Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-(3-Bromo-4-methoxybenzylidene)-nicotinohydrazide monohydrate

Feng-Yu Bao,[a]* Xing-Shun Ding,[b] Hai-Yan Zhang[a] and Ying-Xia Zhou[a]

[a]Department of Applied Chemistry, College of Sciences, Henan Agricultural University, Zhengzhou 450002, People's Republic of China, and [b]Liangbaosi First School, Jiaxiang County, Shandong Province 272404, People's Republic of China
Correspondence e-mail: bfyu2008@126.com

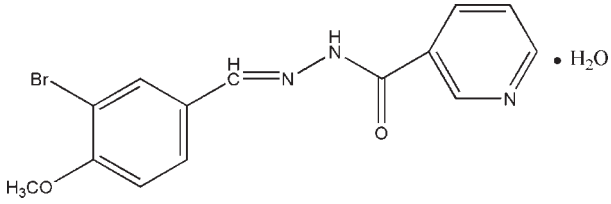
Received 21 August 2009; accepted 25 August 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.069; data-to-parameter ratio = 15.9.

In the title compound, $C_{14}H_{12}BrN_3O_2\cdot H_2O$, the benzene ring is oriented at a dihedral angle of 39.66 (11)° with respect to the pyridine ring. The solvent water molecule links with the organic compound *via* O—H···O, O—H···N and N—H···O hydrogen bonding.

Related literature

For applications of Schiff base compounds, see: Kahwa *et al.* (1986); Santos *et al.* (2001).



Experimental

Crystal data

$C_{14}H_{12}BrN_3O_2\cdot H_2O$ M_r = 352.18
Monoclinic, $P2_1/c$
a = 7.7979 (1) Å
b = 12.5678 (2) Å
c = 14.9419 (3) Å
β = 97.449 (1)°
V = 1451.98 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.85 mm^{-1}
T = 296 K
0.43 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.400, T_{max} = 0.535
12611 measured reflections
3149 independent reflections
2525 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.069
S = 1.01
3149 reflections
198 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N3	0.85 (2)	2.05 (2)	2.886 (2)	168 (2)
O2—H2B···O1^{i}	0.82 (3)	2.47 (3)	3.118 (2)	136 (2)
O2—H2B···N1^{i}	0.82 (3)	2.43 (3)	3.197 (2)	156 (3)
N2—H2···O2^{ii}	0.86	2.18	2.982 (2)	155

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2597).

References

Bruker (1998). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kahwa, I. A., Selbin, I., Hsieh, T. C. Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **118**, 179–185.
Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans*. pp. 838–844.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2282 [doi:10.1107/S160053680903400X]

N′-(3-Bromo-4-methoxybenzylidene)nicotinohydrazide monohydrate

Feng-Yu Bao, Xing-Shun Ding, Hai-Yan Zhang and Ying-Xia Zhou

S1. Comment

The chemistry of Schiff bases has attracted a great deal of interest in recent years. These compounds play an important role in the development of various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of our interest in the coordination chemistry of Schiff bases, we have recently synthesized the title compound and report here its crystal structure.

The title molecule crystallizes in the E conformation, with the C8—N1—N2—C9 torsion angle of 170.02 (19)°. The molecular is non-planar, there is a dihedral angle of 39.66 (11)° between the benzene ring and the pyridine ring. In the crystal structure, the lattice water molecule links with the organic compound *via* O—H···O, O—H···N and N—H···O hydrogen bonding.

S2. Experimental

Nicotinohydrazide (1 mmol, 0.137 g) was dissolved in ethanol (15 ml). The mixture was stirred for several min at 351 K, then 3-bromo-4-methoxybenzaldehyde (1 mmol, 0.215 g) in ethanol (8 mm l) was added dropwise and the mixture was refluxed for 2 h. The solid product was isolated and recrystallized from methanol, colourless single crystals were obtained after 3 d.

S3. Refinement

H atoms of water molecule are located in a difference Fourier map and refined isotropically, with O—H and H···H distances restrained to 0.85 (1) and 1.37 (2) Å, respectively. Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and refined with U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions with C—H = 0.93 and N—H = 0.86 Å, and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C,*N*).

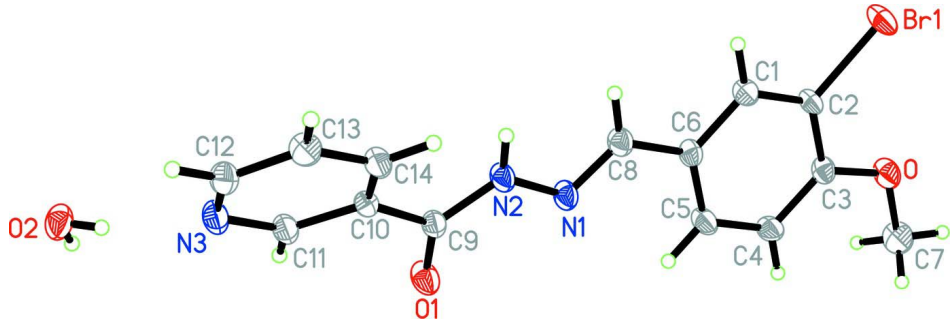


Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

N′-(3-Bromo-4-methoxybenzylidene)nicotinohydrazide monohydrate

Crystal data

$C_{14}H_{12}BrN_3O_2·H_2O$
$M_r = 352.18$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.7979$ (1) Å
$b = 12.5678$ (2) Å
$c = 14.9419$ (3) Å
$\beta = 97.449$ (1)°
$V = 1451.98$ (4) Å^3
$Z = 4$
$F(000) = 712$
$D_x = 1.611$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4706 reflections
$\theta = 3.1$–$27.0°$
$\mu = 2.85$ mm^{-1}
$T = 296$ K
Block, colourless
0.43 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.400$, $T_{max} = 0.535$
12611 measured reflections
3149 independent reflections
2525 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.0°$, $\theta_{min} = 3.1°$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 16$
$l = -19 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.069$
$S = 1.01$
3149 reflections
198 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 0.4288P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.38290 (3)	0.882695 (16)	0.139661 (15)	0.04825 (10)
O1	−0.0836 (2)	0.53946 (11)	−0.37189 (9)	0.0481 (4)
N1	0.0118 (2)	0.66340 (13)	−0.22564 (10)	0.0364 (4)

C2	0.2856 (2)	0.77093 (14)	0.06618 (12)	0.0319 (4)
C6	0.1504 (3)	0.70831 (15)	−0.07779 (12)	0.0330 (4)
N2	−0.0635 (2)	0.70309 (13)	−0.30777 (10)	0.0373 (4)
H2	−0.0792	0.7704	−0.3150	0.045*
C5	0.1501 (3)	0.60611 (15)	−0.04249 (14)	0.0389 (5)
H5A	0.1064	0.5501	−0.0792	0.047*
O	0.3473 (2)	0.65767 (12)	0.19064 (9)	0.0467 (4)
N3	−0.2839 (3)	0.66239 (14)	−0.61976 (11)	0.0445 (4)
C13	−0.3787 (3)	0.81441 (17)	−0.54493 (14)	0.0416 (5)
H13A	−0.4406	0.8779	−0.5484	0.050*
C1	0.2194 (3)	0.79090 (15)	−0.02219 (12)	0.0344 (4)
H1A	0.2207	0.8598	−0.0448	0.041*
C14	−0.2933 (3)	0.78026 (16)	−0.46379 (13)	0.0373 (4)
H14A	−0.2956	0.8207	−0.4118	0.045*
C12	−0.3718 (3)	0.75404 (18)	−0.62074 (13)	0.0438 (5)
H12A	−0.4306	0.7777	−0.6752	0.053*
C11	−0.2044 (3)	0.62932 (16)	−0.54085 (13)	0.0386 (5)
H11A	−0.1453	0.5649	−0.5392	0.046*
C10	−0.2041 (2)	0.68508 (15)	−0.46049 (12)	0.0313 (4)
C3	0.2824 (3)	0.66925 (15)	0.10238 (12)	0.0336 (4)
C4	0.2141 (3)	0.58670 (16)	0.04669 (13)	0.0386 (5)
H4A	0.2116	0.5180	0.0696	0.046*
C8	0.0758 (3)	0.73423 (16)	−0.17017 (13)	0.0373 (4)
H8A	0.0751	0.8048	−0.1890	0.045*
C7	0.3529 (4)	0.55397 (18)	0.22835 (15)	0.0573 (7)
H7A	0.4014	0.5573	0.2907	0.086*
H7B	0.4233	0.5089	0.1962	0.086*
H7C	0.2379	0.5255	0.2237	0.086*
C9	−0.1121 (3)	0.63517 (15)	−0.37660 (12)	0.0332 (4)
O2	−0.1669 (3)	0.57231 (13)	−0.77890 (10)	0.0521 (4)
H2A	−0.214 (3)	0.5932 (19)	−0.7336 (13)	0.068 (9)*
H2B	−0.110 (3)	0.519 (2)	−0.7634 (19)	0.072 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.06384 (18)	0.03325 (12)	0.04243 (13)	−0.00024 (10)	−0.01295 (10)	−0.01048 (9)
O1	0.0686 (11)	0.0340 (8)	0.0379 (8)	0.0075 (7)	−0.0074 (7)	−0.0026 (6)
N1	0.0453 (10)	0.0359 (8)	0.0259 (8)	0.0031 (8)	−0.0035 (7)	0.0004 (7)
C2	0.0353 (11)	0.0284 (9)	0.0298 (9)	−0.0003 (8)	−0.0036 (8)	−0.0066 (7)
C6	0.0352 (11)	0.0358 (10)	0.0265 (9)	0.0022 (8)	−0.0014 (8)	−0.0018 (8)
N2	0.0513 (11)	0.0315 (8)	0.0260 (8)	0.0006 (7)	−0.0065 (7)	0.0007 (6)
C5	0.0460 (13)	0.0336 (10)	0.0345 (10)	−0.0031 (9)	−0.0050 (9)	−0.0054 (8)
O	0.0678 (11)	0.0386 (8)	0.0292 (7)	0.0033 (7)	−0.0105 (7)	0.0033 (6)
N3	0.0600 (12)	0.0418 (10)	0.0293 (8)	−0.0012 (9)	−0.0031 (8)	−0.0037 (7)
C13	0.0437 (13)	0.0374 (10)	0.0419 (11)	0.0047 (9)	−0.0008 (9)	0.0031 (9)
C1	0.0391 (12)	0.0299 (9)	0.0323 (9)	0.0027 (8)	−0.0020 (8)	0.0009 (8)
C14	0.0414 (12)	0.0377 (10)	0.0322 (10)	−0.0007 (9)	0.0021 (8)	−0.0052 (8)

C12	0.0507 (14)	0.0457 (12)	0.0318 (10)	−0.0048 (10)	−0.0074 (9)	0.0049 (9)
C11	0.0499 (13)	0.0342 (10)	0.0300 (10)	−0.0010 (9)	−0.0011 (9)	−0.0027 (8)
C10	0.0328 (11)	0.0329 (9)	0.0270 (9)	−0.0053 (8)	−0.0003 (8)	−0.0013 (7)
C3	0.0371 (11)	0.0350 (10)	0.0270 (9)	0.0045 (9)	−0.0022 (8)	0.0004 (8)
C4	0.0500 (13)	0.0282 (9)	0.0353 (10)	−0.0017 (9)	−0.0028 (9)	0.0032 (8)
C8	0.0429 (12)	0.0372 (10)	0.0299 (9)	0.0001 (9)	−0.0020 (8)	0.0005 (8)
C7	0.0838 (19)	0.0451 (13)	0.0387 (12)	0.0038 (12)	−0.0086 (11)	0.0144 (10)
C9	0.0384 (11)	0.0335 (10)	0.0270 (9)	−0.0006 (8)	0.0016 (8)	−0.0018 (7)
O2	0.0845 (13)	0.0375 (8)	0.0337 (8)	0.0076 (9)	0.0059 (8)	0.0042 (7)

Geometric parameters (Å, º)

Br1—C2	1.8810 (17)	C13—C14	1.374 (3)
O1—C9	1.224 (2)	C13—H13A	0.9300
N1—C8	1.273 (2)	C1—H1A	0.9300
N1—N2	1.383 (2)	C14—C10	1.382 (3)
C2—C1	1.377 (2)	C14—H14A	0.9300
C2—C3	1.389 (3)	C12—H12A	0.9300
C6—C5	1.389 (3)	C11—C10	1.390 (3)
C6—C1	1.393 (3)	C11—H11A	0.9300
C6—C8	1.463 (3)	C10—C9	1.499 (3)
N2—C9	1.352 (2)	C3—C4	1.392 (3)
N2—H2	0.8600	C4—H4A	0.9300
C5—C4	1.383 (3)	C8—H8A	0.9300
C5—H5A	0.9300	C7—H7A	0.9600
O—C3	1.358 (2)	C7—H7B	0.9600
O—C7	1.418 (3)	C7—H7C	0.9600
N3—C11	1.326 (3)	O2—H2A	0.85 (2)
N3—C12	1.340 (3)	O2—H2B	0.82 (3)
C13—C12	1.370 (3)		
C8—N1—N2	114.26 (16)	C13—C12—H12A	118.6
C1—C2—C3	121.17 (17)	N3—C11—C10	123.96 (19)
C1—C2—Br1	119.76 (14)	N3—C11—H11A	118.0
C3—C2—Br1	119.07 (13)	C10—C11—H11A	118.0
C5—C6—C1	118.83 (17)	C14—C10—C11	117.46 (18)
C5—C6—C8	122.96 (17)	C14—C10—C9	125.20 (17)
C1—C6—C8	118.18 (17)	C11—C10—C9	117.30 (17)
C9—N2—N1	119.48 (16)	O—C3—C2	116.98 (17)
C9—N2—H2	120.3	O—C3—C4	124.49 (17)
N1—N2—H2	120.3	C2—C3—C4	118.54 (17)
C4—C5—C6	120.74 (18)	C5—C4—C3	120.46 (18)
C4—C5—H5A	119.6	C5—C4—H4A	119.8
C6—C5—H5A	119.6	C3—C4—H4A	119.8
C3—O—C7	118.23 (16)	N1—C8—C6	122.20 (18)
C11—N3—C12	117.32 (17)	N1—C8—H8A	118.9
C12—C13—C14	119.3 (2)	C6—C8—H8A	118.9
C12—C13—H13A	120.3	O—C7—H7A	109.5

C14—C13—H13A	120.3	O—C7—H7B	109.5
C2—C1—C6	120.25 (17)	H7A—C7—H7B	109.5
C2—C1—H1A	119.9	O—C7—H7C	109.5
C6—C1—H1A	119.9	H7A—C7—H7C	109.5
C13—C14—C10	119.09 (19)	H7B—C7—H7C	109.5
C13—C14—H14A	120.5	O1—C9—N2	123.08 (18)
C10—C14—H14A	120.5	O1—C9—C10	121.55 (17)
N3—C12—C13	122.83 (19)	N2—C9—C10	115.38 (16)
N3—C12—H12A	118.6	H2A—O2—H2B	107 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N3	0.85 (2)	2.05 (2)	2.886 (2)	168 (2)
O2—H2*B*···O1^{i}	0.82 (3)	2.47 (3)	3.118 (2)	136 (2)
O2—H2*B*···N1^{i}	0.82 (3)	2.43 (3)	3.197 (2)	156 (3)
N2—H2···O2^{ii}	0.86	2.18	2.982 (2)	155

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