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2-[(6-Bromo-2-pyridyl)amino]pyridine N-oxide

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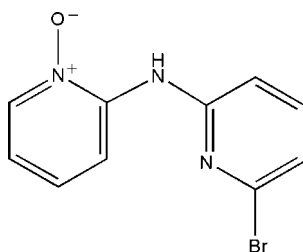
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.030; wR factor = 0.055; data-to-parameter ratio = 13.3.

In the crystal structure of the title compound, $\text{C}_{10}\text{H}_8\text{BrN}_3\text{O}$, the dihedral angle between the two pyridine rings is 2.48 (2)°. A weak intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond is present.

Related literature

For similar structures, see: Wu (2007); Liu & Wen (2007).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{BrN}_3\text{O}$
 $M_r = 266.09$

Monoclinic, $P2_1/n$
 $a = 13.402$ (3) Å

$b = 5.3016$ (10) Å
 $c = 14.562$ (3) Å
 $\beta = 103.498$ (3)°
 $V = 1006.1$ (4) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 4.06$ mm⁻¹
 $T = 298$ (2) K
 $0.52 \times 0.13 \times 0.11$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\min} = 0.545$, $T_{\max} = 0.650$

5867 measured reflections
1850 independent reflections
1263 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.054$
 $S = 0.98$
1850 reflections
139 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O1}$	0.899 (10)	2.12 (2)	2.542 (3)	107.8 (19)

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2103).

References

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supplementary materials

Acta Cryst. (2008). E64, o1557 [doi:10.1107/S1600536808022058]

2-[(6-Bromo-2-pyridyl)amino]pyridine *N*-oxide

Y.-H. Chen, Y.-Q. Liu and X.-R. Zeng

Comment

The structure determination was performed as a part of a project on the synthesis, structures and properties of new polypyridylamine *N*-oxides. In the crystal structure both pyridine rings are nearly coplanar with a dihedral angle of 2.48 (2) °. The molecules are connected into dimers via intermolecular N-H...O hydrogen bonding and the dimers are stacked in the direction of the crystallographic *b*-axis in a sandwich-herringbone structure.

Experimental

A mixture of 2.56 g 6-bromo-*N*-(pyridin-2-yl)pyridin-2-amine (0.01 mol), 8 ml of H₂O₂(30%), 8 ml of acetic acid and 30 ml of methanol were heated under reflux for about six hours. Afterwards the methanol was removed under reduced pressure. The crude yellow coloured product was recrystallized from methanol.

Refinement

The C-H atoms were positioned with idealized geometry and refined isotropic with C-H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$; The H atoms of the amino groups was located in difference map and was refined isotropic with $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{N})$ and a N-H distance restrained to 0.90 Å.

Figures

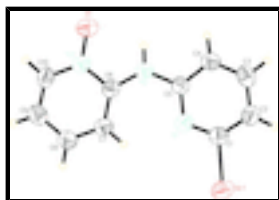


Fig. 1. The molecular structure of (I) with labelling and displacement ellipsoids drawn at the 50% probability level.

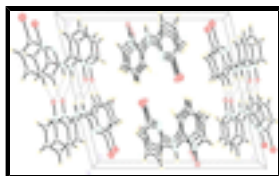


Fig. 2. Crystal structure of the title compound, viewed along the *c* axis. Hydrogen bonding is shown as dashed lines.

2-[(6-Bromo-2-pyridyl)amino]pyridine *N*-oxide

Crystal data

C₁₀H₈BrN₃O

$F(000) = 528.0$

supplementary materials

$$M_r = 266.09$$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$$a = 13.402 (3) \text{ \AA}$$

$$b = 5.3016 (10) \text{ \AA}$$

$$c = 14.562 (3) \text{ \AA}$$

$$\beta = 103.498 (3)^\circ$$

$$V = 1006.1 (4) \text{ \AA}^3$$

$$Z = 4$$

$$D_x = 1.757 \text{ Mg m}^{-3}$$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2721 reflections

$$\theta = 2.4\text{--}26.8^\circ$$

$$\mu = 4.06 \text{ mm}^{-1}$$

$$T = 298 \text{ K}$$

Block, yellow

$$0.52 \times 0.13 \times 0.11 \text{ mm}$$

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 1997)

$$T_{\min} = 0.545, T_{\max} = 0.650$$

5867 measured reflections

1850 independent reflections

1263 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.049$$

$$\theta_{\max} = 25.5^\circ, \theta_{\min} = 1.9^\circ$$

$$h = -16 \rightarrow 15$$

$$k = -6 \rightarrow 6$$

$$l = -17 \rightarrow 17$$

Refinement

Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.029$$

$$wR(F^2) = 0.054$$

$$S = 0.98$$

1850 reflections

139 parameters

1 restraint

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

$$w = 1/[\sigma^2(F_o^2) + (0.0029P)^2 + 0.3286P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.40 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.28 \text{ e \AA}^{-3}$$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.92158 (2)	1.17815 (7)	-0.12095 (2)	0.06564 (15)
C1	0.7817 (2)	1.1290 (5)	-0.11489 (19)	0.0432 (7)
C2	0.7096 (2)	1.2903 (6)	-0.1629 (2)	0.0517 (8)
H2	0.7262	1.4226	-0.1986	0.062*
C3	0.6096 (2)	1.2463 (6)	-0.1558 (2)	0.0539 (8)
H3	0.5567	1.3500	-0.1874	0.065*
C4	0.5892 (2)	1.0499 (5)	-0.1021 (2)	0.0478 (7)
H4	0.5224	1.0174	-0.0973	0.057*
C5	0.6696 (2)	0.9003 (5)	-0.05497 (18)	0.0387 (7)
C6	0.7173 (2)	0.5429 (5)	0.05848 (18)	0.0395 (7)
C7	0.8226 (2)	0.5275 (5)	0.07141 (19)	0.0475 (7)
H7	0.8566	0.6394	0.0401	0.057*
C8	0.8775 (2)	0.3478 (6)	0.1303 (2)	0.0512 (8)
H8	0.9484	0.3392	0.1390	0.061*
C9	0.8273 (2)	0.1797 (6)	0.1766 (2)	0.0510 (7)
H9	0.8636	0.0556	0.2158	0.061*
C10	0.7240 (2)	0.1999 (6)	0.1637 (2)	0.0489 (7)
H10	0.6898	0.0883	0.1950	0.059*
H2A	0.5830 (5)	0.675 (5)	-0.0026 (18)	0.059*
N1	0.76650 (17)	0.9372 (4)	-0.06143 (15)	0.0429 (6)
N2	0.65001 (17)	0.7072 (4)	0.00308 (17)	0.0451 (6)
N3	0.66939 (17)	0.3773 (4)	0.10671 (16)	0.0426 (6)
O1	0.57060 (15)	0.3965 (4)	0.09841 (14)	0.0631 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0485 (2)	0.0779 (3)	0.0748 (2)	-0.01014 (19)	0.02297 (15)	0.0068 (2)
C1	0.0412 (17)	0.048 (2)	0.0399 (17)	-0.0028 (14)	0.0086 (13)	-0.0046 (15)
C2	0.052 (2)	0.050 (2)	0.0534 (19)	-0.0026 (16)	0.0119 (15)	0.0078 (16)
C3	0.053 (2)	0.053 (2)	0.0493 (19)	0.0087 (15)	0.0006 (15)	0.0044 (15)
C4	0.0362 (17)	0.0498 (19)	0.0561 (19)	0.0003 (15)	0.0085 (14)	0.0016 (16)
C5	0.0397 (17)	0.0356 (18)	0.0400 (16)	-0.0020 (13)	0.0077 (13)	-0.0041 (13)
C6	0.0430 (17)	0.0347 (17)	0.0404 (16)	-0.0026 (14)	0.0088 (13)	-0.0023 (14)
C7	0.0388 (17)	0.0476 (19)	0.0568 (19)	-0.0020 (14)	0.0128 (14)	0.0059 (16)
C8	0.0418 (17)	0.053 (2)	0.058 (2)	0.0003 (16)	0.0086 (14)	0.0043 (18)
C9	0.052 (2)	0.0442 (18)	0.0530 (19)	0.0030 (17)	0.0042 (14)	0.0027 (16)
C10	0.057 (2)	0.0437 (19)	0.0451 (18)	-0.0067 (17)	0.0110 (14)	0.0077 (16)
N1	0.0427 (15)	0.0405 (15)	0.0463 (15)	-0.0035 (11)	0.0121 (11)	0.0023 (12)
N2	0.0317 (13)	0.0451 (15)	0.0590 (15)	-0.0018 (12)	0.0112 (11)	0.0079 (13)
N3	0.0393 (15)	0.0444 (16)	0.0453 (14)	-0.0050 (12)	0.0125 (11)	-0.0004 (12)
O1	0.0411 (13)	0.0767 (17)	0.0725 (15)	-0.0049 (11)	0.0153 (11)	0.0112 (11)

supplementary materials

Geometric parameters (Å, °)

Br1—C1	1.916 (3)	C6—N3	1.374 (3)
C1—N1	1.325 (3)	C6—C7	1.382 (3)
C1—C2	1.356 (4)	C7—C8	1.374 (4)
C2—C3	1.388 (4)	C7—H7	0.9300
C2—H2	0.9300	C8—C9	1.383 (4)
C3—C4	1.367 (4)	C8—H8	0.9300
C3—H3	0.9300	C9—C10	1.357 (4)
C4—C5	1.384 (4)	C9—H9	0.9300
C4—H4	0.9300	C10—N3	1.351 (3)
C5—N1	1.338 (3)	C10—H10	0.9300
C5—N2	1.391 (3)	N2—H2A	0.899 (10)
C6—N2	1.372 (3)	N3—O1	1.305 (3)
N1—C1—C2	126.8 (3)	C8—C7—H7	119.8
N1—C1—Br1	114.7 (2)	C6—C7—H7	119.8
C2—C1—Br1	118.5 (2)	C7—C8—C9	120.1 (3)
C1—C2—C3	116.1 (3)	C7—C8—H8	120.0
C1—C2—H2	122.0	C9—C8—H8	120.0
C3—C2—H2	122.0	C10—C9—C8	118.5 (3)
C4—C3—C2	119.7 (3)	C10—C9—H9	120.7
C4—C3—H3	120.1	C8—C9—H9	120.7
C2—C3—H3	120.1	N3—C10—C9	121.9 (3)
C3—C4—C5	118.9 (3)	N3—C10—H10	119.0
C3—C4—H4	120.5	C9—C10—H10	119.0
C5—C4—H4	120.5	C1—N1—C5	116.0 (2)
N1—C5—C4	122.5 (3)	C6—N2—C5	129.2 (2)
N1—C5—N2	118.3 (2)	C6—N2—H2A	116.1 (17)
C4—C5—N2	119.1 (3)	C5—N2—H2A	114.2 (17)
N2—C6—N3	112.6 (2)	O1—N3—C10	120.2 (2)
N2—C6—C7	128.9 (3)	O1—N3—C6	119.2 (2)
N3—C6—C7	118.5 (3)	C10—N3—C6	120.5 (2)
C8—C7—C6	120.4 (3)		

Hydrogen-bond geometry (Å, °)

<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	0.90 (1)	2.12 (2)	2.542 (3)	108.(2)

Fig. 1

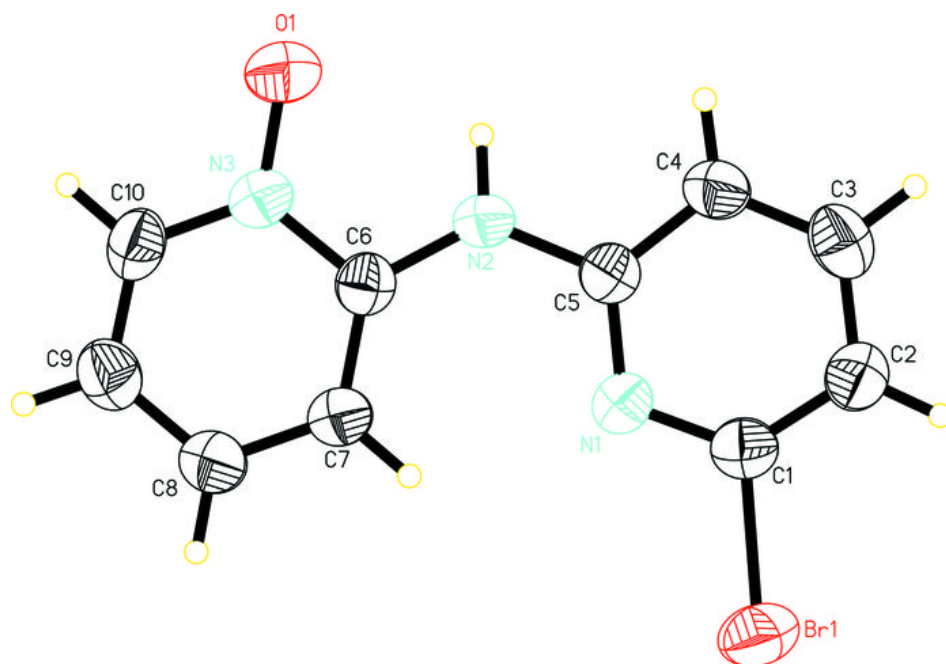


Fig. 2

