

## 5-Chlorosalicylaldehyde salicylhydrazone

## De-Suo Yang

Department of Chemistry and Chemical Engineering, Baoji College of Arts and Sciences, Baoji 721007, People's Republic of China

Correspondence e-mail: desuoyang@yahoo.com.cn

## Key indicators

Single-crystal X-ray study  
 $T = 298$  K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å  
 $R$  factor = 0.073  
 $wR$  factor = 0.153  
 Data-to-parameter ratio = 14.1

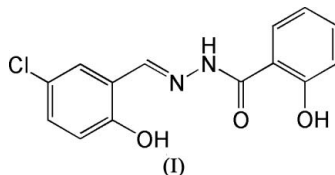
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecule of the title compound,  $\text{C}_{14}\text{H}_{11}\text{ClN}_2\text{O}_3$ , is roughly planar and displays a *trans* configuration with respect to the  $\text{C}=\text{N}$  double bond. The crystal structure is stabilized by intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds.

Received 1 March 2006  
 Accepted 24 March 2006

## Comment

Schiff base compounds have been of great interest for a long time and have played an important role in the development of coordination chemistry (Musie *et al.*, 2001; Bernardo *et al.*, 1996; Paul *et al.*, 2002). As an extension of work on the structural characterization of such compounds, the crystal structure of the title compound, (I), is reported here.



In compound (I) (Fig. 1), all bond lengths are within normal ranges (Allen *et al.*, 1987). The  $\text{C7}=\text{N1}$  bond length of 1.283 (4) Å conforms to the value for a double bond. The bond length of 1.345 (5) Å between atoms C8 and N2 is intermediate between an  $\text{N}-\text{N}$  single bond and an  $\text{N}=\text{N}$  double bond, because of conjugation effects in the molecule. The dihedral angle between the two benzene rings is 12.7 (4)°.

In the crystal structure, molecules are linked through intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds (Table 1), forming chains running along the  $c$  axis (Fig. 2).

## Experimental

Reagents and solvents used were of commercially available quality. The title compound was prepared by the following procedure: 5-chlorosalicylaldehyde (0.1 mmol, 15.7 mg) and 2-hydroxybenzoic acid hydrazide (0.1 mmol, 15.2 mg) were dissolved in MeOH (10 ml). The mixture was stirred at room temperature, giving a clear yellow solution. Crystals of the title compound were formed by gradual evaporation of the solvent over a period of about one week at room temperature.

## Crystal data

$\text{C}_{14}\text{H}_{11}\text{ClN}_2\text{O}_3$   
 $M_r = 290.70$   
 Monoclinic,  $P2_1/c$   
 $a = 18.873$  (2) Å  
 $b = 5.391$  (1) Å  
 $c = 12.851$  (3) Å  
 $\beta = 99.474$  (4)°  
 $V = 1289.6$  (4) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.497$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 551 reflections  
 $\theta = 2.6-23.8^\circ$   
 $\mu = 0.31$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 Flake, yellow  
 $0.14 \times 0.09 \times 0.01$  mm

Data collection

Bruker APEX area-detector diffractometer  
 $\omega$  scan  
 Absorption correction: multi-scan  
*SADABS* (Sheldrick, 1996)  
 $T_{\min} = 0.959$ ,  $T_{\max} = 0.997$   
 9927 measured reflections

2650 independent reflections  
 1393 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.090$   
 $\theta_{\text{max}} = 26.5^\circ$   
 $h = -23 \rightarrow 23$   
 $k = -6 \rightarrow 6$   
 $l = -16 \rightarrow 16$

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.073$   
 $wR(F^2) = 0.153$   
 $S = 1.02$   
 2650 reflections  
 188 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0266P)^2 + 0.8881P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.22 \text{ e } \text{\AA}^{-3}$

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2-H2\cdots O3$	0.90 (1)	1.88 (3)	2.614 (4)	137 (4)
$O3-H3\cdots O2^i$	0.90 (3)	1.70 (3)	2.588 (3)	170 (4)
$O1-H1\cdots O3^{ii}$	0.82	2.56	3.011 (4)	116
$O1-H1\cdots N1$	0.82	2.01	2.717 (4)	144

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

Atoms H2 and H3 were located in a difference Fourier map and refined isotropically, with O–H and N–H distances restrained to 0.90 (1)  $\text{\AA}$ . The other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with an O–H distance of 0.82  $\text{\AA}$ , C–H distances of 0.93  $\text{\AA}$ , and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{O})$ .

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXL97*.

Financial support from the Baoji College of Arts and Sciences research funds (BK2004-2) is gratefully acknowledged.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.  
 Bernardo, K., Leppard, S., Robert, A., Commenges, G., Dahan, F. & Meunier, B. (1996). *Inorg. Chem.* **35**, 387–396.

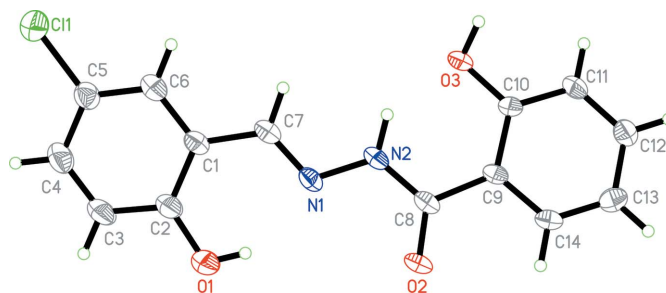


Figure 1  
 The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

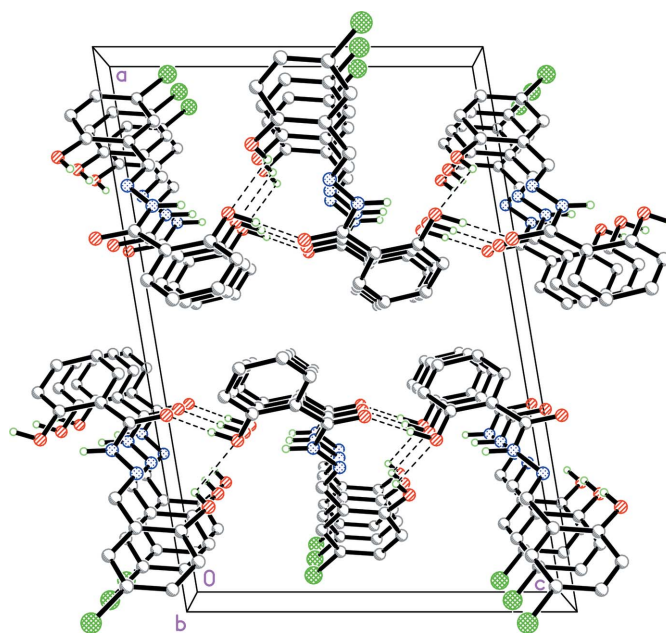


Figure 2  
 Packing of (I) viewed along the  $b$  axis. Dashed lines indicate intermolecular hydrogen bonds.

Bruker (2002). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Music, G. T., Wei, M., Subramaniam, B. & Busch, D. H. (2001). *Inorg. Chem.* **40**, 3336–3341.  
 Paul, S., Barik, A. K., Peng, S. M. & Kar, S. K. (2002). *Inorg. Chem.* **41**, 5803–5809.  
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997a). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.1. Bruker AXS, Inc., Madison, Wisconsin, USA.