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

**Yu-Feng Li**

Microscale Science Institute, Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: liyufeng8111@163.com

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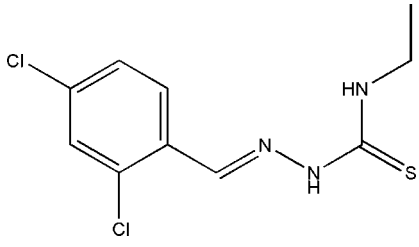
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.060; $wR$ factor = 0.187; data-to-parameter ratio = 18.7.

The title compound, $C_{10}H_{11}Cl_2N_3S$, 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)°. In the crystal, inversion dimers linked by pairs of N—H···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

$C_{10}H_{11}Cl_2N_3S$
$M_r$ = 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)°
$V$ = 1234.4 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.67 mm$^{-1}$
$T$ = 293 K
0.22 × 0.20 × 0.18 mm

### Data collection

Bruker SMART CCD diffractometer
10913 measured reflections
2707 independent reflections
1416 reflections with $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.37 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$A$···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.* E**66**, o1399.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supplementary materials

*Acta Cryst.* (2010). E**66**, o2550 [ **doi:10.1107/S1600536810035671** ]

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

**Y.-F. Li**

## 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).

## 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.

## Refinement

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

## Figures

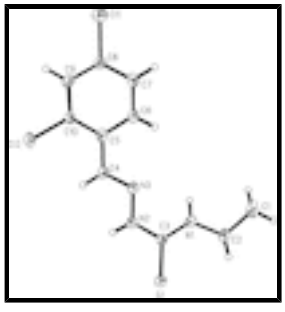


Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids.

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

*Crystal data*

| | |
|---|---|
| $C_{10}H_{11}Cl_2N_3S$ | $F(000)$ = 568 |
| $M_r$ = 276.18 | $D_x$ = 1.486 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, λ = 0.71073 Å |

| | |
|---|---|
| Hall symbol: -P 2yn | Cell parameters from 1416 reflections |
| $a$ = 5.4339 (11) Å | θ = 3.5–27.5° |
| $b$ = 20.526 (4) Å | μ = 0.67 $mm^{-1}$ |
| $c$ = 11.313 (2) Å | $T$ = 293 K |
| β = 101.97 (3)° | Block, colorless |
| $V$ = 1234.4 (4) $Å^3$ | 0.22 × 0.20 × 0.18 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD diffractometer | 1416 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int}$ = 0.109 |
| graphite | $\theta_{max}$ = 27.5°, $\theta_{min}$ = 3.5° |
| phi and ω scans | $h$ = −6→6 |
| 10913 measured reflections | $k$ = −26→26 |
| 2707 independent reflections | $l$ = −14→14 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)]$ = 0.060 | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2)$ = 0.187 | H-atom parameters constrained |
| $S$ = 0.92 | $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 2707 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 145 parameters | $\Delta\rho_{max}$ = 0.37 e $Å^{-3}$ |
| 0 restraints | $\Delta\rho_{min}$ = −0.35 e $Å^{-3}$ |

*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 ($Å^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2A···S1$^{i}$ | 0.86 | 2.56 | 3.409 (5) | 168 |

Symmetry codes: (i) −*x*, −*y*+2, −*z*+2.

**Fig. 1**