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6-Bromo-2′-[4-(dimethylamino)benzylidene]nicotinohydrazide monohydrate

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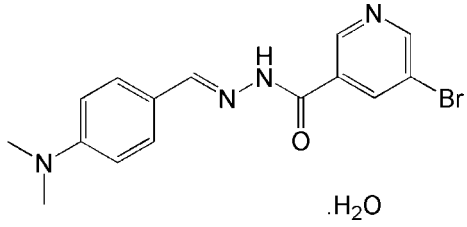
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.086; data-to-parameter ratio = 17.0.

The asymmetric unit of the title compound, $C_{15}H_{15}BrN_4O\cdot H_2O$, consists of a roughly planar 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 4.6 (2)°. In the crystal structure, molecules are linked through intermolecular O—H···O and O—H···N hydrogen bonds.

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

$C_{15}H_{15}BrN_4O\cdot H_2O$
M_r = 365.24
Monoclinic, $P2_1/n$
a = 10.535 (6) Å
b = 12.363 (7) Å
c = 12.889 (7) Å
β = 110.067 (6)°
V = 1576.8 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.62 mm^{-1}
T = 298 (2) K
0.23 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.584, T_{max} = 0.622
9266 measured reflections
3572 independent reflections
2312 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.086
S = 1.01
3572 reflections
210 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*C*···N1^{i}	0.841 (10)	2.171 (17)	2.948 (3)	154 (3)
O2—H2*B*···N3^{ii}	0.837 (10)	2.52 (2)	3.180 (3)	136 (3)
O2—H2*B*···O1^{ii}	0.837 (10)	2.209 (19)	2.956 (3)	149 (3)
N2—H2*A*···O2	0.893 (10)	2.045 (12)	2.921 (3)	167 (3)

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2149).

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supplementary materials

Acta Cryst. (2007). E**63**, o3739 [**doi:10.1107/S1600536807038512**]

6-Bromo-2'-[4-(dimethylamino)benzylidene]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 title compound, $C_{15}H_{15}BrN_4O\cdot H_2O$, consists of a roughly planar Schiff base molecule and a lattice 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═N3 bond length of 1.279 (3) Å conforms to the value for a double bond. The bond length of 1.338 (3) Å between atoms C6 and N2 is intermediate between an N—N single bond and an N═N double bond, because of conjugation effects in the molecule. The dihedral angle between the benzene ring and the pyridine ring is 4.6 (2)°. In the crystal structure, molecules are linked through intermolecular O—H···O and O—H···N hydrogen bonds, forming tetrammers (Fig. 2).

Experimental

4-Dimethylaminobenzaldehyde (0.1 mmol, 15.0 mg) and 5-bromonicotinic acid hydrazide (0.1 mmol, 21.6 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 3 days at room temperature.

Refinement

Atoms H2A, H2B and H2C 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 C—H distances of 0.93–0.96 Å, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(methyl C).

Figures

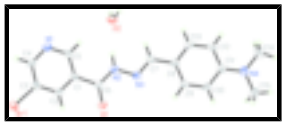

Fig. 1. The structure of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

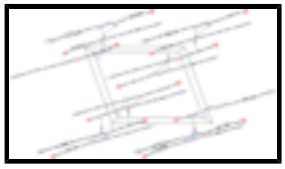

Fig. 2. Molecular packing as viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

6-Bromo-2'-[4-(dimethylamino)benzylidene]nicotinohydrazide monohydrate

Crystal data

$C_{15}H_{15}BrN_4O_1 \cdot H_2O_1$	$F_{000} = 744$
$M_r = 365.24$	$D_x = 1.538$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.535$ (6) Å	Cell parameters from 2425 reflections
$b = 12.363$ (7) Å	$\theta = 2.5$–24.3º
$c = 12.889$ (7) Å	$\mu = 2.62$ mm^{-1}
$\beta = 110.067$ (6)º	$T = 298$ (2) K
$V = 1576.8$ (15) Å^3	Block, colourless
$Z = 4$	0.23 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer	3572 independent reflections
Radiation source: fine-focus sealed tube	2312 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.032$
$T = 298$(2) K	$\theta_{max} = 27.5$º
ω scans	$\theta_{min} = 2.2$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12 \rightarrow 13$
$T_{min} = 0.584$, $T_{max} = 0.622$	$k = -10 \rightarrow 15$
9266 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.086$	$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.2692P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3572 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
210 parameters	$\Delta\rho_{min} = -0.32$ 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}
Br1	0.47397 (3)	0.42451 (2)	0.16814 (2)	0.06254 (13)
O1	0.6679 (2)	0.43639 (13)	0.61464 (15)	0.0656 (6)
O2	0.64188 (19)	0.03201 (13)	0.68079 (17)	0.0555 (5)
N1	0.5521 (2)	0.14776 (17)	0.35409 (18)	0.0521 (6)
N2	0.6671 (2)	0.26731 (15)	0.68069 (16)	0.0420 (5)
N3	0.7079 (2)	0.30497 (16)	0.78919 (16)	0.0424 (5)
N4	0.8832 (2)	0.31198 (18)	1.32054 (18)	0.0597 (6)
C1	0.6001 (2)	0.29484 (18)	0.48329 (19)	0.0373 (5)
C2	0.5636 (2)	0.36808 (19)	0.3968 (2)	0.0414 (6)
H2	0.5657	0.4420	0.4104	0.050*
C3	0.5239 (2)	0.32913 (19)	0.28971 (19)	0.0409 (6)
C4	0.5194 (2)	0.2193 (2)	0.2719 (2)	0.0481 (6)
H4	0.4923	0.1940	0.1996	0.058*
C5	0.5927 (2)	0.18524 (19)	0.4576 (2)	0.0457 (6)
H5	0.6172	0.1356	0.5153	0.055*
C6	0.6478 (2)	0.3389 (2)	0.5989 (2)	0.0422 (6)
C7	0.7264 (2)	0.2307 (2)	0.8620 (2)	0.0434 (6)
H7	0.7138	0.1589	0.8392	0.052*
C8	0.7665 (2)	0.25483 (18)	0.97939 (19)	0.0393 (5)
C9	0.7856 (2)	0.35918 (19)	1.0216 (2)	0.0437 (6)
H9	0.7731	0.4174	0.9734	0.052*
C10	0.8223 (2)	0.3783 (2)	1.1329 (2)	0.0457 (6)
H10	0.8330	0.4493	1.1584	0.055*
C11	0.8443 (2)	0.2932 (2)	1.20932 (19)	0.0427 (6)
C12	0.8246 (3)	0.1880 (2)	1.1664 (2)	0.0481 (6)
H12	0.8372	0.1294	1.2142	0.058*
C13	0.7868 (3)	0.17021 (19)	1.0544 (2)	0.0474 (6)
H13	0.7744	0.0995	1.0281	0.057*
C14	0.9084 (3)	0.2233 (3)	1.3982 (2)	0.0659 (8)
H14A	0.8265	0.1831	1.3854	0.099*
H14B	0.9390	0.2514	1.4722	0.099*
H14C	0.9765	0.1768	1.3886	0.099*
C15	0.9053 (4)	0.4204 (2)	1.3647 (3)	0.0746 (9)

H15A	0.9786	0.4529	1.3478	0.112*
H15B	0.9272	0.4178	1.4434	0.112*
H15C	0.8248	0.4626	1.3323	0.112*
H2A	0.648 (3)	0.1969 (10)	0.671 (2)	0.080*
H2B	0.7067 (18)	−0.006 (2)	0.720 (2)	0.080*
H2C	0.5687 (15)	−0.002 (2)	0.667 (3)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0862 (2)	0.05148 (19)	0.04669 (17)	0.00208 (16)	0.01863 (15)	0.00658 (13)
O1	0.1116 (16)	0.0305 (10)	0.0472 (11)	−0.0100 (10)	0.0175 (11)	−0.0051 (8)
O2	0.0598 (12)	0.0307 (9)	0.0676 (13)	−0.0010 (9)	0.0110 (11)	0.0033 (9)
N1	0.0674 (15)	0.0320 (11)	0.0513 (14)	0.0002 (10)	0.0134 (11)	−0.0068 (10)
N2	0.0529 (13)	0.0318 (10)	0.0420 (12)	−0.0031 (10)	0.0172 (10)	−0.0042 (9)
N3	0.0514 (13)	0.0379 (11)	0.0389 (11)	−0.0020 (9)	0.0168 (10)	−0.0056 (9)
N4	0.0813 (17)	0.0547 (14)	0.0408 (13)	0.0002 (13)	0.0181 (12)	−0.0024 (11)
C1	0.0397 (13)	0.0300 (12)	0.0440 (13)	−0.0039 (10)	0.0166 (11)	−0.0041 (10)
C2	0.0464 (15)	0.0295 (12)	0.0489 (15)	−0.0009 (11)	0.0171 (12)	−0.0045 (11)
C3	0.0422 (14)	0.0391 (14)	0.0412 (13)	0.0008 (11)	0.0142 (11)	0.0018 (11)
C4	0.0557 (16)	0.0424 (15)	0.0433 (15)	−0.0006 (13)	0.0131 (12)	−0.0094 (12)
C5	0.0551 (16)	0.0354 (14)	0.0448 (14)	0.0013 (12)	0.0147 (12)	−0.0022 (11)
C6	0.0486 (15)	0.0347 (14)	0.0436 (14)	−0.0022 (11)	0.0161 (12)	−0.0030 (11)
C7	0.0456 (14)	0.0369 (14)	0.0492 (15)	−0.0025 (11)	0.0183 (12)	−0.0044 (12)
C8	0.0413 (13)	0.0370 (13)	0.0405 (13)	−0.0008 (11)	0.0153 (11)	−0.0002 (10)
C9	0.0538 (15)	0.0333 (13)	0.0448 (14)	−0.0003 (12)	0.0176 (12)	0.0049 (11)
C10	0.0555 (16)	0.0324 (13)	0.0503 (15)	0.0013 (12)	0.0194 (13)	−0.0044 (11)
C11	0.0412 (14)	0.0441 (14)	0.0428 (14)	0.0009 (11)	0.0144 (11)	0.0003 (11)
C12	0.0602 (17)	0.0350 (14)	0.0474 (15)	−0.0014 (12)	0.0163 (13)	0.0083 (11)
C13	0.0589 (16)	0.0297 (13)	0.0517 (16)	−0.0028 (12)	0.0168 (13)	−0.0021 (11)
C14	0.069 (2)	0.083 (2)	0.0444 (16)	−0.0051 (17)	0.0179 (14)	0.0074 (15)
C15	0.099 (2)	0.068 (2)	0.0534 (18)	0.0004 (18)	0.0211 (17)	−0.0172 (15)

Geometric parameters (Å, °)

Br1—C3	1.886 (2)	C5—H5	0.9300
O1—C6	1.229 (3)	C7—C8	1.455 (3)
O2—H2B	0.837 (10)	C7—H7	0.9300
O2—H2C	0.841 (10)	C8—C9	1.388 (3)
N1—C4	1.331 (3)	C8—C13	1.390 (3)
N1—C5	1.337 (3)	C9—C10	1.372 (3)
N2—C6	1.338 (3)	C9—H9	0.9300
N2—N3	1.394 (3)	C10—C11	1.405 (3)
N2—H2A	0.893 (10)	C10—H10	0.9300
N3—C7	1.279 (3)	C11—C12	1.401 (4)
N4—C11	1.369 (3)	C12—C13	1.378 (3)
N4—C15	1.444 (3)	C12—H12	0.9300
N4—C14	1.446 (3)	C13—H13	0.9300
C1—C2	1.384 (3)	C14—H14A	0.9600

C1—C5	1.391 (3)	C14—H14B	0.9600
C1—C6	1.501 (3)	C14—H14C	0.9600
C2—C3	1.384 (3)	C15—H15A	0.9600
C2—H2	0.9300	C15—H15B	0.9600
C3—C4	1.376 (3)	C15—H15C	0.9600
C4—H4	0.9300		
H2B—O2—H2C	111 (2)	C9—C8—C13	117.4 (2)
C4—N1—C5	118.1 (2)	C9—C8—C7	123.3 (2)
C6—N2—N3	118.75 (19)	C13—C8—C7	119.3 (2)
C6—N2—H2A	124.3 (19)	C10—C9—C8	121.4 (2)
N3—N2—H2A	116.7 (19)	C10—C9—H9	119.3
C7—N3—N2	114.4 (2)	C8—C9—H9	119.3
C11—N4—C15	121.3 (2)	C9—C10—C11	121.6 (2)
C11—N4—C14	121.0 (2)	C9—C10—H10	119.2
C15—N4—C14	117.7 (2)	C11—C10—H10	119.2
C2—C1—C5	117.9 (2)	N4—C11—C12	121.4 (2)
C2—C1—C6	117.9 (2)	N4—C11—C10	121.7 (2)
C5—C1—C6	124.2 (2)	C12—C11—C10	116.9 (2)
C1—C2—C3	118.7 (2)	C13—C12—C11	120.8 (2)
C1—C2—H2	120.6	C13—C12—H12	119.6
C3—C2—H2	120.6	C11—C12—H12	119.6
C4—C3—C2	119.4 (2)	C12—C13—C8	121.9 (2)
C4—C3—Br1	119.66 (19)	C12—C13—H13	119.0
C2—C3—Br1	120.92 (18)	C8—C13—H13	119.0
N1—C4—C3	122.6 (2)	N4—C14—H14A	109.5
N1—C4—H4	118.7	N4—C14—H14B	109.5
C3—C4—H4	118.7	H14A—C14—H14B	109.5
N1—C5—C1	123.2 (2)	N4—C14—H14C	109.5
N1—C5—H5	118.4	H14A—C14—H14C	109.5
C1—C5—H5	118.4	H14B—C14—H14C	109.5
O1—C6—N2	123.2 (2)	N4—C15—H15A	109.5
O1—C6—C1	120.0 (2)	N4—C15—H15B	109.5
N2—C6—C1	116.8 (2)	H15A—C15—H15B	109.5
N3—C7—C8	122.1 (2)	N4—C15—H15C	109.5
N3—C7—H7	118.9	H15A—C15—H15C	109.5
C8—C7—H7	118.9	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2C···N1^{i}	0.841 (10)	2.171 (17)	2.948 (3)	154 (3)
O2—H2B···N3^{ii}	0.837 (10)	2.52 (2)	3.180 (3)	136 (3)
O2—H2B···O1^{ii}	0.837 (10)	2.209 (19)	2.956 (3)	149 (3)
N2—H2A···O2	0.893 (10)	2.045 (12)	2.921 (3)	167 (3)

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

Fig. 1

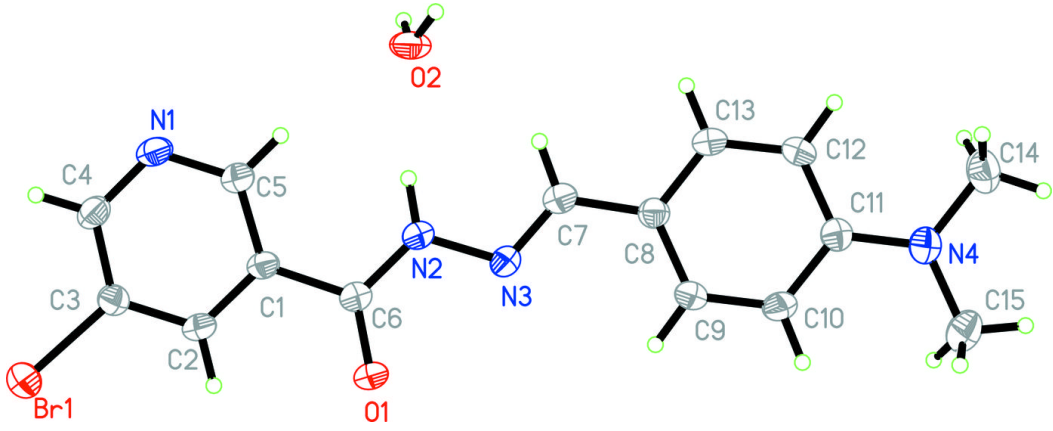
O2
N1
C4
C5
C13
C12
C14
C11
C7
C8
N2
N3
N4
C3
C1
C6
C15
C9
C10
C2
Br1
O1

Fig. 2