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5-Bromo-*N*′-(2-hydroxybenzylidene)-nicotinohydrazide

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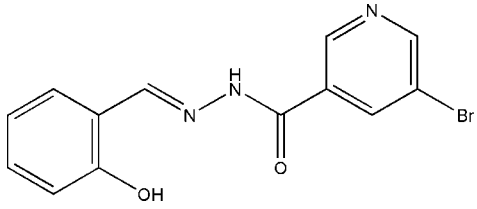
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.057; wR factor = 0.137; data-to-parameter ratio = 15.2.

The title Schiff base compound, $C_{13}H_{10}BrN_3O_2$, was derived from the condensation reaction of salicylaldehyde with 5-bromonicotinohydrazide. The dihedral angle between the benzene ring and the pyridine ring is 13.2 (4)°. In the crystal structure, molecules are linked through N—H···O and C—H···O intermolecular hydrogen bonds, forming chains running along the c axis.

Related literature

For related structures, see: Tang, (2006, 2007*a*,*b*); for reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{13}H_{10}BrN_3O_2$
M_r = 320.15
Monoclinic, $P2_1/c$
a = 10.940 (2) Å
b = 13.673 (3) Å
c = 8.6900 (17) Å
β = 92.88 (3)°
V = 1298.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.17 mm^{-1}
T = 293 (2) K
0.23 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.530, T_{max} = 0.570
(expected range = 0.493–0.531)
10307 measured reflections
2679 independent reflections
1498 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.137
S = 1.00
2679 reflections
176 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.899 (10)	1.937 (18)	2.814 (5)	164 (5)
O1—H1···N1	0.82	1.94	2.651 (5)	145
C7—H7···O2^{i}	0.93	2.44	3.195 (5)	138

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

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: SJ2400).

References

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supplementary materials

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5-Bromo-*N*'-(2-hydroxybenzylidene)nicotinohydrazide

C.-B. Tang

Comment

Recently, the author has reported the structures of several Schiff base complexes (Tang, 2006, 2007*a*,b) and, in continuation of work in this area, reports herein the structure of the title compound, (I), Fig. 1, a new Schiff base compound.

In the title compound (Fig. 1), the dihedral angle between the benzene ring and the pyridine ring is 13.2 (4)°. The torsion angles C1—C7—N1—N2, C7—N1—N2—C8, and N1—N2—C8—C9 are 1.7 (4), 10.8 (4), and 3.4 (4)°, 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 and C–H···O intermolecular hydrogen bonds (Table 1), forming chains running along the *c* axis (Fig. 2).

Experimental

Salicylaldehyde (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 over several days.

Refinement

H2 was located from a difference Fourier map and refined isotropically, with the 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, U_{iso}(H) = 1.2U_{eq}(C) and 0.82 Å, U_{iso} = 1.5U_{eq} (O) for the OH group..

Figures

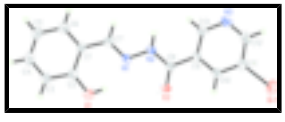

Fig. 1. The molecular structure of the compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

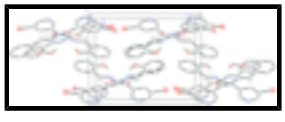

Fig. 2. Molecular packing of (I) with hydrogen bonds drawn as dashed lines.

5-Bromo-*N*'-(2-hydroxybenzylidene)nicotinohydrazide

Crystal data

$C_{13}H_{10}BrN_3O_2$ F_{000} = 640

M_r = 320.15	D_x = 1.638 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 929 reflections
a = 10.940 (2) Å	θ = 2.6–24.0º
b = 13.673 (3) Å	μ = 3.17 mm^{-1}
c = 8.6900 (17) Å	T = 293 (2) K
β = 92.88 (3)º	Cut from needle, colorless
V = 1298.2 (4) Å^3	0.23 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD area detector diffractometer	2679 independent reflections
Radiation source: fine-focus sealed tube	1498 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.079
T = 293(2) K	θ_{max} = 26.5º
ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→13
T_{min} = 0.530, T_{max} = 0.570	k = −17→17
10307 measured reflections	l = −10→10

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.137	$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 0.272P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = <0.001
2679 reflections	$\Delta\rho_{max}$ = 0.54 e Å^{-3}
176 parameters	$\Delta\rho_{min}$ = −0.59 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	0.85979 (6)	−0.19758 (4)	1.10713 (8)	0.0724 (3)
O1	0.5721 (4)	0.3989 (3)	0.7037 (4)	0.0600 (10)
H1	0.6197	0.3607	0.7482	0.090*
O2	0.7822 (3)	0.1592 (2)	0.8338 (4)	0.0491 (9)
N1	0.7140 (4)	0.3355 (3)	0.9392 (4)	0.0407 (10)
N2	0.7708 (4)	0.2650 (3)	1.0324 (4)	0.0427 (10)
N3	0.9790 (4)	0.0604 (4)	1.3039 (5)	0.0622 (13)
C1	0.6465 (4)	0.5014 (3)	0.9151 (5)	0.0390 (11)
C2	0.5799 (4)	0.4870 (4)	0.7732 (5)	0.0428 (12)
C3	0.5214 (5)	0.5671 (4)	0.7012 (6)	0.0560 (15)
H3	0.4772	0.5584	0.6080	0.067*
C4	0.5287 (5)	0.6577 (4)	0.7664 (7)	0.0613 (16)
H4	0.4900	0.7101	0.7163	0.074*
C5	0.5919 (6)	0.6727 (4)	0.9040 (7)	0.0612 (15)
H5	0.5957	0.7350	0.9467	0.073*
C6	0.6502 (5)	0.5954 (4)	0.9801 (6)	0.0520 (13)
H6	0.6920	0.6059	1.0745	0.062*
C7	0.7063 (4)	0.4212 (3)	0.9972 (5)	0.0430 (12)
H7	0.7403	0.4324	1.0959	0.052*
C8	0.7997 (4)	0.1786 (3)	0.9701 (5)	0.0390 (12)
C9	0.8541 (4)	0.1038 (3)	1.0785 (5)	0.0368 (11)
C10	0.8345 (4)	0.0060 (3)	1.0471 (5)	0.0419 (12)
H10	0.7877	−0.0131	0.9599	0.050*
C11	0.8848 (4)	−0.0628 (3)	1.1463 (5)	0.0417 (12)
C12	0.9565 (5)	−0.0326 (4)	1.2724 (6)	0.0585 (15)
H12	0.9907	−0.0799	1.3384	0.070*
C13	0.9273 (5)	0.1269 (4)	1.2081 (5)	0.0486 (13)
H13	0.9415	0.1926	1.2299	0.058*
H2	0.777 (5)	0.278 (4)	1.1340 (17)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0660 (4)	0.0424 (4)	0.1080 (6)	0.0007 (3)	−0.0041 (4)	0.0148 (3)
O1	0.075 (3)	0.049 (2)	0.054 (2)	0.008 (2)	−0.014 (2)	−0.0102 (19)
O2	0.077 (3)	0.0414 (19)	0.0277 (19)	0.0006 (17)	−0.0089 (17)	−0.0015 (15)
N1	0.051 (3)	0.038 (2)	0.033 (2)	0.0049 (19)	−0.0038 (19)	0.0082 (19)
N2	0.061 (3)	0.040 (2)	0.027 (2)	0.002 (2)	−0.004 (2)	0.0015 (19)
N3	0.070 (3)	0.066 (3)	0.049 (3)	0.017 (3)	−0.019 (2)	−0.005 (2)

C1	0.041 (3)	0.038 (3)	0.038 (3)	−0.003 (2)	0.001 (2)	0.003 (2)
C2	0.045 (3)	0.045 (3)	0.039 (3)	0.003 (2)	0.006 (2)	0.002 (2)
C3	0.066 (4)	0.058 (4)	0.044 (3)	0.018 (3)	−0.003 (3)	0.005 (3)
C4	0.069 (4)	0.044 (3)	0.071 (4)	0.018 (3)	0.010 (3)	0.012 (3)
C5	0.074 (4)	0.043 (3)	0.067 (4)	0.005 (3)	0.005 (3)	−0.002 (3)
C6	0.057 (4)	0.050 (3)	0.048 (3)	−0.002 (3)	−0.002 (3)	−0.005 (3)
C7	0.048 (3)	0.043 (3)	0.039 (3)	−0.005 (2)	0.002 (2)	−0.001 (2)
C8	0.042 (3)	0.043 (3)	0.032 (3)	−0.001 (2)	−0.002 (2)	0.003 (2)
C9	0.038 (3)	0.039 (3)	0.033 (3)	0.004 (2)	0.000 (2)	0.003 (2)
C10	0.041 (3)	0.044 (3)	0.041 (3)	−0.001 (2)	0.001 (2)	0.007 (2)
C11	0.037 (3)	0.041 (3)	0.047 (3)	0.003 (2)	0.006 (2)	0.005 (2)
C12	0.060 (4)	0.063 (4)	0.052 (4)	0.018 (3)	−0.003 (3)	0.015 (3)
C13	0.054 (3)	0.046 (3)	0.044 (3)	0.004 (3)	−0.011 (3)	−0.007 (3)

Geometric parameters (Å, °)

Br1—C11	1.892 (5)	C3—H3	0.9300
O1—C2	1.349 (5)	C4—C5	1.367 (8)
O1—H1	0.8200	C4—H4	0.9300
O2—C8	1.220 (5)	C5—C6	1.385 (7)
N1—C7	1.280 (5)	C5—H5	0.9300
N1—N2	1.386 (5)	C6—H6	0.9300
N2—C8	1.344 (6)	C7—H7	0.9300
N2—H2	0.899 (10)	C8—C9	1.493 (6)
N3—C12	1.321 (6)	C9—C10	1.380 (6)
N3—C13	1.339 (6)	C9—C13	1.385 (6)
C1—C6	1.404 (6)	C10—C11	1.372 (6)
C1—C2	1.414 (6)	C10—H10	0.9300
C1—C7	1.447 (6)	C11—C12	1.378 (7)
C2—C3	1.400 (6)	C12—H12	0.9300
C3—C4	1.362 (7)	C13—H13	0.9300
C2—O1—H1	109.5	C1—C6—H6	119.9
C7—N1—N2	116.3 (4)	N1—C7—C1	122.4 (4)
C8—N2—N1	118.9 (4)	N1—C7—H7	118.8
C8—N2—H2	124 (4)	C1—C7—H7	118.8
N1—N2—H2	117 (4)	O2—C8—N2	123.6 (4)
C12—N3—C13	117.1 (5)	O2—C8—C9	120.1 (4)
C6—C1—C2	118.8 (4)	N2—C8—C9	116.2 (4)
C6—C1—C7	119.5 (4)	C10—C9—C13	117.3 (4)
C2—C1—C7	121.7 (4)	C10—C9—C8	119.1 (4)
O1—C2—C3	118.8 (4)	C13—C9—C8	123.6 (4)
O1—C2—C1	122.2 (4)	C11—C10—C9	119.1 (5)
C3—C2—C1	119.0 (5)	C11—C10—H10	120.4
C4—C3—C2	120.6 (5)	C9—C10—H10	120.4
C4—C3—H3	119.7	C10—C11—C12	119.3 (5)
C2—C3—H3	119.7	C10—C11—Br1	120.3 (4)
C3—C4—C5	121.1 (5)	C12—C11—Br1	120.4 (4)
C3—C4—H4	119.4	N3—C12—C11	123.1 (5)
C5—C4—H4	119.4	N3—C12—H12	118.5

C4—C5—C6	120.3 (5)	C11—C12—H12	118.5
C4—C5—H5	119.9	N3—C13—C9	124.1 (5)
C6—C5—H5	119.9	N3—C13—H13	117.9
C5—C6—C1	120.2 (5)	C9—C13—H13	117.9
C5—C6—H6	119.9		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.899 (10)	1.937 (18)	2.814 (5)	164 (5)
O1—H1···N1	0.82	1.94	2.651 (5)	145
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Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2.

Fig. 1

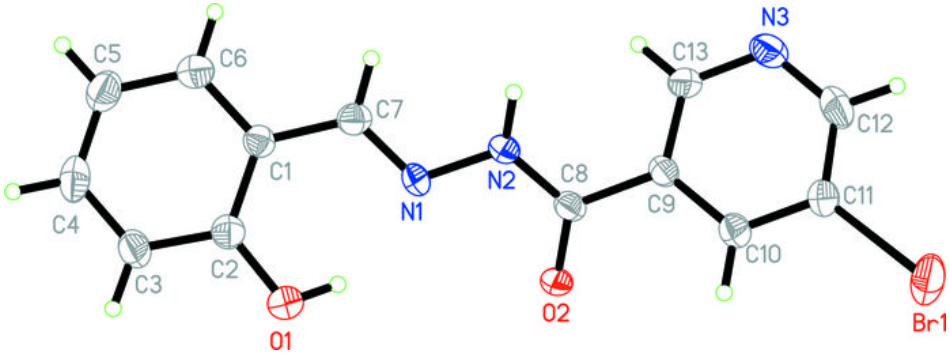

Fig. 2