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6-Chloro-*N*′-[4-(dimethylamino)benzylidene]nicotinohydrazide monohydrate

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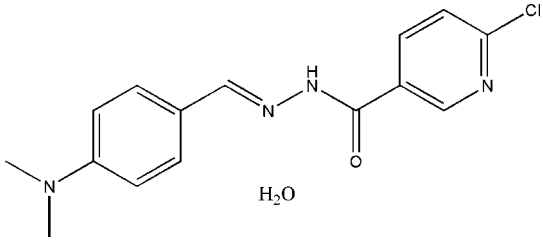
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.058; wR factor = 0.155; data-to-parameter ratio = 16.4.

The asymmetric unit of the title compound, $C_{15}H_{15}ClN_4O\cdot H_2O$, consists of a roughly planar Schiff base molecule which displays a *trans* configuration with respect to the C=N and C—N bonds, and a water molecule. The crystal structure is stabilized by intermolecular N—H···O, O—H···O, O—H···N and C—H···O hydrogen bonds, forming chains running along the *c* axis.

Related literature

For related literature, see: Chen *et al.* (1997); Fan *et al.* (2007); Kim *et al.* (2005); Nimitsiriwat *et al.* (2004); Ren *et al.* (2002).



Experimental

Crystal data

$C_{15}H_{15}ClN_4O\cdot H_2O$
M_r = 320.78
Monoclinic, $P2_1/c$
a = 18.422 (4) Å
b = 7.3390 (15) Å
c = 11.797 (2) Å
β = 107.92 (3)°
V = 1517.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 298 (2) K
0.23 × 0.22 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.942, T_{max} = 0.944
11439 measured reflections
3445 independent reflections
2214 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.155
S = 1.04
3445 reflections
210 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O2^{i}	0.897 (10)	2.020 (14)	2.874 (3)	159 (3)
O2—H2B···O1^{ii}	0.847 (10)	2.118 (10)	2.957 (2)	171 (3)
O2—H2A···N2^{iii}	0.851 (10)	2.209 (10)	3.058 (3)	176 (3)
O2—H2A···O1^{iii}	0.851 (10)	2.61 (2)	3.064 (3)	115 (2)
C15—H15···O1^{iv}	0.93	2.46	3.284 (3)	148

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+2$; (iii) $x-1, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 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*.

Financial support from the Third Affiliated Hospital of Suzhou University is acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2197).

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supplementary materials

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6-Chloro-*N*'-[4-(dimethylamino)benzylidene]nicotinohydrazide monohydrate

F. Zhi

Comment

Schiff base compounds have been widely investigated over a century (Fan *et al.*, 2007; Kim *et al.*, 2005; Nimitsiriwat *et al.*, 2004). Some of the compounds have been found to have pharmacological and antibacterial activity (Chen *et al.*, 1997; Ren *et al.*, 2002). In this paper, the crystal structure of a new Schiff base compound derived from the condensation reaction of 4-dimethylaminobenzaldehyde with 6-chloronicotinic acid hydrazide is reported.

The compound consists of a roughly planar Schiff base molecule and a lattice water molecule (Fig. 1). The Schiff base molecule of the compound displays a *trans* configuration with respect to the C═N and C—N bonds. The dihedral angle between the C1—C6 phenyl ring and the C11—C15/N4 pyridine ring is 2.9 (3)°. In the crystal structure, The molecules are linked through intermolecular C—H···O hydrogen bonds (Table 1), forming chains running along the *c* axis (Fig. 2).

Experimental

4-Dimethylaminobenzaldehyde (0.1 mmol, 14.9 mg) and 6-chloronicotinic acid hydrazide (0.1 mmol, 17.1 mg) were dissolved in a methanol solution (10 ml). The mixture was stirred at room temperature to give a clear yellow solution. Crystals of the title compound were formed by gradual evaporation of the solvent for a week at room temperature.

Refinement

H2A, H2B, H3A were located from a difference Fourier map and refined isotropically, with N–H distance restrained to 0.90 (1) Å, O–H distances restrained to 0.85 (1) Å, and H···H distance restrained to 1.37 (2) Å. Other H atoms were constrained to ideal geometries, with C–H = 0.93–0.96 Å, and with U_{iso}(H) set to 1.2U_{eq}(C) and 1.5U_{eq}(methyl C).

Figures

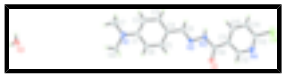

Fig. 1. The structure of (I) at the 30% probability level.

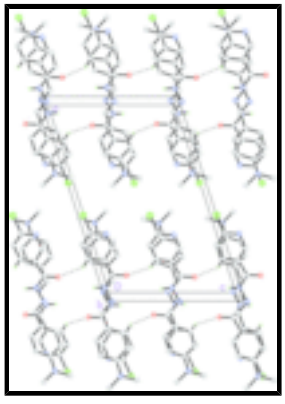

Fig. 2. Molecular packing of (I), viewed along the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

6-Chloro-*N*'-[4-(dimethylamino)benzylidene]nicotinohydrazide monohydrate

Crystal data

$C_{15}H_{15}ClN_4O·H_2O$	F_{000} = 672
M_r = 320.78	D_x = 1.404 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 18.422 (4) Å	Cell parameters from 1507 reflections
b = 7.3390 (15) Å	θ = 2.3–24.0º
c = 11.797 (2) Å	μ = 0.27 mm^{-1}
β = 107.92 (3)º	T = 298 (2) K
V = 1517.6 (5) Å^3	Block, yellow
Z = 4	0.23 × 0.22 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3445 independent reflections
Radiation source: fine-focus sealed tube	2214 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.046
T = 298(2) K	θ_{max} = 27.5º
ω scan	θ_{min} = 1.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −23→23
T_{min} = 0.942, T_{max} = 0.944	k = −9→9
11439 measured reflections	l = −15→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.059	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.155	$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.2814P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = <0.001
3445 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
210 parameters	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	1.41748 (4)	0.60270 (12)	1.02080 (8)	0.0788 (3)
O1	1.11240 (9)	0.6324 (3)	1.20002 (14)	0.0513 (5)
O2	0.02124 (10)	0.5647 (3)	0.77271 (15)	0.0544 (5)
N1	0.62665 (12)	0.9079 (3)	0.9291 (2)	0.0587 (6)
N2	0.98581 (10)	0.7481 (3)	1.03019 (16)	0.0389 (5)
N3	1.05463 (10)	0.7141 (3)	1.00841 (16)	0.0386 (5)
N4	1.32175 (13)	0.5662 (3)	1.1454 (2)	0.0615 (6)
C1	0.70174 (13)	0.8874 (3)	0.9330 (2)	0.0413 (6)
C2	0.72558 (13)	0.9210 (3)	0.8331 (2)	0.0439 (6)
H2	0.6904	0.9610	0.7627	0.053*
C3	0.80053 (13)	0.8957 (3)	0.8379 (2)	0.0423 (6)
H3	0.8149	0.9200	0.7705	0.051*
C4	0.85538 (12)	0.8347 (3)	0.94081 (19)	0.0365 (5)
C5	0.83183 (13)	0.8043 (3)	1.04081 (19)	0.0394 (6)
H5	0.8672	0.7648	1.1112	0.047*
C6	0.75736 (13)	0.8315 (3)	1.0374 (2)	0.0448 (6)
H6	0.7436	0.8124	1.1061	0.054*
C7	0.56829 (16)	0.9391 (5)	0.8170 (3)	0.0850 (11)
H7A	0.5756	1.0568	0.7868	0.128*
H7B	0.5190	0.9343	0.8287	0.128*
H7C	0.5714	0.8469	0.7610	0.128*
C8	0.60392 (15)	0.8896 (4)	1.0351 (3)	0.0674 (9)
H8A	0.6205	0.7737	1.0716	0.101*
H8B	0.5494	0.8978	1.0144	0.101*
H8C	0.6267	0.9853	1.0900	0.101*
C9	0.93207 (12)	0.7991 (3)	0.93799 (19)	0.0373 (5)
H9	0.9428	0.8139	0.8665	0.045*
C10	1.11496 (13)	0.6599 (3)	1.09861 (19)	0.0368 (5)
C11	1.18830 (12)	0.6416 (3)	1.06983 (19)	0.0349 (5)
C12	1.25144 (14)	0.5798 (3)	1.1585 (2)	0.0480 (6)
H12	1.2454	0.5458	1.2310	0.058*
C13	1.32678 (14)	0.6166 (3)	1.0393 (2)	0.0466 (6)
C14	1.26868 (13)	0.6766 (3)	0.9465 (2)	0.0417 (6)

H14	1.2760	0.7070	0.8743	0.050*
C15	1.19900 (13)	0.6908 (3)	0.9626 (2)	0.0442 (6)
H15	1.1579	0.7342	0.9010	0.053*
H2A	0.0116 (15)	0.622 (4)	0.7072 (16)	0.080*
H3A	1.0529 (16)	0.689 (4)	0.9332 (12)	0.080*
H2B	−0.0190 (10)	0.508 (4)	0.772 (2)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0456 (4)	0.0869 (6)	0.1114 (7)	0.0044 (4)	0.0351 (4)	−0.0091 (5)
O1	0.0487 (10)	0.0743 (13)	0.0327 (9)	0.0007 (9)	0.0151 (8)	0.0016 (8)
O2	0.0577 (12)	0.0678 (14)	0.0418 (10)	−0.0083 (9)	0.0213 (9)	0.0025 (9)
N1	0.0388 (12)	0.0818 (18)	0.0530 (14)	0.0044 (11)	0.0106 (11)	−0.0059 (12)
N2	0.0387 (10)	0.0424 (12)	0.0390 (10)	−0.0011 (9)	0.0169 (9)	−0.0021 (9)
N3	0.0365 (10)	0.0489 (12)	0.0323 (10)	0.0006 (9)	0.0132 (9)	−0.0023 (9)
N4	0.0505 (14)	0.0716 (17)	0.0591 (15)	0.0073 (12)	0.0121 (12)	0.0024 (13)
C1	0.0377 (12)	0.0415 (14)	0.0447 (14)	−0.0013 (11)	0.0125 (11)	−0.0072 (11)
C2	0.0398 (13)	0.0474 (15)	0.0390 (13)	0.0032 (11)	0.0039 (11)	0.0001 (11)
C3	0.0478 (14)	0.0443 (15)	0.0355 (13)	0.0008 (11)	0.0139 (11)	0.0014 (11)
C4	0.0369 (12)	0.0358 (13)	0.0368 (12)	−0.0031 (10)	0.0115 (10)	−0.0003 (10)
C5	0.0405 (13)	0.0435 (15)	0.0335 (12)	0.0010 (11)	0.0105 (10)	0.0010 (10)
C6	0.0457 (14)	0.0552 (16)	0.0379 (13)	0.0029 (12)	0.0192 (11)	0.0003 (12)
C7	0.0423 (16)	0.134 (3)	0.069 (2)	0.0129 (18)	0.0032 (15)	−0.015 (2)
C8	0.0472 (16)	0.089 (2)	0.074 (2)	0.0065 (15)	0.0307 (15)	0.0002 (17)
C9	0.0425 (13)	0.0378 (14)	0.0337 (12)	−0.0016 (11)	0.0148 (10)	0.0005 (10)
C10	0.0410 (13)	0.0378 (14)	0.0330 (12)	−0.0036 (10)	0.0134 (10)	−0.0044 (10)
C11	0.0374 (12)	0.0315 (13)	0.0340 (12)	−0.0028 (9)	0.0084 (10)	−0.0026 (10)
C12	0.0472 (15)	0.0575 (17)	0.0383 (13)	0.0057 (12)	0.0119 (12)	0.0058 (12)
C13	0.0411 (13)	0.0458 (16)	0.0564 (16)	0.0008 (11)	0.0203 (12)	−0.0066 (12)
C14	0.0400 (13)	0.0502 (15)	0.0385 (12)	−0.0003 (11)	0.0172 (11)	0.0017 (11)
C15	0.0447 (13)	0.0502 (16)	0.0359 (12)	0.0002 (12)	0.0100 (11)	0.0043 (11)

Geometric parameters (Å, °)

Cl1—C13	1.753 (2)	C4—C9	1.447 (3)
O1—C10	1.228 (2)	C5—C6	1.375 (3)
O2—H2A	0.851 (10)	C5—H5	0.9300
O2—H2B	0.847 (10)	C6—H6	0.9300
N1—C1	1.378 (3)	C7—H7A	0.9600
N1—C7	1.443 (4)	C7—H7B	0.9600
N1—C8	1.443 (4)	C7—H7C	0.9600
N2—C9	1.281 (3)	C8—H8A	0.9600
N2—N3	1.391 (2)	C8—H8B	0.9600
N3—C10	1.341 (3)	C8—H8C	0.9600
N3—H3A	0.897 (10)	C9—H9	0.9300
N4—C13	1.336 (3)	C10—C11	1.497 (3)
N4—C12	1.354 (3)	C11—C12	1.380 (3)
C1—C6	1.400 (3)	C11—C15	1.386 (3)

C1—C2	1.400 (3)	C12—H12	0.9300
C2—C3	1.377 (3)	C13—C14	1.348 (3)
C2—H2	0.9300	C14—C15	1.359 (3)
C3—C4	1.393 (3)	C14—H14	0.9300
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.395 (3)		
H2A—O2—H2B	107 (2)	N1—C7—H7C	109.5
C1—N1—C7	120.3 (2)	H7A—C7—H7C	109.5
C1—N1—C8	121.1 (2)	H7B—C7—H7C	109.5
C7—N1—C8	118.4 (2)	N1—C8—H8A	109.5
C9—N2—N3	113.94 (18)	N1—C8—H8B	109.5
C10—N3—N2	119.10 (17)	H8A—C8—H8B	109.5
C10—N3—H3A	119.5 (19)	N1—C8—H8C	109.5
N2—N3—H3A	117.6 (19)	H8A—C8—H8C	109.5
C13—N4—C12	115.5 (2)	H8B—C8—H8C	109.5
N1—C1—C6	121.1 (2)	N2—C9—C4	122.6 (2)
N1—C1—C2	121.6 (2)	N2—C9—H9	118.7
C6—C1—C2	117.3 (2)	C4—C9—H9	118.7
C3—C2—C1	120.8 (2)	O1—C10—N3	123.4 (2)
C3—C2—H2	119.6	O1—C10—C11	120.9 (2)
C1—C2—H2	119.6	N3—C10—C11	115.60 (18)
C2—C3—C4	121.9 (2)	C12—C11—C15	117.0 (2)
C2—C3—H3	119.1	C12—C11—C10	117.8 (2)
C4—C3—H3	119.1	C15—C11—C10	125.1 (2)
C3—C4—C5	117.2 (2)	N4—C12—C11	123.4 (2)
C3—C4—C9	119.3 (2)	N4—C12—H12	118.3
C5—C4—C9	123.4 (2)	C11—C12—H12	118.3
C6—C5—C4	121.3 (2)	N4—C13—C14	125.8 (2)
C6—C5—H5	119.3	N4—C13—Cl1	116.4 (2)
C4—C5—H5	119.3	C14—C13—Cl1	117.8 (2)
C5—C6—C1	121.4 (2)	C13—C14—C15	117.4 (2)
C5—C6—H6	119.3	C13—C14—H14	121.3
C1—C6—H6	119.3	C15—C14—H14	121.3
N1—C7—H7A	109.5	C14—C15—C11	120.9 (2)
N1—C7—H7B	109.5	C14—C15—H15	119.5
H7A—C7—H7B	109.5	C11—C15—H15	119.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···O2^{i}	0.897 (10)	2.020 (14)	2.874 (3)	159 (3)
O2—H2B···O1^{ii}	0.847 (10)	2.118 (10)	2.957 (2)	171 (3)
O2—H2A···N2^{iii}	0.851 (10)	2.209 (10)	3.058 (3)	176 (3)
O2—H2A···O1^{iii}	0.851 (10)	2.61 (2)	3.064 (3)	115 (2)
C15—H15···O1^{iv}	0.93	2.46	3.284 (3)	148

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*+2; (iii) *x*−1, −*y*+3/2, *z*−1/2; (iv) *x*, −*y*+3/2, *z*−1/2.

Fig. 1

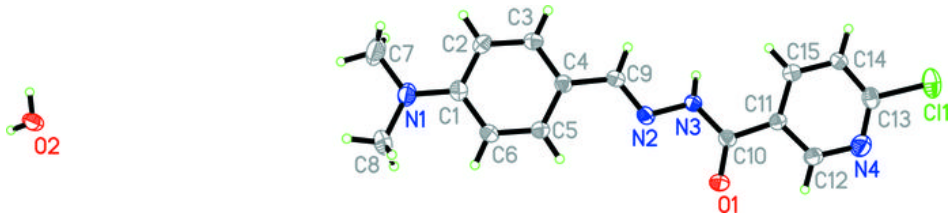
O2
C7
C2
C3
C4
C9
C15
C14
Cl1
N1
C1
C5
N2
N3
C11
C13
C8
C6
C10
N4
C12
O1

Fig. 2