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N'-(4-Bromobenzylidene)isonicotinohydrazide

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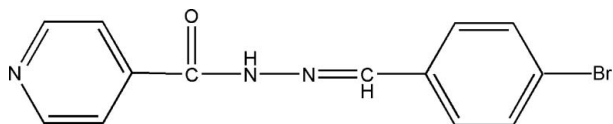
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Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.036; wR factor = 0.094; data-to-parameter ratio = 18.2.

The title compound, $\text{C}_{13}\text{H}_{10}\text{BrN}_3\text{O}$, was prepared by the reaction of isonicotinohydrazide and 4-bromobenzaldehyde. The dihedral angle between the benzene and pyridine rings is 8.60 (12)°. The crystal packing is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions.

Related literature

For background on Schiff bases, see: Chiu *et al.* (1998). For comparative bond-length data, see: Cimerman *et al.* (1997).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_{10}\text{BrN}_3\text{O}$ $M_r = 304.15$

Monoclinic, $P2_1/c$
 $a = 18.715$ (9) Å
 $b = 6.517$ (3) Å
 $c = 10.126$ (5) Å
 $\beta = 95.512$ (9)°
 $V = 1229.3$ (11) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.33$ mm⁻¹
 $T = 273$ (2) K
 $0.25 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: none
 7721 measured reflections

2992 independent reflections
 1914 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.094$
 $S = 1.01$
 2992 reflections

164 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.39$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.55$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O1}^{\text{i}}$	0.86	2.14	2.966 (3)	161
$\text{C12}-\text{H12A}\cdots\text{O1}^{\text{i}}$	0.93	2.60	3.377 (3)	142

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2659).

References

- Bruker (1997). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Chiu, P., Chen, B. & Cheng, K. F. (1998). *Tetrahedron Lett.* **39**, 9229–9232.
 Cimerman, Z., Galic, N. & Bosner, B. (1997). *Anal. Chim. Acta*, **343**, 145–153.
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supplementary materials

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N'-(4-Bromobenzylidene)isonicotinohydrazide

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Comment

Schiff bases have received considerable attention in the literature. They are attractive from several points of view, such as the possibility of analytical application (Cimerman *et al.*, 1997). As part of our search for new schiff base compounds we synthesized the title compound (I), and herein we report the crystal structure of (I).

In (I) (Fig. 1),

As seen in Fig. 1, the C12—N3 bond length of 1.276 (3) Å is comparable with C—N double bond [1.284 (2) Å] reported (Chiu *et al.*, 1998). In the title molecule, the benzene ring (C6—C10) is essentially planar with a maximum deviation of 0.009 (2) Å for C6 and C9, while the pyridine ring is planar, with a maximum deviation of 0.012 (2) Å for C3. The dihedral angle between the benzene and pyridine rings is 8.60 (12)°.

The crystal packing is stabilized by intermolecular C—H···O and N—H···O hydrogen-bonding interactions.

Experimental

A mixture of the isonicotinohydrazide (0.1 mol), and 4-bromobenzaldehyde (0.1 mol) was stirred in refluxing ethanol (20 mL) for 4 h to afford the title compound (0.082 mol, yield 82%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with N—H = 0.86 Å and C—H = 0.93 Å, and with $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

All H atoms were placed in idealized positions and refined with riding constraints, with N—H = 0.86 Å and C—H = 0.93 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

Figures

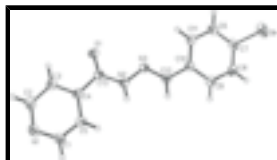


Fig. 1. An ORTEP view of the title compound (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

N¹-(4-Bromobenzylidene)isonicotinohydrazide

Crystal data

C ₁₃ H ₁₀ BrN ₃ O	$F_{000} = 608$
$M_r = 304.15$	$D_x = 1.643 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 18.715 (9) \text{ \AA}$	Cell parameters from 2167 reflections
$b = 6.517 (3) \text{ \AA}$	$\theta = 3.3\text{--}24.3^\circ$
$c = 10.126 (5) \text{ \AA}$	$\mu = 3.33 \text{ mm}^{-1}$
$\beta = 95.512 (9)^\circ$	$T = 273 (2) \text{ K}$
$V = 1229.3 (11) \text{ \AA}^3$	Block, yellow
$Z = 4$	$0.25 \times 0.20 \times 0.18 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	1914 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.034$
Monochromator: graphite	$\theta_{\text{max}} = 28.4^\circ$
$T = 273(2) \text{ K}$	$\theta_{\text{min}} = 2.2^\circ$
φ and ω scans	$h = -18 \rightarrow 25$
Absorption correction: none	$k = -7 \rightarrow 8$
7721 measured reflections	$l = -13 \rightarrow 13$
2992 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.1134P]$
$wR(F^2) = 0.094$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2992 reflections	$\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
164 parameters	$\Delta\rho_{\text{min}} = -0.55 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0072 (10)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br	0.038461 (17)	0.64201 (4)	0.85386 (4)	0.08031 (18)
N3	0.25010 (10)	1.5120 (3)	0.93706 (17)	0.0390 (4)
N2	0.29511 (10)	1.6713 (3)	0.90925 (18)	0.0386 (4)
H2A	0.3079	1.6847	0.8304	0.046*
O1	0.30311 (10)	1.7983 (3)	1.11880 (16)	0.0529 (4)
C7	0.10215 (13)	0.8680 (3)	0.8543 (3)	0.0457 (6)
C10	0.19040 (12)	1.2072 (3)	0.8503 (2)	0.0369 (5)
N1	0.46415 (11)	2.2729 (3)	0.8926 (2)	0.0545 (6)
C4	0.36913 (11)	1.9665 (3)	0.9610 (2)	0.0349 (5)
C13	0.31906 (12)	1.8058 (3)	1.0047 (2)	0.0362 (5)
C9	0.19163 (13)	1.0539 (4)	0.7549 (2)	0.0446 (6)
H9A	0.2229	1.0656	0.6893	0.054*
C12	0.23656 (13)	1.3861 (3)	0.8414 (2)	0.0417 (5)
H12A	0.2570	1.4094	0.7626	0.050*
C3	0.37459 (13)	2.1531 (3)	1.0273 (2)	0.0448 (6)
H3B	0.3471	2.1787	1.0972	0.054*
C5	0.41305 (13)	1.9365 (4)	0.8605 (2)	0.0455 (6)
H5A	0.4115	1.8140	0.8133	0.055*
C11	0.14423 (13)	1.1841 (4)	0.9499 (2)	0.0438 (6)
H11A	0.1429	1.2844	1.0150	0.053*
C8	0.14730 (14)	0.8850 (3)	0.7560 (3)	0.0488 (6)
H8A	0.1480	0.7843	0.6911	0.059*
C6	0.10088 (13)	1.0151 (4)	0.9526 (2)	0.0473 (6)
H6A	0.0708	0.9994	1.0199	0.057*
C2	0.42134 (14)	2.3003 (4)	0.9882 (3)	0.0515 (6)
H2B	0.4229	2.4263	1.0315	0.062*
C1	0.45933 (14)	2.0919 (4)	0.8314 (3)	0.0552 (7)
H1B	0.4891	2.0684	0.7646	0.066*

Atomic displacement parameters (\AA^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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supplementary materials

Br	0.0729 (3)	0.04607 (19)	0.1216 (4)	-0.01855 (14)	0.0075 (2)	-0.00034 (16)
N3	0.0430 (11)	0.0399 (10)	0.0350 (10)	-0.0045 (8)	0.0083 (8)	0.0038 (9)
N2	0.0456 (11)	0.0415 (10)	0.0301 (10)	-0.0063 (8)	0.0103 (8)	0.0034 (8)
O1	0.0674 (12)	0.0593 (10)	0.0341 (9)	-0.0110 (9)	0.0160 (8)	-0.0036 (8)
C7	0.0409 (13)	0.0333 (12)	0.0618 (16)	0.0013 (9)	-0.0016 (12)	0.0032 (11)
C10	0.0383 (12)	0.0367 (11)	0.0352 (12)	0.0003 (9)	0.0013 (10)	0.0032 (10)
N1	0.0568 (14)	0.0567 (14)	0.0492 (13)	-0.0143 (11)	0.0019 (11)	0.0077 (11)
C4	0.0363 (12)	0.0387 (12)	0.0294 (11)	0.0036 (9)	0.0022 (9)	0.0023 (9)
C13	0.0380 (13)	0.0390 (11)	0.0321 (12)	0.0051 (9)	0.0055 (10)	0.0024 (10)
C9	0.0509 (14)	0.0462 (13)	0.0375 (13)	0.0051 (11)	0.0081 (11)	-0.0005 (11)
C12	0.0469 (14)	0.0440 (13)	0.0355 (12)	-0.0016 (10)	0.0102 (11)	0.0037 (11)
C3	0.0457 (14)	0.0457 (14)	0.0437 (13)	0.0035 (10)	0.0086 (11)	-0.0041 (11)
C5	0.0482 (14)	0.0493 (13)	0.0400 (13)	-0.0052 (11)	0.0097 (11)	-0.0050 (11)
C11	0.0472 (14)	0.0443 (13)	0.0403 (13)	-0.0005 (10)	0.0060 (11)	-0.0057 (10)
C8	0.0571 (16)	0.0396 (13)	0.0487 (15)	0.0076 (11)	0.0005 (13)	-0.0090 (11)
C6	0.0431 (14)	0.0510 (14)	0.0489 (14)	-0.0025 (11)	0.0103 (11)	0.0065 (12)
C2	0.0532 (16)	0.0402 (13)	0.0596 (16)	-0.0022 (11)	-0.0029 (14)	-0.0006 (12)
C1	0.0541 (16)	0.0706 (18)	0.0428 (14)	-0.0117 (13)	0.0139 (12)	0.0010 (13)

Geometric parameters (Å, °)

Br—C7	1.894 (2)	C4—C13	1.500 (3)
N3—C12	1.276 (3)	C9—C8	1.379 (3)
N3—N2	1.383 (2)	C9—H9A	0.9300
N2—C13	1.349 (3)	C12—H12A	0.9300
N2—H2A	0.8600	C3—C2	1.382 (3)
O1—C13	1.222 (3)	C3—H3B	0.9300
C7—C8	1.371 (4)	C5—C1	1.382 (3)
C7—C6	1.384 (3)	C5—H5A	0.9300
C10—C9	1.391 (3)	C11—C6	1.370 (3)
C10—C11	1.398 (3)	C11—H11A	0.9300
C10—C12	1.459 (3)	C8—H8A	0.9300
N1—C2	1.326 (3)	C6—H6A	0.9300
N1—C1	1.331 (3)	C2—H2B	0.9300
C4—C5	1.382 (3)	C1—H1B	0.9300
C4—C3	1.388 (3)		
C12—N3—N2	114.10 (18)	C10—C12—H12A	118.6
C13—N2—N3	120.59 (18)	C4—C3—C2	119.3 (2)
C13—N2—H2A	119.7	C4—C3—H3B	120.4
N3—N2—H2A	119.7	C2—C3—H3B	120.4
C8—C7—C6	121.4 (2)	C4—C5—C1	118.9 (2)
C8—C7—Br	119.52 (19)	C4—C5—H5A	120.6
C6—C7—Br	119.09 (19)	C1—C5—H5A	120.6
C9—C10—C11	118.4 (2)	C6—C11—C10	120.6 (2)
C9—C10—C12	118.8 (2)	C6—C11—H11A	119.7
C11—C10—C12	122.8 (2)	C10—C11—H11A	119.7
C2—N1—C1	116.1 (2)	C7—C8—C9	118.9 (2)
C5—C4—C3	117.3 (2)	C7—C8—H8A	120.5
C5—C4—C13	123.5 (2)	C9—C8—H8A	120.5

C3—C4—C13	119.2 (2)	C11—C6—C7	119.5 (2)
O1—C13—N2	123.8 (2)	C11—C6—H6A	120.3
O1—C13—C4	121.6 (2)	C7—C6—H6A	120.3
N2—C13—C4	114.59 (18)	N1—C2—C3	124.0 (2)
C8—C9—C10	121.2 (2)	N1—C2—H2B	118.0
C8—C9—H9A	119.4	C3—C2—H2B	118.0
C10—C9—H9A	119.4	N1—C1—C5	124.4 (2)
N3—C12—C10	122.9 (2)	N1—C1—H1B	117.8
N3—C12—H12A	118.6	C5—C1—H1B	117.8

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2A \cdots O1 ⁱ	0.86	2.14	2.966 (3)	161
C12—H12A \cdots O1 ⁱ	0.93	2.60	3.377 (3)	142

Symmetry codes: (i) $x, -y+7/2, z-1/2$.

Fig. 1

