Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Yun-Hua Chen,[a] Yin-Qiu Liu[b] and Xi-Rui Zeng[b]*

[a]Zhejiang Hisun Pharmaceutical Co. Ltd, JiangXi Province Key Laboratory of Coordination Chemistry, 343009 Ji'an, JiangXi, People's Republic of China, and [b]College of Chemistry and Chemical Engineering, JiangXi Province Key Laboratory of Coordination Chemistry, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China
Correspondence e-mail: xiruizeng@hotmail.com

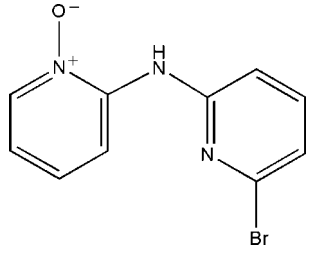
Received 25 April 2008; accepted 15 July 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–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, $C_{10}H_8BrN_3O$, the dihedral angle between the two pyridine rings is 2.48 (2)°. A weak intramolecular N—H···O hydrogen bond is present.

Related literature

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



Experimental

Crystal data

$C_{10}H_8BrN_3O$
M_r = 266.09
Monoclinic, $P2_1/n$
a = 13.402 (3) Å
b = 5.3016 (10) Å
c = 14.562 (3) Å
β = 103.498 (3)°
V = 1006.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.06 mm^{-1}
T = 298 (2) K
0.52 × 0.13 × 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_{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
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···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*.

The authors are grateful for support from the Key Laboratory of Coordination Chemistry, JingGangShan University, China

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

References

Bruker (1997). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Liu, Y.-Q. & Wen, H.-R. (2007). *Acta Cryst.* E**63**, o4690.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wu, J. (2007). *Acta Cryst.* E**63**, o4413.

supporting information

Acta Cryst. (2008). E**64**, o1557 [doi:10.1107/S1600536808022058]

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

Yun-Hua Chen, Yin-Qiu Liu and Xi-Rui Zeng

S1. 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 an 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.

S2. Experimental

A mixture of 2.56 g 6-bromo-*N*-(pyridin-2-yl)pyridin-2-amine (0.01 mol), 8 ml of $H_{2O}2$(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.

S3. Refinement

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

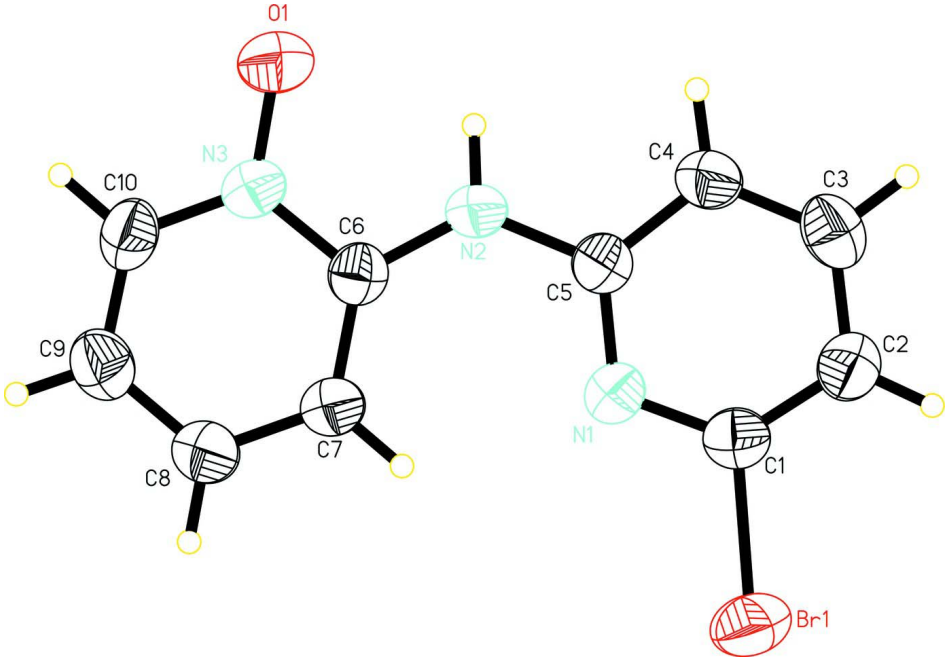


Figure 1

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

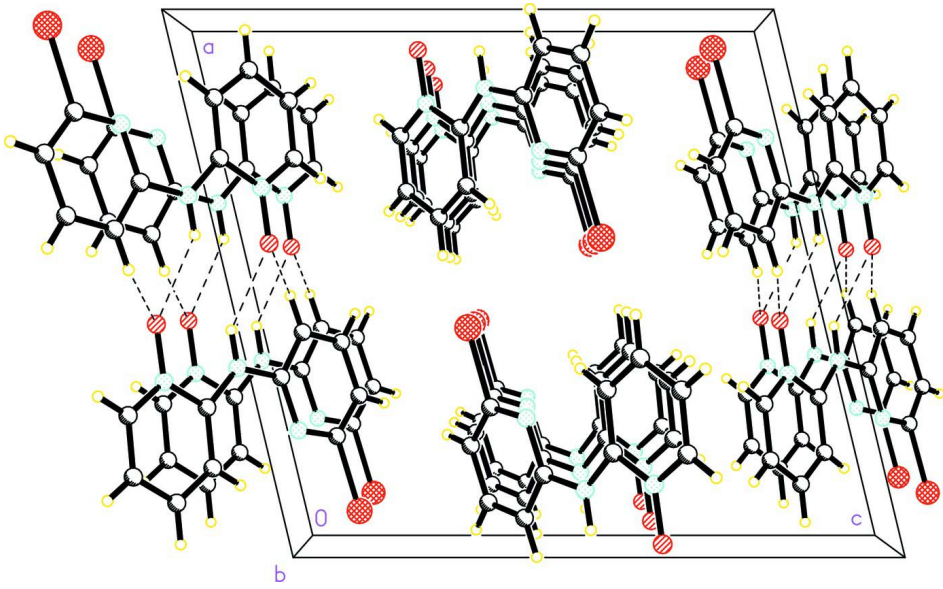


Figure 2

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

$C_{10}H_8BrN_3O$
$M_r = 266.09$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 13.402$ (3) Å
$b = 5.3016$ (10) Å
$c = 14.562$ (3) Å
$\beta = 103.498$ (3)°
$V = 1006.1$ (4) Å^3
$Z = 4$
$F(000) = 528.0$
$D_x = 1.757$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2721 reflections
$\theta = 2.4$–$26.8°$
$\mu = 4.06$ mm^{-1}
$T = 298$ K
Block, yellow
0.52 × 0.13 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
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 $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.9°$
$h = -16 \rightarrow 15$
$k = -6 \rightarrow 6$
$l = -17 \rightarrow 17$

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
$w = 1/[\sigma^2(F_o^2) + (0.0029P)^2 + 0.3286P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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)

Geometric parameters (Å, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.90 (1)	2.12 (2)	2.542 (3)	108 (2)