Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-Chloro-2′-(2-hydroxybenzylidene)-nicotinohydrazide monohydrate

De-Suo Yang

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

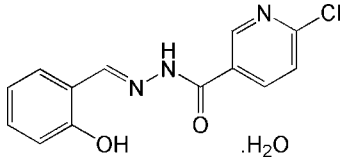
Received 24 July 2007; accepted 5 August 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.036; wR factor = 0.091; data-to-parameter ratio = 11.2.

The asymmetric unit of the title compound, $C_{13}H_{10}ClN_3O_2\cdot H_2O$, consists of a Schiff base molecule and a solvent water molecule. The Schiff base molecule displays a *trans* configuration with respect to the C=N double bond. The dihedral angle between the benzene and pyridine rings is 22.3 (3)°. In the crystal structure, molecules are linked through intermolecular O—H···O and N—H···O hydrogen bonds, forming layers parallel to the *bc* plane.

Related literature

For related structures, see Yang (2006*a*,*b*,*c*,*d*,*e*, 2007); Yang & Guo (2006). For related literature, see: Allen *et al.* (1987); Bernardo *et al.* (1996); Musie *et al.* (2001); Paul *et al.* (2002).



Experimental

Crystal data

$C_{13}H_{10}ClN_3O_2\cdot H_2O$
M_r = 293.71
Monoclinic, *Cc*
a = 28.125 (11) Å
b = 3.8193 (15) Å
c = 12.281 (5) Å
β = 92.764 (5)°
V = 1317.7 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 298 (2) K
0.32 × 0.28 × 0.27 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.910, T_{max} = 0.923
3648 measured reflections
2141 independent reflections
1826 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.091
S = 1.04
2141 reflections
192 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), 654 Friedel pairs
Flack parameter: 0.15 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2^{i}	0.849 (10)	1.98 (2)	2.771 (3)	155 (4)
O3—H3B···O1^{ii}	0.856 (10)	2.15 (2)	2.917 (4)	149 (4)
N2—H2···O3^{iii}	0.899 (10)	1.982 (14)	2.864 (3)	167 (4)
O1—H1···N1	0.82	1.89	2.610 (3)	147

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

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

The author acknowledges Key Laboratory Construction Support from the Education Office of Shanxi Province (project No. 05JS43).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2148).

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* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 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.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Yang, D.-S. (2006*a*). *Acta Cryst.* E**62**, o1395–o1396.
Yang, D.-S. (2006*b*). *Acta Cryst.* E**62**, o1591–o1592.
Yang, D.-S. (2006*c*). *Acta Cryst.* E**62**, o2365–o2366.
Yang, D.-S. (2006*d*). *Acta Cryst.* E**62**, o3755–o3756.
Yang, D.-S. (2006*e*). *Acta Cryst.* E**62**, o3792–o3793.
Yang, D.-S. (2007). *J. Chem. Crystallogr.* **37**, 343–348.
Yang, D.-S. & Guo, J.-B. (2006). *Acta Cryst.* E**62**, o4414–o4415.

  doi:10.1107/S1600536807038500

supplementary materials

Acta Cryst. (2007). E**63**, o3738 [**doi:10.1107/S1600536807038500**]

6-Chloro-2'-(2-hydroxybenzylidene)nicotinohydrazide monohydrate

D.-S. Yang

Comment

Schiff base compounds have been of great interest for a long time. These compounds play an important role in the development of coordination chemistry (Musie *et al.*, 2001; Bernardo *et al.*, 1996; Paul *et al.*, 2002). Recently, we have reported a few Schiff base compounds (Yang, 2006*a*,b,c,d,e, 2007; Yang & Guo, 2006). As a further investigation of this work, the crystal structure of the title compound is reported here.

The asymmetric unit of the title compound, $C_{13}H_{10}ClN_3O_2\cdot H_2O$, consists of a Schiff base molecule and a water molecule (Fig. 1). The Schiff base molecule displays a *trans* configuration with respect to the C═N double bond. All the bond lengths are within normal ranges (Allen *et al.*, 1987). The C7═N1 bond length of 1.279 (3) Å conforms to the value for a double bond. The bond length of 1.343 (4) Å between atoms C8 and N2 is intermediate between an C—N single bond and an C═N double bond, because of conjugation effects in the molecule. The dihedral angle between the benzene ring and the pyridine ring is 22.3 (3)°. In the crystal structure, molecules are linked through intermolecular O—H···O and N—H···O hydrogen bonds, forming layers parallel to the *bc* plane (Fig. 2).

Experimental

Salicylaldehyde (0.1 mmol, 12.0 mg) and 6-chloronicotinic acid hydrazide (0.1 mmol, 17.0 mg) were dissolved in MeOH (10 ml). The mixture was stirred at room temperature to give a clear colorless solution. Crystals of the title compound were formed by gradual evaporation of the solvent over a period of 5 days at room temperature.

Refinement

Atoms H2, H3A and H3B were located in a difference Fourier map and refined isotropically, with O—H distances restrained to 0.85 (1) Å, N—H distance restrained to 0.90 (1) Å, H···H distance restrained to 1.37 (2) Å. Other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with O—H distance of 0.82 Å, C—H distances of 0.93 Å, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(O).

Figures

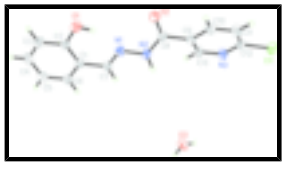


Fig. 1. The structure of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Intramolecular hydrogen bonds are shown as dashed lines.

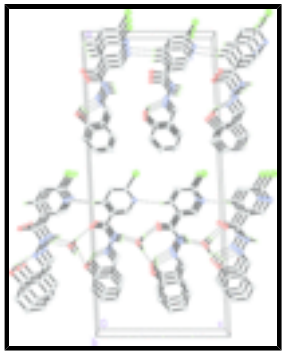


Fig. 2. Molecular packing as viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

6-Chloro-2'-(2-hydroxybenzylidene)nicotinohydrazide monohydrate

Crystal data

$C_{13}H_{10}ClN_3O_2 \cdot H_2O_1$	$F_{000} = 608$
$M_r = 293.71$	$D_x = 1.480$ Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation λ = 0.71073 Å
a = 28.125 (11) Å	Cell parameters from 1313 reflections
b = 3.8193 (15) Å	θ = 2.5–26.7º
c = 12.281 (5) Å	μ = 0.30 mm^{-1}
β = 92.764 (5)º	*T* = 298 (2) K
V = 1317.7 (9) Å^3	Block, colourless
Z = 4	0.32 × 0.28 × 0.27 mm

Data collection

Bruker SMART CCD diffractometer	2141 independent reflections
Radiation source: fine-focus sealed tube	1826 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
T = 298(2) K	$\theta_{max} = 27.5$º
ω scans	$\theta_{min} = 2.9$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −33→36
$T_{min} = 0.910$, $T_{max} = 0.923$	*k* = −4→4
3648 measured reflections	*l* = −15→13

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.2959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.091$	$(\Delta/\sigma)_{max} < 0.001$

S = 1.04	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
2141 reflections	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
192 parameters	Extinction correction: none
6 restraints	Absolute structure: Flack (1983), 643 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.15 (9)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.53442 (3)	0.6124 (2)	0.35665 (6)	0.0658 (3)
O1	0.22659 (8)	−0.2060 (6)	−0.05359 (16)	0.0570 (6)
H1	0.2498	−0.1108	−0.0233	0.086*
O2	0.36101 (7)	0.0985 (6)	0.00747 (15)	0.0554 (6)
O3	0.30098 (9)	0.3904 (7)	0.84650 (17)	0.0631 (6)
N1	0.27769 (8)	0.0670 (6)	0.10791 (18)	0.0420 (5)
N2	0.31879 (8)	0.2013 (7)	0.15714 (18)	0.0435 (5)
C1	0.19752 (9)	−0.1146 (7)	0.1247 (2)	0.0355 (6)
C2	0.19157 (10)	−0.2361 (7)	0.0176 (2)	0.0396 (6)
C3	0.14878 (11)	−0.3907 (8)	−0.0177 (2)	0.0504 (8)
H3	0.1446	−0.4695	−0.0892	0.060*
C4	0.11279 (11)	−0.4269 (8)	0.0528 (3)	0.0527 (8)
H4	0.0844	−0.5320	0.0287	0.063*
C5	0.11802 (11)	−0.3107 (8)	0.1582 (3)	0.0524 (7)
H5	0.0933	−0.3369	0.2053	0.063*
C6	0.16008 (11)	−0.1548 (7)	0.1940 (2)	0.0446 (6)
H6	0.1636	−0.0752	0.2655	0.054*
C7	0.24163 (9)	0.0418 (7)	0.1670 (2)	0.0399 (6)
H7	0.2437	0.1251	0.2383	0.048*
C8	0.35906 (10)	0.1958 (7)	0.1026 (2)	0.0410 (6)
C9	0.40330 (9)	0.3079 (7)	0.16605 (19)	0.0388 (6)
C10	0.44136 (11)	0.4366 (8)	0.1104 (2)	0.0480 (7)
H10	0.4391	0.4588	0.0349	0.058*
C11	0.48227 (11)	0.5306 (9)	0.1682 (2)	0.0525 (8)
H11	0.5083	0.6190	0.1333	0.063*

C12	0.48349 (10)	0.4898 (8)	0.2797 (2)	0.0449 (7)
N3	0.44856 (9)	0.3663 (7)	0.33543 (19)	0.0485 (6)
C13	0.40865 (10)	0.2742 (8)	0.2784 (2)	0.0459 (7)
H13	0.3834	0.1838	0.3159	0.055*
H2	0.3168 (13)	0.308 (10)	0.2220 (17)	0.080*
H3B	0.2754 (7)	0.427 (11)	0.880 (2)	0.080*
H3A	0.3231 (8)	0.362 (10)	0.895 (2)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0493 (4)	0.0758 (6)	0.0706 (5)	−0.0109 (4)	−0.0159 (4)	−0.0117 (4)
O1	0.0617 (13)	0.0726 (16)	0.0371 (10)	−0.0075 (12)	0.0061 (10)	−0.0067 (10)
O2	0.0486 (12)	0.0836 (16)	0.0335 (10)	0.0052 (11)	−0.0034 (8)	−0.0114 (9)
O3	0.0608 (14)	0.0869 (18)	0.0417 (12)	0.0111 (14)	0.0046 (10)	0.0117 (11)
N1	0.0385 (12)	0.0491 (13)	0.0377 (12)	0.0004 (11)	−0.0044 (10)	−0.0004 (10)
N2	0.0392 (12)	0.0571 (15)	0.0337 (11)	−0.0034 (11)	−0.0037 (10)	−0.0062 (10)
C1	0.0364 (13)	0.0331 (14)	0.0364 (13)	0.0042 (11)	−0.0043 (10)	0.0022 (10)
C2	0.0466 (15)	0.0392 (15)	0.0327 (13)	0.0031 (13)	−0.0022 (12)	−0.0008 (10)
C3	0.0596 (19)	0.0456 (18)	0.0441 (16)	0.0002 (14)	−0.0161 (14)	−0.0011 (12)
C4	0.0434 (16)	0.0455 (17)	0.068 (2)	−0.0047 (14)	−0.0133 (15)	0.0086 (14)
C5	0.0452 (16)	0.0514 (18)	0.061 (2)	0.0013 (14)	0.0037 (15)	0.0076 (14)
C6	0.0485 (16)	0.0469 (16)	0.0386 (14)	0.0027 (13)	0.0027 (12)	0.0020 (11)
C7	0.0434 (15)	0.0432 (15)	0.0323 (12)	0.0027 (12)	−0.0059 (11)	0.0001 (11)
C8	0.0432 (15)	0.0454 (16)	0.0335 (13)	0.0039 (12)	−0.0067 (11)	−0.0003 (11)
C9	0.0382 (14)	0.0438 (16)	0.0343 (13)	0.0021 (11)	−0.0006 (11)	−0.0002 (10)
C10	0.0458 (15)	0.065 (2)	0.0337 (13)	−0.0018 (15)	0.0035 (11)	0.0057 (13)
C11	0.0427 (16)	0.065 (2)	0.0495 (17)	−0.0066 (14)	0.0028 (13)	0.0060 (14)
C12	0.0374 (15)	0.0464 (16)	0.0502 (15)	−0.0016 (12)	−0.0067 (12)	−0.0063 (12)
N3	0.0422 (13)	0.0643 (17)	0.0384 (12)	−0.0036 (12)	−0.0038 (10)	−0.0032 (11)
C13	0.0413 (15)	0.0594 (19)	0.0366 (15)	−0.0067 (13)	−0.0016 (11)	0.0025 (12)

Geometric parameters (Å, °)

Cl1—C12	1.742 (3)	C4—C5	1.370 (5)
O1—C2	1.353 (3)	C4—H4	0.9300
O1—H1	0.8200	C5—C6	1.377 (4)
O2—C8	1.230 (3)	C5—H5	0.9300
O3—H3B	0.856 (10)	C6—H6	0.9300
O3—H3A	0.849 (10)	C7—H7	0.9300
N1—C7	1.279 (3)	C8—C9	1.498 (3)
N1—N2	1.377 (3)	C9—C13	1.387 (4)
N2—C8	1.343 (4)	C9—C10	1.387 (4)
N2—H2	0.899 (10)	C10—C11	1.370 (4)
C1—C6	1.394 (4)	C10—H10	0.9300
C1—C2	1.398 (3)	C11—C12	1.377 (4)
C1—C7	1.451 (3)	C11—H11	0.9300
C2—C3	1.391 (4)	C12—N3	1.312 (4)
C3—C4	1.370 (4)	N3—C13	1.341 (3)

C3—H3	0.9300	C13—H13	0.9300
C2—O1—H1	109.5	C1—C6—H6	119.5
H3B—O3—H3A	107 (2)	N1—C7—C1	121.0 (2)
C7—N1—N2	116.8 (2)	N1—C7—H7	119.5
C8—N2—N1	119.0 (2)	C1—C7—H7	119.5
C8—N2—H2	123 (2)	O2—C8—N2	123.8 (2)
N1—N2—H2	118 (2)	O2—C8—C9	120.4 (3)
C6—C1—C2	118.6 (2)	N2—C8—C9	115.8 (2)
C6—C1—C7	119.0 (2)	C13—C9—C10	118.3 (2)
C2—C1—C7	122.4 (2)	C13—C9—C8	122.6 (2)
O1—C2—C3	118.7 (2)	C10—C9—C8	119.1 (2)
O1—C2—C1	121.5 (2)	C11—C10—C9	119.1 (3)
C3—C2—C1	119.8 (3)	C11—C10—H10	120.4
C4—C3—C2	120.0 (3)	C9—C10—H10	120.4
C4—C3—H3	120.0	C10—C11—C12	117.7 (3)
C2—C3—H3	120.0	C10—C11—H11	121.1
C3—C4—C5	121.0 (3)	C12—C11—H11	121.1
C3—C4—H4	119.5	N3—C12—C11	125.2 (3)
C5—C4—H4	119.5	N3—C12—Cl1	115.5 (2)
C4—C5—C6	119.5 (3)	C11—C12—Cl1	119.3 (2)
C4—C5—H5	120.2	C12—N3—C13	116.8 (2)
C6—C5—H5	120.2	N3—C13—C9	122.9 (3)
C5—C6—C1	121.0 (3)	N3—C13—H13	118.5
C5—C6—H6	119.5	C9—C13—H13	118.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O2^{i}	0.849 (10)	1.98 (2)	2.771 (3)	155 (4)
O3—H3B···O1^{ii}	0.856 (10)	2.15 (2)	2.917 (4)	149 (4)
N2—H2···O3^{iii}	0.899 (10)	1.982 (14)	2.864 (3)	167 (4)
O1—H1···N1	0.82	1.89	2.610 (3)	147

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

Fig. 1

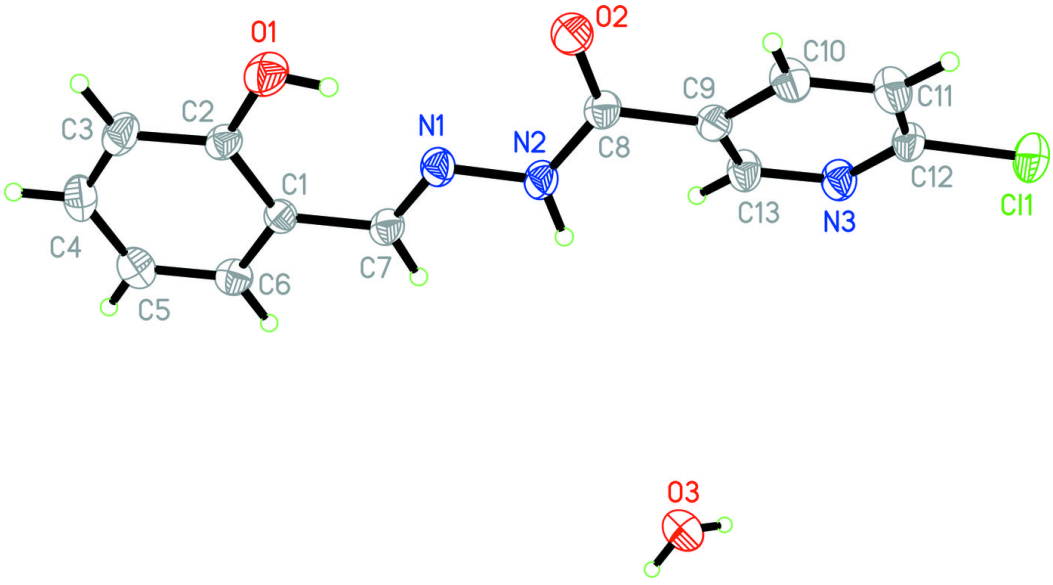


Fig. 2