Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(6-Bromo-3-pyridyl)-8-methylimidazo-[1,2-*a*]pyrazine

Buwen Huang,[a] Eugene Rui,[a] Martin Wythes,[a] Pei-Pei Kung,[a] Curtis Moore,[b] Arnold L. Rheingold[b] and Alex Yanovsky[a]*

[a]Pfizer Global Research and Development, La Jolla Labs, 10770 Science Center Drive, San Diego, CA 92121, USA, and [b]Department of Chemistry and Biochemistry, University of California, San Diego, 9500 Gilman Drive, La Jolla, CA 92093, USA
Correspondence e-mail: alex.yanovsky@pfizer.com

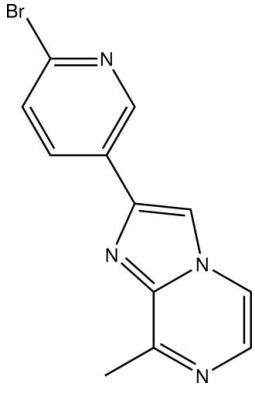
Received 1 June 2010; accepted 14 June 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.057; wR factor = 0.154; data-to-parameter ratio = 17.2.

The structure of the title compound, $C_{12}H_9BrN_4$, prepared by the reaction of 2-bromo-1-(6-bromo-3-pyridyl)ethanone with 2-amino-3-methylpyrazine indicates that the compound with the bromopyridyl substituent at position 2 of the imidazopyrazine fused-ring system represents the major product of this reaction. The plane of the pyridine ring forms a dihedral angle of 16.2 (2)° with the essentially planar (r.m.s. deviation = 0.006 Å) imidazopyrazine system. In the crystal, molecules are linked by weak C—H···N interactions.

Related literature

For the structure of the related imidazo(1,2-*a*)pyrazine deivative, see: Lumma & Springer (1981).



Experimental

Crystal data

$C_{12}H_9BrN_4$
M_r = 289.14
Monoclinic, $P2_1/n$
a = 3.9007 (14) Å
b = 13.545 (5) Å
c = 20.673 (8) Å
β = 93.059 (5)°
V = 1090.7 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.75 mm^{-1}
T = 100 K
0.27 × 0.11 × 0.05 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.431, T_{max} = 0.835
19939 measured reflections
2668 independent reflections
1887 reflections with $I > 2\sigma(I)$
R_{int} = 0.085

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.154
S = 1.05
2668 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.23 e Å^{-3}
$\Delta\rho_{min}$ = −1.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···N1^{i}	0.95	2.52	3.438 (6)	163
C10—H10···N2^{ii}	0.95	2.60	3.484 (7)	156

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x+\frac{5}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5478).

References

Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Lumma, W. C. Jr & Springer, J. P. (1981). *J. Org. Chem.* **46**, 3735–3736.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o1723 [doi:10.1107/S1600536810022993]

2-(6-Bromo-3-pyridyl)-8-methylimidazo[1,2-*a*]pyrazine

Buwen Huang, Eugene Rui, Martin Wythes, Pei-Pei Kung, Curtis Moore, Arnold L. Rheingold and Alex Yanovsky

S1. Comment

The reaction of 2-bromo-1-(6-bromo3-pyridyl)ethanone with 2-amino-3-methylpyrazine may potentially produce either 2- or 3-(6-bromo3-pyridyl)-8-methylimidazo[1,2-*a*]pyrazine. The present study shows that the compound with bromo-pyridyl substituent in position 2 of imidazopyrazine represents the major product of this reaction (Fig. 1).

The plane of the pyridine ring N1, C1—C5 forms the dihedral angle of 16.2 (2)° with the essentially planar imidazo-pyrazine system N2, N3, N4, C6—C11. Strange though it may seem, only one purely organic structure with non-protontated non-fused imidazo(1,2 - a)pyrazine system with only carbon substituents has been published heretofore (Lumma & Springer, 1981). The geometry of the bicyclic fragment in this structure is in good agreement with that of the title compound.

S2. Experimental

A mixture of 2-bromo-1-(6-bromo-3-pyridyl)-ethanone (2.70 g, 9.68 mmol), 2-amino-3-methylpyrazine (1.06 g, 9.68 mmol), and sodium bicarbonate (1.22 g, 14.5 mmol) in 40 ml of 2-propanol was heated at 80°C overnight. After cooling down to rt, the reaction mixture was concentrated to dryness. The resulting residue was partitioned between ethyl acetate (100 ml) and water (100 ml). The organic phase was washed with brine (1 × 100 ml), dried over sodium sulfate, concentrated to dryness, and purified by column chromatography with 0 --> 5% MeOH/EA to afford the desired product as a solid (1.25 g, 44.7% yield).

Colourless needles of (I) were grown by slow evaporation of an ethanol/dichloroethane solution.

S3. Refinement

All H atoms were placed in geometrically calculated positions (C—H 0.95 Å for aromatic and 0.98 Å for methyl H atoms, respectively) and included in the refinement in riding motion approximation. The U_{iso}(H) were set to 1.2U_{eq} of the carrying atom (1.5U_{eq} for methyl H atoms). The maximum residual density peak 1.23 e/Å^3 is located at a distance of 0.99 Å from the Br1 atom; the deepest hole -1.30 e/Å^3 is at a distance of 0.78 Å from the Br1 atom.

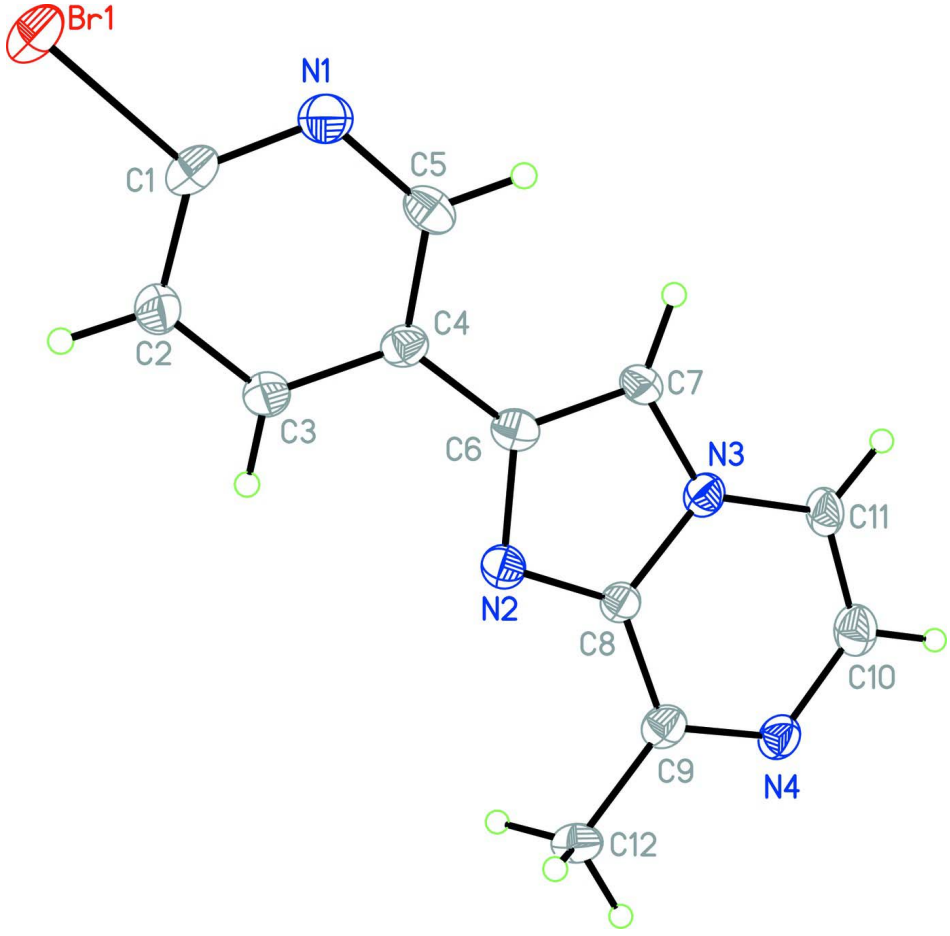


Figure 1

Molecular structure of the title compound, showing 50% probability displacement ellipsoids. H atoms are drawn as circles of arbitrary small radius.

2-(6-Bromo-3-pyridyl)-8-methylimidazo[1,2-*a*]pyrazine

Crystal data

$C_{12}H_9BrN_4$
$M_r = 289.14$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 3.9007\ (14)$ Å
$b = 13.545\ (5)$ Å
$c = 20.673\ (8)$ Å
$\beta = 93.059\ (5)°$
$V = 1090.7\ (7)$ Å^3
$Z = 4$
$F(000) = 576$
$D_x = 1.761$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6098 reflections
$\theta = 3.3–27.2°$
$\mu = 3.75$ mm^{-1}
$T = 100$ K
Needle, colorless
0.27 × 0.11 × 0.05 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.431$, $T_{max} = 0.835$
19939 measured reflections
2668 independent reflections

1887 reflections with $I > 2\sigma(I)$
$R_{int} = 0.085$
$\theta_{max} = 28.7°$, $\theta_{min} = 1.8°$
$h = -5 \rightarrow 5$
$k = -17 \rightarrow 17$
$l = -27 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.154$
$S = 1.05$
2668 reflections
155 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 3.9758P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.23$ e Å^{-3}
$\Delta\rho_{min} = -1.30$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.27478 (13)	0.63452 (4)	1.03763 (3)	0.0279 (2)
N1	0.5377 (11)	0.8165 (3)	1.0087 (2)	0.0247 (9)
N2	1.0290 (10)	0.9281 (3)	0.8097 (2)	0.0195 (8)
N3	1.0640 (10)	1.0898 (3)	0.83304 (19)	0.0193 (8)
N4	1.4045 (11)	1.1347 (3)	0.7232 (2)	0.0221 (9)
C1	0.4653 (12)	0.7301 (4)	0.9823 (2)	0.0215 (10)
C2	0.5223 (13)	0.7047 (4)	0.9189 (2)	0.0241 (11)
H2	0.4687	0.6407	0.9026	0.029*
C3	0.6590 (12)	0.7755 (4)	0.8807 (2)	0.0214 (10)
H3	0.7011	0.7611	0.8369	0.026*
C4	0.7370 (12)	0.8692 (3)	0.9062 (2)	0.0190 (10)
C5	0.6736 (12)	0.8850 (4)	0.9704 (2)	0.0220 (10)
H5	0.7290	0.9478	0.9887	0.026*
C6	0.8818 (12)	0.9470 (3)	0.8672 (2)	0.0185 (10)
C7	0.8997 (12)	1.0472 (3)	0.8822 (2)	0.0207 (10)
H7	0.8150	1.0790	0.9191	0.025*
C8	1.1395 (12)	1.0161 (3)	0.7898 (2)	0.0174 (9)
C9	1.3167 (12)	1.0417 (4)	0.7337 (2)	0.0207 (10)
C10	1.3238 (13)	1.2048 (4)	0.7677 (3)	0.0256 (11)
H10	1.3897	1.2710	0.7598	0.031*
C11	1.1591 (13)	1.1865 (3)	0.8214 (3)	0.0224 (10)
H11	1.1088	1.2380	0.8507	0.027*

C12	1.4005 (13)	0.9642 (4)	0.6864 (2)	0.0241 (11)
H12A	1.5153	0.9944	0.6503	0.036*
H12B	1.1887	0.9319	0.6700	0.036*
H12C	1.5530	0.9152	0.7077	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0245 (3)	0.0267 (3)	0.0331 (3)	−0.0011 (2)	0.0081 (2)	0.0102 (2)
N1	0.027 (2)	0.024 (2)	0.023 (2)	0.0019 (18)	0.0048 (17)	0.0007 (18)
N2	0.018 (2)	0.0173 (19)	0.024 (2)	0.0029 (16)	0.0018 (16)	−0.0005 (16)
N3	0.018 (2)	0.016 (2)	0.025 (2)	0.0009 (16)	0.0031 (16)	0.0015 (16)
N4	0.021 (2)	0.018 (2)	0.027 (2)	−0.0009 (17)	0.0047 (16)	0.0024 (17)
C1	0.017 (2)	0.023 (2)	0.025 (2)	0.0025 (19)	0.0039 (18)	0.008 (2)
C2	0.024 (3)	0.019 (2)	0.029 (3)	−0.001 (2)	0.005 (2)	−0.002 (2)
C3	0.019 (2)	0.022 (2)	0.023 (2)	−0.0002 (19)	0.0032 (19)	−0.001 (2)
C4	0.017 (2)	0.020 (2)	0.020 (2)	0.0028 (19)	0.0033 (17)	0.0016 (19)
C5	0.023 (2)	0.022 (2)	0.022 (2)	0.0055 (19)	−0.0005 (19)	−0.0042 (19)
C6	0.015 (2)	0.020 (2)	0.021 (2)	0.0061 (18)	0.0003 (18)	0.0002 (19)
C7	0.025 (3)	0.016 (2)	0.022 (2)	0.0009 (19)	0.0065 (19)	−0.0035 (19)
C8	0.016 (2)	0.015 (2)	0.021 (2)	0.0013 (17)	0.0026 (18)	0.0006 (18)
C9	0.017 (2)	0.019 (2)	0.026 (2)	0.0029 (19)	0.0026 (19)	0.003 (2)
C10	0.027 (3)	0.019 (2)	0.031 (3)	−0.001 (2)	0.001 (2)	0.001 (2)
C11	0.020 (2)	0.013 (2)	0.034 (3)	0.0008 (18)	0.002 (2)	−0.003 (2)
C12	0.024 (3)	0.027 (3)	0.021 (2)	0.002 (2)	0.0069 (19)	0.000 (2)

Geometric parameters (Å, º)

Br1—C1	1.905 (5)	C3—H3	0.9500
N1—C1	1.316 (7)	C4—C5	1.381 (7)
N1—C5	1.347 (7)	C4—C6	1.458 (7)
N2—C8	1.340 (6)	C5—H5	0.9500
N2—C6	1.371 (6)	C6—C7	1.393 (7)
N3—C7	1.359 (6)	C7—H7	0.9500
N3—C8	1.382 (6)	C8—C9	1.424 (7)
N3—C11	1.386 (6)	C9—C12	1.482 (7)
N4—C9	1.327 (6)	C10—C11	1.335 (7)
N4—C10	1.371 (7)	C10—H10	0.9500
C1—C2	1.384 (7)	C11—H11	0.9500
C2—C3	1.369 (7)	C12—H12A	0.9800
C2—H2	0.9500	C12—H12B	0.9800
C3—C4	1.401 (7)	C12—H12C	0.9800
C1—N1—C5	116.8 (4)	C7—C6—C4	126.6 (4)
C8—N2—C6	104.9 (4)	N3—C7—C6	105.4 (4)
C7—N3—C8	107.6 (4)	N3—C7—H7	127.3
C7—N3—C11	132.2 (4)	C6—C7—H7	127.3
C8—N3—C11	120.2 (4)	N2—C8—N3	111.1 (4)

C9—N4—C10	118.5 (4)	N2—C8—C9	130.2 (4)
N1—C1—C2	125.0 (5)	N3—C8—C9	118.7 (4)
N1—C1—Br1	115.9 (4)	N4—C9—C8	120.4 (4)
C2—C1—Br1	119.1 (4)	N4—C9—C12	119.8 (5)
C3—C2—C1	117.3 (5)	C8—C9—C12	119.9 (4)
C3—C2—H2	121.3	C11—C10—N4	124.6 (5)
C1—C2—H2	121.3	C11—C10—H10	117.7
C2—C3—C4	120.2 (5)	N4—C10—H10	117.7
C2—C3—H3	119.9	C10—C11—N3	117.6 (5)
C4—C3—H3	119.9	C10—C11—H11	121.2
C5—C4—C3	117.0 (4)	N3—C11—H11	121.2
C5—C4—C6	121.0 (4)	C9—C12—H12A	109.5
C3—C4—C6	122.0 (4)	C9—C12—H12B	109.5
N1—C5—C4	123.8 (5)	H12A—C12—H12B	109.5
N1—C5—H5	118.1	C9—C12—H12C	109.5
C4—C5—H5	118.1	H12A—C12—H12C	109.5
N2—C6—C7	110.9 (4)	H12B—C12—H12C	109.5
N2—C6—C4	122.4 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···N1^{i}	0.95	2.52	3.438 (6)	163
C10—H10···N2^{ii}	0.95	2.60	3.484 (7)	156

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+2; (ii) −*x*+5/2, *y*+1/2, −*z*+3/2.