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5-Bromo-*N*′-(4-hydroxybenzylidene)-nicotinohydrazide

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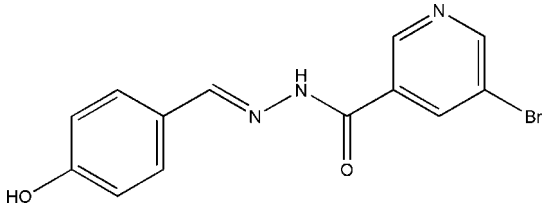
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.093; data-to-parameter ratio = 15.0.

The title Schiff base compound, $C_{13}H_{10}BrN_3O_2$, was derived from the condensation reaction of 4-hydroxybenzaldehyde with 5-bromonicotinohydrazide. The dihedral angle between the benzene and pyridine rings is 15.2 (3)°. In the crystal structure, molecules are linked through intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds, forming chains running along the *a* axis.

Related literature

For related literature, see: Tang (2006, 2007*a*,*b*); Allen *et al.* (1987).



Experimental

Crystal data

$C_{13}H_{10}BrN_3O_2$
M_r = 320.15
Orthorhombic, *Pbca*
a = 10.868 (2) Å
b = 8.0470 (16) Å
c = 29.209 (6) Å
V = 2554.5 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.22 mm^{-1}
T = 298 (2) K
0.30 × 0.27 × 0.27 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.445, T_{max} = 0.477
(expected range = 0.391–0.419)
19259 measured reflections
2635 independent reflections
1985 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.093
S = 1.03
2635 reflections
176 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.90 (3)	2.051 (16)	2.919 (3)	163 (4)
O1—H1···O2^{ii}	0.82	1.99	2.766 (3)	157
C12—H12···O1^{iii}	0.93	2.60	3.079 (3)	113

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

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 Jiaying University Research Fund is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2183).

References

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supplementary materials

Acta Cryst. (2007). E**63**, o4841 [**doi:10.1107/S1600536807060552**]

5-Bromo-*N*'-(4-hydroxybenzylidene)nicotinohydrazide

C.-B. Tang

Comment

Recently, the author has reported a few Schiff base complexes (Tang, 2006, 2007*a*,b). As a continuation of work in this area, the author reports herein the crystal structure of the title new Schiff base compound.

In the title compound (Fig. 1), the dihedral angle between the benzene ring and the pyridine ring is 15.2 (3)°. The torsion angles C6—C7—N1—N2, C7—N1—N2—C8, and N1—N2—C8—C9 are 0.1 (3), 6.4 (3), and 7.5 (3)°, respectively. All the bond lengths are within normal values (Allen *et al.*, 1987).

In the crystal structure of the compound, molecules are linked through N—H···O, O—H···O and C—H···O intermolecular hydrogen bonds (Table 1), forming chains running along the *a* axis (Fig. 2).

Experimental

4-Hydroxybenzaldehyde (0.1 mmol, 12.2 mg) and 5-bromonicotinic acid hydrazide (0.1 mmol, 21.6 mg) were dissolved in an ethanol solution (20 ml). The mixture was stirred at reflux for 10 min to give a clear yellowish solution. Yellowish needle-like crystals of the compound were formed by slow evaporation of the solvent for a few days.

Refinement

H2A was located from a difference Fourier map and refined isotropically, with N—H distance restrained to 0.90 (1) Å, and with the U_{iso}(H) fixed at 0.08 Å^2. Other H atoms were constrained to ideal geometries, with C—H = 0.93 Å, and with U_{iso}(H) set to 1.2U_{eq}(C).

Figures

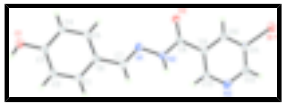

Fig. 1. The molecular structure of the compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

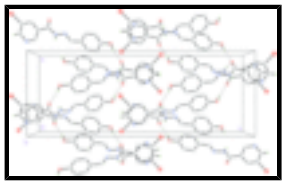

Fig. 2. Molecular packing of the compound as viewd down the b direction.

5-Bromo-*N*'-(4-hydroxybenzylidene)nicotinohydrazide

Crystal data

$C_{13}H_{10}BrN_3O_2$	$D_x = 1.665$ Mg m^{-3}
$M_r = 320.15$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 3414 reflections
$a = 10.868$ (2) Å	$\theta = 2.3$–24.5º
$b = 8.0470$ (16) Å	$\mu = 3.22$ mm^{-1}
$c = 29.209$ (6) Å	$T = 298$ (2) K
$V = 2554.5$ (9) Å^3	Cut from needle, yellow
$Z = 8$	0.30 × 0.27 × 0.27 mm
$F_{000} = 1280$	

Data collection

Bruker SMART CCD area-detector diffractometer	2635 independent reflections
Radiation source: fine-focus sealed tube	1985 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.051$
$T = 298$(2) K	$\theta_{max} = 26.5$º
ω scans	$\theta_{min} = 1.4$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -13\rightarrow13$
$T_{min} = 0.445$, $T_{max} = 0.477$	$k = -10\rightarrow10$
19259 measured reflections	$l = -36\rightarrow36$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 1.4703P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2635 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
176 parameters	$\Delta\rho_{min} = -0.41$ e Å^{-3}
1 restraint	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	0.52751 (3)	0.13520 (5)	0.591535 (11)	0.05060 (15)
O1	0.9032 (2)	0.2855 (3)	0.13604 (7)	0.0557 (7)
H1	0.9699	0.2569	0.1256	0.084*
O2	0.62259 (18)	0.2733 (2)	0.41182 (6)	0.0358 (5)
N1	0.7749 (2)	0.1576 (3)	0.34578 (8)	0.0354 (6)
N2	0.7782 (2)	0.1014 (3)	0.39053 (8)	0.0327 (6)
N3	0.8556 (2)	0.0176 (3)	0.53001 (8)	0.0411 (6)
C1	0.9661 (3)	0.0851 (4)	0.24568 (11)	0.0391 (7)
H1A	1.0241	0.0159	0.2593	0.047*
C2	0.9798 (3)	0.1293 (4)	0.20024 (10)	0.0396 (7)
H2	1.0463	0.0900	0.1833	0.048*
C3	0.8936 (3)	0.2329 (4)	0.18014 (10)	0.0362 (7)
C4	0.7925 (3)	0.2873 (4)	0.20510 (10)	0.0408 (7)
H4	0.7333	0.3539	0.1913	0.049*
C5	0.7802 (3)	0.2427 (4)	0.25011 (10)	0.0366 (7)
H5	0.7126	0.2802	0.2667	0.044*
C6	0.8670 (3)	0.1424 (4)	0.27146 (9)	0.0343 (6)
C7	0.8575 (3)	0.0957 (4)	0.31978 (10)	0.0370 (7)
H7	0.9129	0.0192	0.3318	0.044*
C8	0.7029 (3)	0.1711 (3)	0.42159 (9)	0.0292 (6)
C9	0.7235 (3)	0.1184 (3)	0.47016 (9)	0.0301 (6)
C10	0.6278 (3)	0.1405 (3)	0.50138 (9)	0.0326 (6)
H10	0.5519	0.1825	0.4922	0.039*
C11	0.6498 (3)	0.0980 (4)	0.54622 (9)	0.0341 (7)
C12	0.7616 (3)	0.0351 (4)	0.55927 (10)	0.0387 (7)
H12	0.7726	0.0033	0.5896	0.046*
C13	0.8356 (3)	0.0611 (4)	0.48648 (10)	0.0356 (7)
H13	0.9006	0.0524	0.4659	0.043*
H2A	0.824 (3)	0.011 (3)	0.3966 (13)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0535 (2)	0.0622 (3)	0.0361 (2)	0.00292 (17)	0.01472 (14)	0.00424 (16)
O1	0.0568 (15)	0.0783 (17)	0.0319 (12)	0.0191 (14)	0.0142 (11)	0.0169 (12)
O2	0.0381 (11)	0.0381 (11)	0.0312 (11)	0.0066 (10)	−0.0035 (9)	0.0020 (9)
N1	0.0356 (14)	0.0440 (15)	0.0266 (12)	0.0001 (11)	0.0021 (10)	0.0039 (10)
N2	0.0334 (14)	0.0394 (15)	0.0254 (12)	0.0045 (11)	0.0020 (10)	0.0037 (10)
N3	0.0397 (14)	0.0521 (16)	0.0313 (13)	0.0064 (12)	−0.0068 (11)	−0.0029 (12)
C1	0.0324 (16)	0.0488 (18)	0.0361 (16)	0.0071 (14)	−0.0007 (13)	0.0044 (15)
C2	0.0330 (16)	0.052 (2)	0.0337 (16)	0.0038 (15)	0.0067 (13)	−0.0034 (14)
C3	0.0399 (18)	0.0411 (18)	0.0277 (15)	−0.0024 (14)	0.0041 (13)	0.0029 (13)
C4	0.0372 (17)	0.0499 (19)	0.0354 (16)	0.0101 (15)	0.0043 (13)	0.0087 (14)
C5	0.0326 (16)	0.0429 (17)	0.0343 (16)	0.0048 (14)	0.0076 (13)	0.0000 (14)
C6	0.0338 (16)	0.0394 (17)	0.0298 (15)	−0.0006 (14)	0.0032 (12)	0.0012 (13)
C7	0.0362 (17)	0.0427 (18)	0.0322 (16)	0.0040 (14)	0.0022 (13)	0.0029 (13)
C8	0.0299 (15)	0.0293 (15)	0.0284 (14)	−0.0053 (12)	−0.0013 (11)	0.0022 (11)
C9	0.0325 (15)	0.0290 (15)	0.0288 (14)	−0.0021 (12)	−0.0024 (11)	−0.0007 (11)
C10	0.0310 (15)	0.0377 (16)	0.0291 (15)	0.0021 (13)	0.0002 (12)	0.0008 (13)
C11	0.0379 (16)	0.0362 (16)	0.0282 (14)	−0.0015 (13)	0.0041 (12)	−0.0007 (12)
C12	0.0460 (18)	0.0444 (18)	0.0256 (14)	0.0012 (14)	−0.0060 (13)	−0.0032 (13)
C13	0.0329 (16)	0.0420 (17)	0.0320 (15)	0.0005 (13)	−0.0011 (12)	−0.0024 (13)

Geometric parameters (Å, °)

Br1—C11	1.900 (3)	C3—C4	1.389 (4)
O1—C3	1.360 (3)	C4—C5	1.369 (4)
O1—H1	0.8200	C4—H4	0.9300
O2—C8	1.232 (3)	C5—C6	1.389 (4)
N1—C7	1.277 (4)	C5—H5	0.9300
N1—N2	1.384 (3)	C6—C7	1.464 (4)
N2—C8	1.344 (4)	C7—H7	0.9300
N2—H2A	0.90 (3)	C8—C9	1.498 (4)
N3—C13	1.337 (4)	C9—C13	1.387 (4)
N3—C12	1.339 (4)	C9—C10	1.395 (4)
C1—C2	1.382 (4)	C10—C11	1.375 (4)
C1—C6	1.393 (4)	C10—H10	0.9300
C1—H1A	0.9300	C11—C12	1.370 (4)
C2—C3	1.385 (4)	C12—H12	0.9300
C2—H2	0.9300	C13—H13	0.9300
C3—O1—H1	109.5	C1—C6—C7	119.4 (3)
C7—N1—N2	114.6 (2)	N1—C7—C6	121.5 (3)
C8—N2—N1	119.1 (2)	N1—C7—H7	119.3
C8—N2—H2A	123 (3)	C6—C7—H7	119.3
N1—N2—H2A	118 (3)	O2—C8—N2	123.6 (3)
C13—N3—C12	117.1 (3)	O2—C8—C9	120.9 (2)
C2—C1—C6	121.1 (3)	N2—C8—C9	115.5 (2)

C2—C1—H1A	119.4	C13—C9—C10	118.2 (3)
C6—C1—H1A	119.4	C13—C9—C8	123.4 (3)
C1—C2—C3	119.3 (3)	C10—C9—C8	118.2 (2)
C1—C2—H2	120.4	C11—C10—C9	117.5 (3)
C3—C2—H2	120.4	C11—C10—H10	121.3
O1—C3—C2	122.5 (3)	C9—C10—H10	121.3
O1—C3—C4	117.4 (3)	C12—C11—C10	120.8 (3)
C2—C3—C4	120.2 (3)	C12—C11—Br1	119.0 (2)
C5—C4—C3	119.9 (3)	C10—C11—Br1	120.2 (2)
C5—C4—H4	120.1	N3—C12—C11	122.5 (3)
C3—C4—H4	120.1	N3—C12—H12	118.7
C4—C5—C6	121.2 (3)	C11—C12—H12	118.7
C4—C5—H5	119.4	N3—C13—C9	123.8 (3)
C6—C5—H5	119.4	N3—C13—H13	118.1
C5—C6—C1	118.3 (3)	C9—C13—H13	118.1
C5—C6—C7	122.3 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O2^{i}	0.90 (3)	2.051 (16)	2.919 (3)	163 (4)
O1—H1···O2^{ii}	0.82	1.99	2.766 (3)	157
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Symmetry codes: (i) −*x*+3/2, *y*−1/2, *z*; (ii) *x*+1/2, *y*, −*z*+1/2; (iii) *x*, −*y*+1/2, *z*+1/2.

Fig. 1

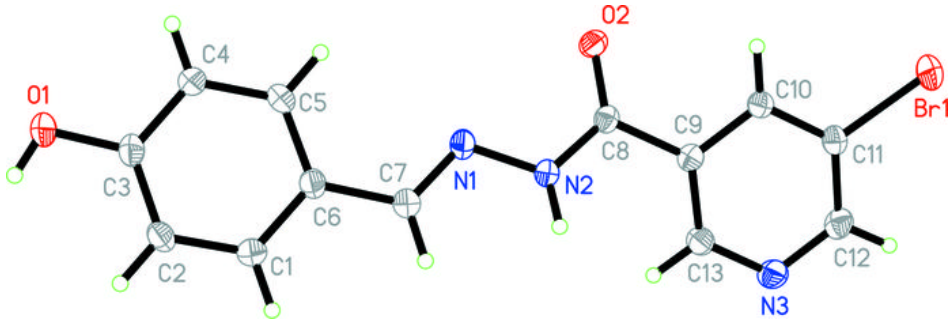

Fig. 2