

## De-Suo Yang\* and Jin-Bao Guo

Department of Chemistry and Chemical Engineering, Baoji University 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  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.035  
 $wR$  factor = 0.079  
Data-to-parameter ratio = 14.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

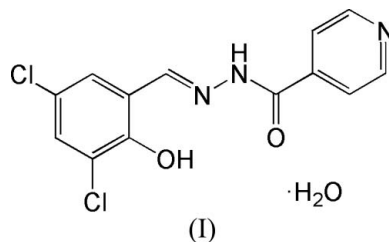
## 2'-[1-(3,5-Dichloro-2-hydroxyphenyl)methylidene]isonicotinohydrazide monohydrate

The asymmetric unit of the title compound,  $\text{C}_{13}\text{H}_9\text{Cl}_2\text{N}_3\text{O}_2 \cdot \text{H}_2\text{O}$ , consists of a Schiff base molecule and a solvent water molecule. The Schiff base molecule is approximately planar and displays a *trans* configuration with respect to the  $\text{C}=\text{N}$  double bond. The crystal structure is stabilized by intermolecular  $\text{N}-\text{H} \cdots \text{O}$ ,  $\text{O}-\text{H} \cdots \text{O}$ ,  $\text{C}-\text{H} \cdots \text{N}$ , and  $\text{C}-\text{H} \cdots \text{O}$  hydrogen bonds, forming a network.

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## Comment

Schiff base compounds have been of great interest for a long time and play 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 our work on the structural characterization of such compounds (Yang, 2006*a,b,c*), the crystal structure of the title compound, (I), is reported here.

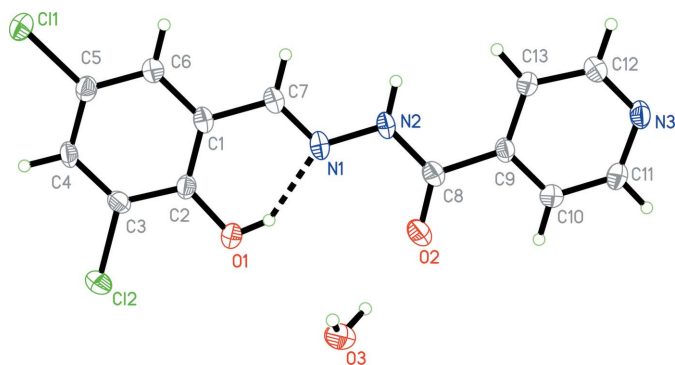


The asymmetric unit of (I) comprises a Schiff base molecule and a solvent water molecule (Fig. 1), in which all the bond lengths are within normal ranges (Allen *et al.*, 1987) and comparable with those of similar compounds (Qian *et al.*, 2006; Qiu, Xu *et al.*, 2006; Qiu, Fang, Liu & Zhu, 2006; Qiu, Fang, Yang *et al.*, 2006; Yang, 2006*d,e*). The  $\text{C}7=\text{N}1$  bond length of 1.278 (3) Å confirms it as a double bond, with a *trans* configuration. The  $\text{C}8-\text{N}2$  bond is also relatively short [1.358 (3) Å], suggesting some degree of delocalization in the acetohydrazide system. The dihedral angle between the benzene ring and the pyridine ring is 5.6 (2)°.

In the crystal structure, water molecules are linked to the Schiff base molecules through  $\text{O}-\text{H} \cdots \text{O}$  hydrogen bonds (Table 1). The molecules are further linked through intermolecular  $\text{N}-\text{H} \cdots \text{O}$ ,  $\text{C}-\text{H} \cdots \text{N}$  and  $\text{C}-\text{H} \cdots \text{O}$  hydrogen bonds, forming a network (Fig. 2).

## Experimental

3,5-Dichlorosalicylaldehyde (0.1 mmol, 19.2 mg) and pyridine-4-carboxylic acid hydrazide (0.1 mmol, 13.7 mg) were dissolved in  $\text{MeOH}-\text{H}_2\text{O}$  (10 ml, 10:1 *v/v*). The mixture was stirred at room temperature to give a clear yellow solution. Crystals of the title

**Figure 1**

The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. An intramolecular hydrogen bond is shown as a dashed line.

compound were formed by gradual evaporation of the solvent over a period of about one week at room temperature. Analysis, found: C 47.40, H 3.49, N 12.73%; calculated for  $C_{13}H_{11}Cl_2N_3O_3$ : C 47.58, H 3.38, N 12.81%.

**Crystal data**

$C_{13}H_9Cl_2N_3O_2 \cdot H_2O$   
 $M_r = 328.15$   
 Monoclinic,  $P2_1$   
 $a = 3.887$  (1) Å  
 $b = 13.638$  (2) Å  
 $c = 13.231$  (2) Å  
 $\beta = 94.546$  (2)°  
 $V = 699.2$  (2) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.558$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.48$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 Plate, yellow  
 $0.27 \times 0.20 \times 0.08$  mm

**Data collection**

Bruker SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{min} = 0.882$ ,  $T_{max} = 0.963$

5674 measured reflections  
 2831 independent reflections  
 2544 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.023$   
 $\theta_{max} = 26.5^\circ$

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.079$   
 $S = 1.02$   
 2831 reflections  
 200 parameters  
 H atoms treated by a mixture of independent and constrained refinement

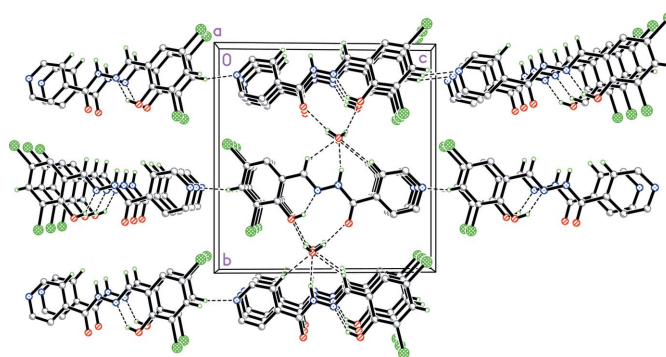
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.18$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983),  
 1334 Friedel pairs  
 Flack parameter: 0.02 (6)

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1 \cdots N1$	0.82	1.87	2.580 (3)	145
$N2-H2 \cdots O3^i$	0.894 (16)	1.991 (15)	2.863 (3)	165
$O3-H3A \cdots O1^{ii}$	0.84 (3)	2.11 (3)	2.895 (3)	155
$O3-H3B \cdots O2$	0.84 (2)	2.00 (2)	2.829 (3)	167 (3)
$C4-H4 \cdots N3^{iii}$	0.93	2.49	3.394 (3)	165
$C7-H7 \cdots O3^i$	0.93	2.56	3.320 (3)	140
$C13-H13 \cdots O3^i$	0.93	2.47	3.364 (3)	162

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

**Figure 2**

The packing of (I), viewed along the  $a$  axis. Dashed lines indicate intermolecular hydrogen bonds.

The N–H and O–H atoms H2, H3A and H3B were located in a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å, the O–H distances restrained to 0.85 (1) Å and the H···H distance restrained to 1.37 (2) Å. 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 Å and C–H distances of 0.93 Å, and with  $U_{iso}(H) = 1.2U_{eq}(C)$  and  $1.5U_{eq}(O)$ .

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.

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**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.
- Bruker (2002). SAINT (Version 5.60) and SMART (Version 5.60). Bruker AXS Inc., Madison, Wisconsin, USA.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Musie, 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.
- Qian, H.-Y., Yin, Z.-G., Jia, J., Liu, S.-M. & Feng, L.-Q. (2006). *Acta Cryst.* **E62**, o3623–o3624.
- Qiu, X.-Y., Fang, X.-N., Liu, W.-S. & Zhu, H.-L. (2006). *Acta Cryst.* **E62**, o2685–o2686.
- Qiu, X.-Y., Fang, X.-N., Yang, S.-L., Liu, W.-S. & Zhu, H.-L. (2006). *Acta Cryst.* **E62**, o2687–o2688.
- Qiu, X.-Y., Xu, H.-J., Liu, W.-S. & Zhu, H.-L. (2006). *Acta Cryst.* **E62**, o2304–o2305.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (1997a). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). SHELXTL. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Yang, D.-S. (2006a). *Acta Cryst.* **E62**, o1395–o1396.
- Yang, D.-S. (2006b). *Acta Cryst.* **E62**, o1591–o1592.
- Yang, D.-S. (2006c). *Acta Cryst.* **E62**, o2365–o2366.
- Yang, D.-S. (2006d). *Acta Cryst.* **E62**, o3755–o3756.
- Yang, D.-S. (2006e). *Acta Cryst.* **E62**, o3792–o3793.