Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2,4-Dichlorobenzaldehyde picolinoylhydrazone

Yue-Qin Xie,[a] Liang-Quan Sheng,[b] Wei Zhu[b] and Si-Chang Shao[b]*

[a]Department of Chemistry, Anhui Institute of Education, HeFei Anhui 230032, People's Republic of China, and [b]Department of Chemistry, Fuyang Normal College, Fuyang Anhui 236041, People's Republic of China

Correspondence e-mail: shaosic@fync.edu.cn

Received 17 November 2005
Accepted 28 November 2005
Online 7 December 2005

Key indicators

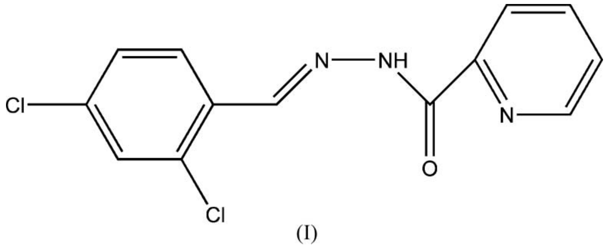
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.098
Data-to-parameter ratio = 13.3

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

The title molecule, $C_{13}H_9Cl_2N_3O$, adopts a *trans* configuration with respect to the C═N double bond. There are two molecules in the asymmetric unit. The dihedral angles between the two rings are 17.2 (3) and 145.4 (3)°. The crystal structure is stabilized by intermolecular N—H⋯O hydrogen bonds which link the molecules into a chain structure.

Comment

Schiff base compounds have been of great interest for many years. These compounds play an important role in the development of coordination chemistry related to catalysis and enzymatic reactions (Brunner *et al.*, 1983), magnetism and molecular architectures (Miller *et al.*, 2000). The electron-withdrawing effect of the chlorine substituents in the dichlorobenzylidene unit may reduce the coordinating ability of the N atoms of the hydrazone. As an extension of work on the structural characterization of picolinohydrazone Schiff base compounds (Shao *et al.*, 2004), we report here the crystal structure of (I), a new salicylhydrazone with 2,4-dichlorobenzaldehyde.



(I)

In the title compound, (I), which crystallizes with two unique molecules in the asymmetric unit (Fig. 1), the C—N bonds in the hydrazone units are characteristically short (Table 1) because of conjugation effects. All other bond lengths are within normal ranges (Allen *et al.*, 1987). The dihedral angles between the pairs of rings in each molecule are 17.2 (3)° (C1/C2/C3/C4/C5/C6 with C9/C10/C11/C12/C13/N3) and 145.4 (3)° (C14/C15/C16/C17/C18/C19 with C22/C23/C24/C25/C26/N6). The crystal structure is stabilized by intermolecular N—H⋯O hydrogen bonds (Table 2 and Fig. 2).

Experimental

2,4-Dichlorobenzaldehyde (0.2 mmol, 35 mg) and picolinohydrazide (0.2 mmol, 27.4 mg) were dissolved in methanol (10 ml). The mixture was stirred at room temperature for 10 min. to give a clear yellow solution. The solution was set aside for 8 d to allow slow evaporation of the solvent. Large colourless block-shaped crystals separated; these were collected and washed three times with water.

Crystal data

$C_{13}H_9Cl_2N_3O$
$M_r = 294.13$
Monoclinic, $P2_1/n$
$a = 10.500\ (11)$ Å
$b = 17.763\ (18)$ Å
$c = 14.757\ (15)$ Å
$\beta = 103.794\ (14)^\circ$
$V = 2673\ (5)$ Å^3
$Z = 8$
$D_x = 1.462$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1925 reflections
$\theta = 4.6$–39.8°
$\mu = 0.48$ mm^{-1}
$T = 298\ (2)$ K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.831$, $T_{\max} = 0.910$
14951 measured reflections
4662 independent reflections
2532 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 25.0^\circ$
$h = -12 \rightarrow 12$
$k = -20 \rightarrow 18$
$l = -17 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.098$
$S = 0.86$
4662 reflections
351 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.219 (3)	N2—C8	1.339 (3)
O2—C21	1.213 (3)	N4—C20	1.277 (3)
N1—C7	1.267 (3)	N4—N5	1.369 (3)
N1—N2	1.366 (3)	N5—C21	1.348 (3)
C7—N1—N2	115.9 (2)	O1—C8—N2	123.5 (2)
C8—N2—N1	119.0 (2)	O1—C8—C9	121.8 (3)
C20—N4—N5	116.1 (2)	O2—C21—N5	124.2 (2)
C21—N5—N4	118.2 (2)	O2—C21—C22	121.6 (3)
C7—N1—N2—C8	−180.0 (2)	C20—N4—N5—C21	178.9 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.85 (2)	2.29 (2)	3.105 (4)	159 (2)
N5—H5*A*···O1$^{\rm i}$	0.95 (2)	2.26 (2)	3.117 (4)	150 (2)

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

Atoms H2 and H5*A* were located in a difference Fourier map and their positions were refined freely with isotropic displacement parameters. All other H atoms were placed in geometrically idealized positions (C—H 0.93 Å) and constrained to ride on their parent atoms with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL97*.

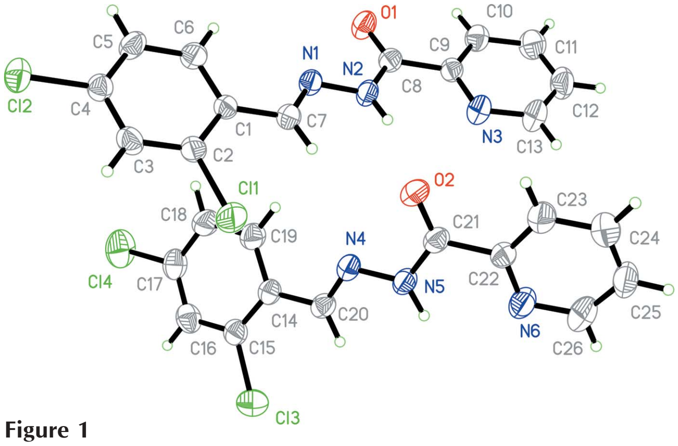


Figure 1
The structure of the asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

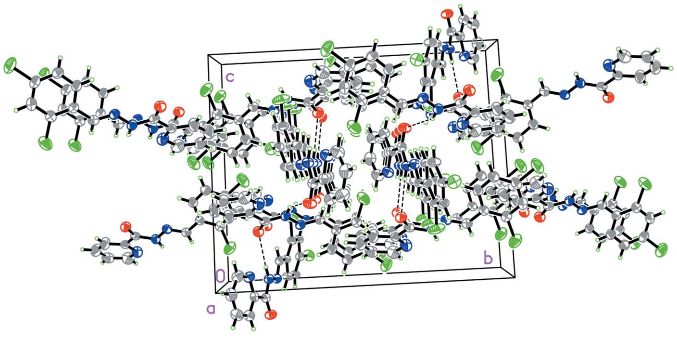


Figure 2
The crystal packing of (I), viewed along the *a* axis. Dashed lines indicate intermolecular hydrogen bonds.

The authors thank the Education Office of Anhui Province, China, for research grant No. 2005kj137.

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.
Bruker (2000). *SHELXTL*. Version 6.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2002). *SADABS, SAINT and SMART*. Bruker AXS Inc., Madison, Winconsin.
Brunner, H., Riepl, G., Weitzer, H. (1983). *Angew. Chem. Int. Ed. Engl.* **22**, 331–332.
Miller, J. S., Epstein A. J. (2000). *Coord. Chem. Rev.* **206–207**, 651–660.
Shao, S. C., You, Z. L., F, S. H., Tang, L. L., Lin, Y. S. Zhu, H. L. (2004). *Acta Cryst.* E**60**, o2181–o2182.
Sheldrick G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Acta Cryst. (2006). E**62**, o26–o27 Xie *et al.* • $C_{13}H_9Cl_2N_3O$ **o27**