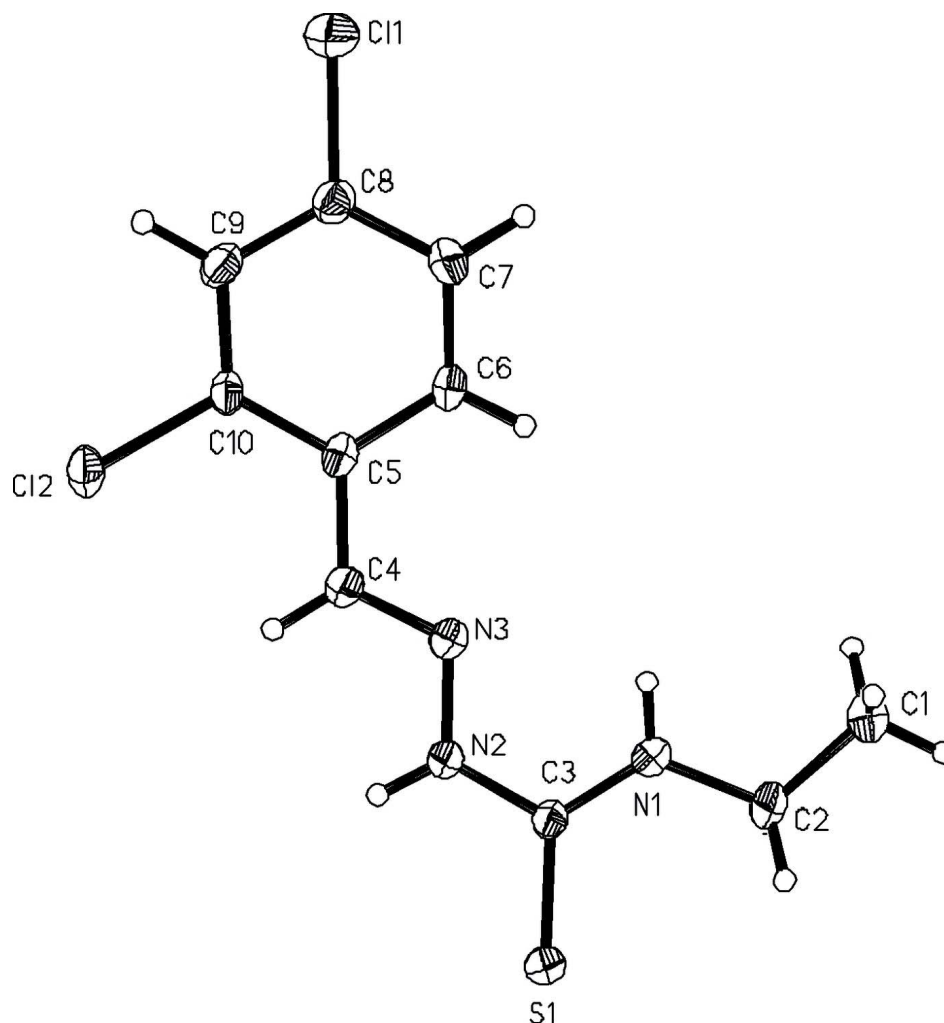
Bond lengths and angles agree with those observed in a related structure (Li & Jian, 2010).

**S2. Experimental**

A mixture of 4-ethylthiosemicarbazide (0.1 mol) and 2,4-dichlorobenzaldehyde (0.1 mol) was stirred in refluxing ethanol (30 mL) for 2 h to afford the title compound (0.090 mol, yield 90%). Colourless blocks of (I) were obtained by recrystallization from ethanol at room temperature.

**S3. Refinement**

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances=0.97 Å, and with  $U_{\text{iso}}=1.2-1.5U_{\text{eq}}$ .

**Figure 1**

The structure of (I) showing 30% probability displacement ellipsoids.

### 1-(2,4-Dichlorobenzylidene)-4-ethylthiosemicarbazide

#### Crystal data

$C_{10}H_{11}Cl_2N_3S$

$M_r = 276.18$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P 2_1/n$

$a = 5.4339$  (11) Å

$b = 20.526$  (4) Å

$c = 11.313$  (2) Å

$\beta = 101.97$  (3)°

$V = 1234.4$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 568$

$D_x = 1.486$  Mg m<sup>-3</sup>

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

Cell parameters from 1416 reflections

$\theta = 3.5$ – $27.5$ °

$\mu = 0.67$  mm<sup>-1</sup>

$T = 293$  K

Block, colorless

$0.22 \times 0.20 \times 0.18$  mm

Data collection

Bruker SMART CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and  $\omega$  scans

10913 measured reflections

2707 independent reflections

1416 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.109$

$\theta_{\text{max}} = 27.5^\circ$ ,  $\theta_{\text{min}} = 3.5^\circ$

$h = -6 \rightarrow 6$

$k = -26 \rightarrow 26$

$l = -14 \rightarrow 14$

Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.060$

$wR(F^2) = 0.187$

$S = 0.92$

2707 reflections

145 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

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\text{max}} = 0.37 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.35 \text{ e } \text{\AA}^{-3}$

Special details

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

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	-0.1063 (2)	0.99987 (6)	0.79802 (13)	0.0434 (4)
Cl2	0.7303 (2)	0.85591 (6)	1.34950 (13)	0.0499 (4)
Cl1	1.4115 (2)	0.70212 (6)	1.20880 (15)	0.0558 (4)
N3	0.4756 (6)	0.90330 (15)	0.9707 (4)	0.0355 (9)
N2	0.2667 (7)	0.94274 (17)	0.9454 (4)	0.0390 (9)
H2A	0.2030	0.9583	1.0029	0.047*
N1	0.2795 (7)	0.93533 (18)	0.7450 (4)	0.0410 (10)
H1A	0.4117	0.9118	0.7681	0.049*
C5	0.7795 (7)	0.84768 (18)	1.1156 (4)	0.0317 (10)
C3	0.1623 (7)	0.95672 (19)	0.8288 (4)	0.0324 (10)
C8	1.1713 (8)	0.7596 (2)	1.1742 (5)	0.0381 (11)
C9	1.0643 (8)	0.78286 (19)	1.2644 (5)	0.0393 (12)
H9A	1.1209	0.7695	1.3440	0.047*
C4	0.5640 (8)	0.89200 (19)	1.0829 (5)	0.0373 (11)
H4A	0.4932	0.9114	1.1423	0.045*
C7	1.0936 (9)	0.7793 (2)	1.0562 (5)	0.0418 (12)
H7A	1.1715	0.7632	0.9964	0.050*

C10	0.8684 (8)	0.82717 (19)	1.2342 (4)	0.0332 (10)
C6	0.8994 (8)	0.8232 (2)	1.0276 (5)	0.0384 (11)
H6A	0.8471	0.8368	0.9480	0.046*
C2	0.2011 (10)	0.9487 (3)	0.6169 (5)	0.0492 (13)
H2B	0.2201	0.9949	0.6029	0.059*
H2C	0.0246	0.9377	0.5906	0.059*
C1	0.3510 (11)	0.9109 (3)	0.5440 (6)	0.0641 (16)
H1B	0.2935	0.9209	0.4599	0.096*
H1C	0.3306	0.8651	0.5567	0.096*
H1D	0.5255	0.9223	0.5686	0.096*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0362 (7)	0.0577 (7)	0.0350 (8)	0.0114 (4)	0.0044 (5)	0.0005 (5)
Cl2	0.0628 (8)	0.0614 (7)	0.0280 (8)	0.0123 (5)	0.0151 (6)	0.0009 (6)
Cl1	0.0561 (8)	0.0551 (7)	0.0546 (11)	0.0202 (5)	0.0078 (7)	0.0014 (6)
N3	0.037 (2)	0.0394 (19)	0.030 (3)	0.0028 (13)	0.0062 (17)	0.0035 (16)
N2	0.039 (2)	0.051 (2)	0.026 (3)	0.0122 (15)	0.0047 (17)	0.0013 (17)
N1	0.037 (2)	0.054 (2)	0.030 (3)	0.0091 (15)	0.0019 (18)	0.0029 (18)
C5	0.039 (2)	0.030 (2)	0.025 (3)	-0.0023 (15)	0.0038 (19)	0.0018 (17)
C3	0.031 (2)	0.040 (2)	0.026 (3)	-0.0020 (16)	0.0043 (19)	-0.0015 (18)
C8	0.040 (2)	0.038 (2)	0.034 (3)	0.0015 (17)	0.003 (2)	-0.0022 (19)
C9	0.045 (3)	0.041 (2)	0.029 (3)	0.0050 (17)	-0.001 (2)	0.0030 (19)
C4	0.038 (2)	0.042 (2)	0.030 (3)	0.0049 (17)	0.004 (2)	-0.0011 (19)
C7	0.045 (3)	0.047 (2)	0.035 (3)	0.0042 (18)	0.012 (2)	-0.007 (2)
C10	0.041 (2)	0.037 (2)	0.022 (3)	0.0003 (16)	0.0089 (19)	-0.0005 (18)
C6	0.045 (3)	0.047 (2)	0.023 (3)	0.0012 (18)	0.005 (2)	-0.003 (2)
C2	0.055 (3)	0.066 (3)	0.025 (3)	0.011 (2)	0.004 (2)	0.004 (2)
C1	0.057 (3)	0.099 (4)	0.038 (4)	0.015 (3)	0.013 (3)	-0.004 (3)

*Geometric parameters (Å, °)*

S1—C3	1.681 (4)	C8—C7	1.376 (7)
Cl2—C10	1.738 (5)	C9—C10	1.388 (6)
Cl1—C8	1.743 (4)	C9—H9A	0.9300
N3—C4	1.282 (6)	C4—H4A	0.9300
N3—N2	1.375 (5)	C7—C6	1.373 (6)
N2—C3	1.354 (6)	C7—H7A	0.9300
N2—H2A	0.8600	C6—H6A	0.9300
N1—C3	1.322 (6)	C2—C1	1.491 (7)
N1—C2	1.449 (7)	C2—H2B	0.9700
N1—H1A	0.8600	C2—H2C	0.9700
C5—C6	1.392 (7)	C1—H1B	0.9600
C5—C10	1.393 (6)	C1—H1C	0.9600
C5—C4	1.468 (6)	C1—H1D	0.9600
C8—C9	1.362 (7)		

C4—N3—N2	115.8 (4)	C6—C7—C8	119.2 (5)
C3—N2—N3	119.2 (4)	C6—C7—H7A	120.4
C3—N2—H2A	120.4	C8—C7—H7A	120.4
N3—N2—H2A	120.4	C9—C10—C5	122.1 (4)
C3—N1—C2	124.7 (4)	C9—C10—Cl2	117.8 (4)
C3—N1—H1A	117.7	C5—C10—Cl2	120.2 (3)
C2—N1—H1A	117.7	C7—C6—C5	121.3 (5)
C6—C5—C10	117.3 (4)	C7—C6—H6A	119.3
C6—C5—C4	120.7 (4)	C5—C6—H6A	119.3
C10—C5—C4	121.9 (4)	N1—C2—C1	111.9 (4)
N1—C3—N2	117.5 (4)	N1—C2—H2B	109.2
N1—C3—S1	123.6 (4)	C1—C2—H2B	109.2
N2—C3—S1	118.9 (4)	N1—C2—H2C	109.2
C9—C8—C7	122.1 (4)	C1—C2—H2C	109.2
C9—C8—Cl1	119.0 (4)	H2B—C2—H2C	107.9
C7—C8—Cl1	118.8 (4)	C2—C1—H1B	109.5
C8—C9—C10	118.0 (5)	C2—C1—H1C	109.5
C8—C9—H9A	121.0	H1B—C1—H1C	109.5
C10—C9—H9A	121.0	C2—C1—H1D	109.5
N3—C4—C5	118.5 (5)	H1B—C1—H1D	109.5
N3—C4—H4A	120.8	H1C—C1—H1D	109.5
C5—C4—H4A	120.8		

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N2—H2A...S1 <sup>i</sup>	0.86	2.56	3.409 (5)	168

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