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# 2-Chloro-5-nitrobenzaldehyde thiosemicarbazone

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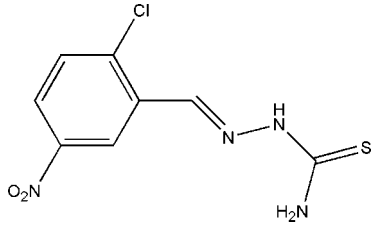
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.039; $wR$ factor = 0.111; data-to-parameter ratio = 15.2.

The title Schiff base compound, $C_8H_7ClN_4O_2S$, was prepared by the reaction of equimolar quantities of 2-chloro-5-nitrobenzaldehyde with thiosemicarbazide in methanol. The molecule adopts a *trans* configuration with respect to the azomethine group and the dihedral angle between the benzene ring and the thiosemicarbazide group is 6.8 (3)°. In the crystal, molecules are linked through intermolecular N—H···S hydrogen bonds, forming chains propagating in [010].

## Related literature

For the crystal structures of similar Schiff base compounds, see: Ferrari *et al.* (1999); Shanmuga Sundara Raj *et al.* (2000); Chattopadhyay *et al.* (1988). For a similar compound reported by the author, see: Hao (2010). For reference structural data, see: Allen *et al.* (1987).




## Experimental

### Crystal data

$C_8H_7ClN_4O_2S$ $M_r$ = 258.69
Monoclinic, $P2_1/c$
$a$ = 11.611 (2) Å
$b$ = 8.439 (2) Å
$c$ = 12.016 (3) Å
$\beta$ = 113.909 (2)°
$V$ = 1076.4 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.54 mm$^{-1}$
$T$ = 298 K
0.18 × 0.17 × 0.17 mm

### Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.909, $T_{max}$ = 0.914
6657 measured reflections
2344 independent reflections
1573 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
$S$ = 1.02
2344 reflections
154 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N4—H4$A$···S1$^{i}$ | 0.89 (1) | 2.53 (1) | 3.408 (2) | 173 (2) |
| N3—H3···S1$^{ii}$ | 0.90 (1) | 2.46 (1) | 3.3266 (19) | 161 (2) |

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5633).

## References

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# supporting information

*Acta Cryst.* (2010). E**66**, o2528 [doi:10.1107/S1600536810035701]

## 2-Chloro-5-nitrobenzaldehyde thiosemicarbazone

**Yu-Mei Hao**

### S1. Comment

Recently, the crystal structures of a number of Schiff base compounds bearing the hydrazone groups derived from the thiosemicarbazide with aldehydes have been reported (Ferrari *et al.*, 1999; Shanmuga Sundara Raj *et al.*, 2000; Chattopadhyay *et al.*, 1988). Recently, the author has reported a Schiff base compound derived from the thiosemicarbazide with 2-hydroxy-4-methoxybenzaldehyde (Hao, 2010), in this paper, the title new Schiff base compound, (I), Fig. 1, is reported.

The molecule of the title compound adopts a *trans* configuration with respect to the azomethine group. All the bond lengths are within normal values (Allen *et al.*, 1987). The dihedral angle between the C1-C6 benzene ring and the plane defined by N2-N3-C8-S1-N4 is 6.8 (3)°, indicating the planar of the molecule. In the crystal structure, molecules are linked through intermolecular N—H···S hydrogen bonds (Table 1), to form chains (Fig. 2).

### S2. Experimental

2-Chloro-5-nitrobenzaldehyde (0.1 mmol, 18.6 mg) and thiosemicarbazide (0.1 mmol, 9.1 mg) were refluxed in methanol (30 ml) for 30 min to give a clear yellow solution. Yellow blocks of (I) were formed by slow evaporation of the solvent over several days at room temperature.

### S3. Refinement

H3, H4A and H4B were located from a difference Fourier map and refined isotropically, with the N—H and H···H distances restrained to 0.90 (1) Å and 1.43 (2) Å, respectively, and with $U_{iso}$ restrained to 0.08Å$^2$. Other H atoms were constrained to ideal geometries, with d(C—H) = 0.93 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C).

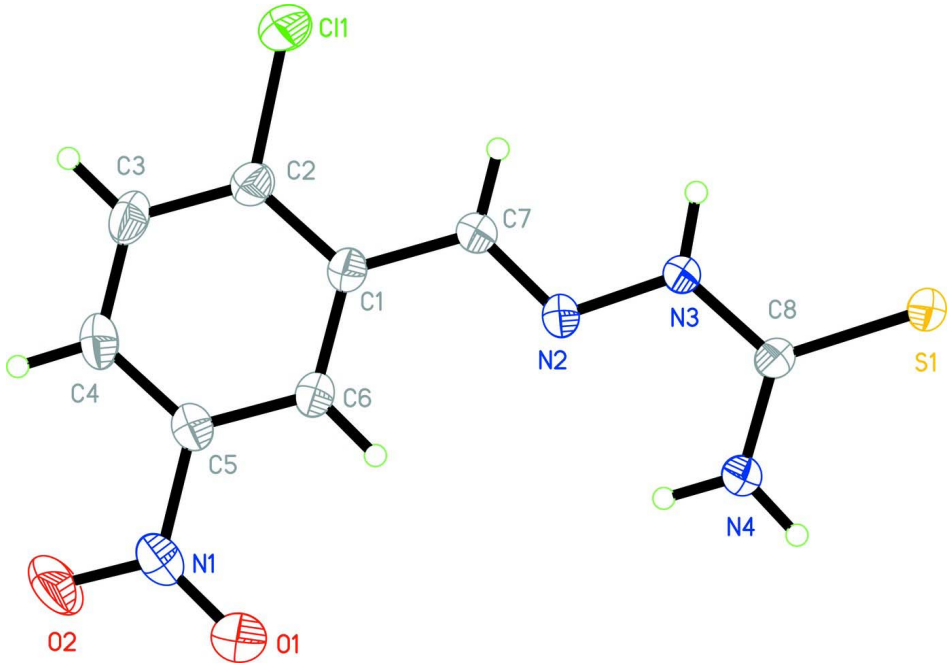


**Figure 1**

The molecular structure of the title compound with 30% probability ellipsoids.

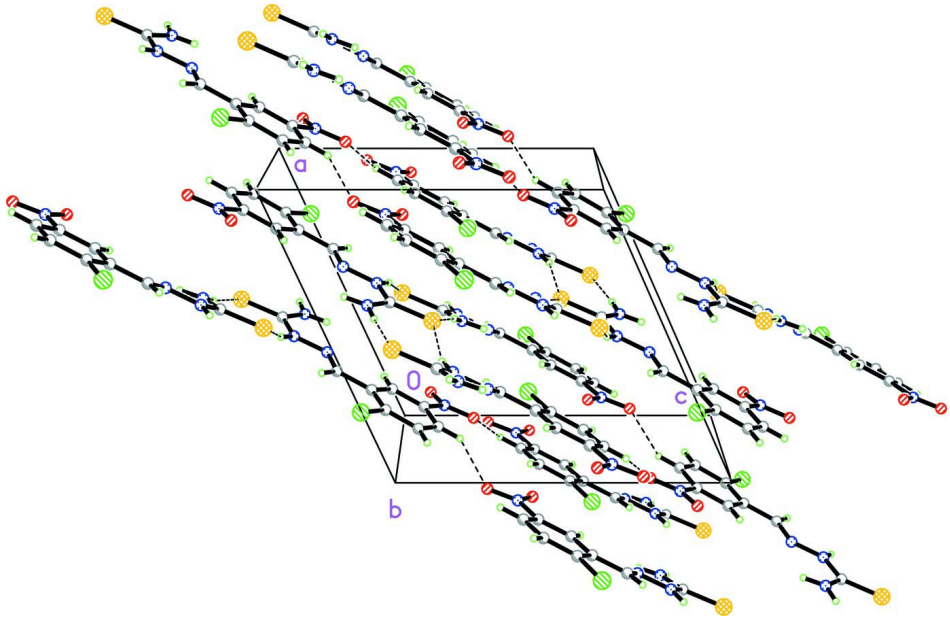


**Figure 2**

Crystal packing of the title compound with hydrogen bonds drawn as dashed lines.

### 2-Chloro-5-nitrobenzaldehyde thiosemicarbazone

*Crystal data*

$C_8H_7ClN_4O_2S$
$M_r$ = 258.69
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 11.611 (2) Å
$b$ = 8.439 (2) Å

$c$ = 12.016 (3) Å
$\beta$ = 113.909 (2)°
$V$ = 1076.4 (4) Å$^3$
$Z$ = 4
$F$(000) = 528
$D_x$ = 1.596 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å

Cell parameters from 1613 reflections
$\theta$ = 2.8–24.7°
$\mu$ = 0.54 mm$^{-1}$
$T$ = 298 K
Block, yellow
0.18 × 0.17 × 0.17 mm

*Data collection*

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.909, $T_{max}$ = 0.914

6657 measured reflections
2344 independent reflections
1573 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038
$\theta_{max}$ = 27.0°, $\theta_{min}$ = 3.0°
$h$ = −14→14
$k$ = −7→10
$l$ = −14→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
$S$ = 1.02
2344 reflections
154 parameters
4 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
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.1148P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.22502 (8) | −0.41154 (8) | 0.48549 (7) | 0.0669 (3) |
| N1 | 0.11203 (19) | 0.1863 (3) | 0.67884 (19) | 0.0541 (6) |
| N2 | 0.33718 (17) | 0.0503 (2) | 0.40689 (17) | 0.0402 (5) |
| N3 | 0.39548 (18) | 0.0489 (2) | 0.32765 (18) | 0.0409 (5) |
| N4 | 0.4229 (2) | 0.3150 (2) | 0.3570 (2) | 0.0543 (6) |
| O1 | 0.13241 (19) | 0.3120 (2) | 0.64124 (19) | 0.0694 (6) |
| O2 | 0.0674 (2) | 0.1755 (3) | 0.7540 (2) | 0.0965 (8) |
| S1 | 0.50185 (6) | 0.18861 (7) | 0.19837 (6) | 0.0460 (2) |
| C1 | 0.2313 (2) | −0.0903 (3) | 0.50752 (19) | 0.0375 (5) |

| | | | | |
|---|---|---|---|---|
| C2 | 0.1926 (2) | −0.2336 (3) | 0.5392 (2) | 0.0424 (6) |
| C3 | 0.1273 (2) | −0.2416 (3) | 0.6133 (2) | 0.0504 (6) |
| H3A | 0.1013 | −0.3389 | 0.6314 | 0.061* |
| C4 | 0.1014 (2) | −0.1031 (3) | 0.6600 (2) | 0.0506 (6) |
| H4 | 0.0584 | −0.1054 | 0.7107 | 0.061* |
| C5 | 0.1405 (2) | 0.0385 (3) | 0.6300 (2) | 0.0422 (6) |
| C6 | 0.2036 (2) | 0.0480 (3) | 0.5549 (2) | 0.0406 (5) |
| H6 | 0.2275 | 0.1460 | 0.5360 | 0.049* |
| C7 | 0.2974 (2) | −0.0829 (3) | 0.4273 (2) | 0.0407 (5) |
| H7 | 0.3105 | −0.1747 | 0.3912 | 0.049* |
| C8 | 0.4371 (2) | 0.1867 (2) | 0.3007 (2) | 0.0382 (5) |
| H3 | 0.412 (2) | −0.0446 (19) | 0.301 (2) | 0.080* |
| H4B | 0.394 (3) | 0.312 (3) | 0.4146 (19) | 0.080* |
| H4A | 0.450 (2) | 0.4093 (18) | 0.346 (2) | 0.080* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.1005 (6) | 0.0371 (4) | 0.0800 (5) | −0.0034 (3) | 0.0541 (4) | 0.0020 (3) |
| N1 | 0.0526 (13) | 0.0621 (15) | 0.0564 (14) | 0.0075 (11) | 0.0311 (11) | −0.0029 (11) |
| N2 | 0.0496 (11) | 0.0372 (11) | 0.0447 (12) | 0.0008 (9) | 0.0303 (9) | 0.0032 (9) |
| N3 | 0.0569 (12) | 0.0283 (10) | 0.0533 (13) | 0.0023 (9) | 0.0385 (10) | 0.0013 (9) |
| N4 | 0.0835 (16) | 0.0338 (11) | 0.0712 (15) | −0.0090 (11) | 0.0578 (13) | −0.0081 (11) |
| O1 | 0.0868 (14) | 0.0503 (12) | 0.0874 (15) | 0.0001 (10) | 0.0521 (12) | −0.0058 (11) |
| O2 | 0.143 (2) | 0.0869 (16) | 0.1135 (18) | 0.0285 (15) | 0.1077 (17) | 0.0100 (14) |
| S1 | 0.0628 (4) | 0.0345 (3) | 0.0580 (4) | −0.0034 (3) | 0.0423 (3) | 0.0004 (3) |
| C1 | 0.0400 (12) | 0.0374 (13) | 0.0381 (13) | −0.0023 (10) | 0.0190 (11) | 0.0026 (10) |
| C2 | 0.0497 (14) | 0.0366 (12) | 0.0432 (14) | −0.0023 (10) | 0.0213 (11) | 0.0017 (10) |
| C3 | 0.0563 (15) | 0.0509 (15) | 0.0503 (15) | −0.0118 (12) | 0.0281 (13) | 0.0073 (12) |
| C4 | 0.0515 (15) | 0.0632 (17) | 0.0483 (15) | −0.0046 (13) | 0.0317 (12) | 0.0042 (13) |
| C5 | 0.0416 (13) | 0.0480 (14) | 0.0413 (14) | 0.0005 (11) | 0.0214 (11) | −0.0010 (11) |
| C6 | 0.0431 (13) | 0.0384 (13) | 0.0457 (14) | −0.0031 (10) | 0.0236 (11) | 0.0037 (10) |
| C7 | 0.0496 (14) | 0.0333 (12) | 0.0486 (14) | 0.0012 (10) | 0.0294 (12) | −0.0009 (11) |
| C8 | 0.0435 (13) | 0.0310 (12) | 0.0460 (14) | −0.0001 (10) | 0.0243 (11) | 0.0023 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C2 | 1.735 (2) | C1—C6 | 1.393 (3) |
| N1—O2 | 1.214 (3) | C1—C2 | 1.396 (3) |
| N1—O1 | 1.214 (3) | C1—C7 | 1.456 (3) |
| N1—C5 | 1.471 (3) | C2—C3 | 1.386 (3) |
| N2—C7 | 1.277 (3) | C3—C4 | 1.382 (4) |
| N2—N3 | 1.374 (2) | C3—H3A | 0.9300 |
| N3—C8 | 1.348 (3) | C4—C5 | 1.378 (3) |
| N3—H3 | 0.899 (10) | C4—H4 | 0.9300 |
| N4—C8 | 1.322 (3) | C5—C6 | 1.376 (3) |
| N4—H4B | 0.88 (3) | C6—H6 | 0.9300 |
| N4—H4A | 0.886 (10) | C7—H7 | 0.9300 |

| | | | |
|---|---|---|---|
| S1—C8 | 1.681 (2) | | |
| | | | |
| O2—N1—O1 | 123.3 (2) | C4—C3—H3A | 120.5 |
| O2—N1—C5 | 117.8 (2) | C2—C3—H3A | 120.5 |
| O1—N1—C5 | 118.9 (2) | C5—C4—C3 | 118.6 (2) |
| C7—N2—N3 | 116.36 (19) | C5—C4—H4 | 120.7 |
| C8—N3—N2 | 118.95 (18) | C3—C4—H4 | 120.7 |
| C8—N3—H3 | 121.7 (18) | C6—C5—C4 | 122.8 (2) |
| N2—N3—H3 | 119.1 (18) | C6—C5—N1 | 118.5 (2) |
| C8—N4—H4B | 122.8 (17) | C4—C5—N1 | 118.6 (2) |
| C8—N4—H4A | 122.1 (17) | C5—C6—C1 | 119.5 (2) |
| H4B—N4—H4A | 115 (2) | C5—C6—H6 | 120.3 |
| C6—C1—C2 | 117.5 (2) | C1—C6—H6 | 120.3 |
| C6—C1—C7 | 120.4 (2) | N2—C7—C1 | 119.6 (2) |
| C2—C1—C7 | 122.1 (2) | N2—C7—H7 | 120.2 |
| C3—C2—C1 | 122.6 (2) | C1—C7—H7 | 120.2 |
| C3—C2—Cl1 | 117.04 (19) | N4—C8—N3 | 116.9 (2) |
| C1—C2—Cl1 | 120.40 (18) | N4—C8—S1 | 123.49 (17) |
| C4—C3—C2 | 119.1 (2) | N3—C8—S1 | 119.59 (16) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N4—H4*A*···S1$^{i}$ | 0.89 (1) | 2.53 (1) | 3.408 (2) | 173 (2) |
| N3—H3···S1$^{ii}$ | 0.90 (1) | 2.46 (1) | 3.3266 (19) | 161 (2) |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*+1, *y*−1/2, −*z*+1/2.