Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-Bromo-3-methyl-1*H*-imidazo[4,5-*b*]-pyridin-2(3*H*)-one

Hend Bel Ghacham,[a] Youssef Kandri Rodi,[a] Frédéric Capet,[b] El Mokhtar Essassi[c] and Seik Weng Ng[d]*

[a]Laboratoire de Chimie Organique Appliquée, Faculté des Sciences et Techniques, Université Sidi Mohamed Ben Abdallah, Fés, Morocco, [b]Unité de Catalyse et de Chimie du Solide (UCCS), UMR 8181, Ecole Nationale Supérieure de Chimie de Lille, Lille, France, [c]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, and [d]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

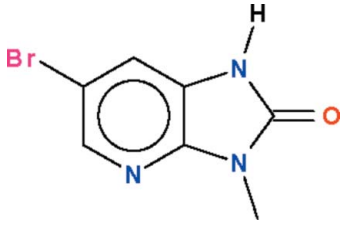
Received 6 April 2010; accepted 6 April 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.030; wR factor = 0.077; data-to-parameter ratio = 12.3.

The title compound, $C_7H_6BrN_3O$, was obtained from the reaction of 6-bromo-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one with methyl iodide. All non-H atoms lie in a common plane [r.m.s deviation = 0.017 (1) Å]. The amino group is a hydrogen-bond donor to the carbonyl group of an inversion-related molecule, the pair of hydrogen bonds giving rise to a hydrogen-bonded dimer.

Related literature

For the synthesis of the title compound, see: Grivas & Lindström (1995); Smolyar *et al.* (2007).



Experimental

Crystal data

$C_7H_6BrN_3O$ M_r = 228.06
Triclinic, $P\overline{1}$
a = 4.4151 (1) Å
b = 9.6004 (2) Å
c = 10.5330 (3) Å
α = 116.248 (1)°
β = 93.074 (2)°
γ = 91.687 (1)°
V = 399.14 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.10 mm^{-1}
T = 293 K
0.36 × 0.17 × 0.10 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.478, T_{max} = 0.630
4790 measured reflections
1401 independent reflections
1199 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
Standard reflections: 0

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.077
S = 1.05
1401 reflections
114 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O1^{i}	0.86 (1)	1.95 (1)	2.804 (3)	176 (4)

Symmetry code: (i) $-x, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors thank the University Sidi Mohammed Ben Abdallah, the Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5243).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Grivas, S. & Lindström, S. (1995). *J. Heterocycl. Chem.* **32**, 467-471.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smolyar, N. N., Lopatinskaya, Kh. Ya., Vasilechko, A. B., Lomov, D. A. & Yutilov, Yu. M. (2007). *Russ. J. Org. Chem.* **43**, 417–421.
Westrip, S. P. (2010). *publCIF*. In preparation.

supporting information

Acta Cryst. (2010). E**66**, o1080 [https://doi.org/10.1107/S1600536810012791]

6-Bromo-3-methyl-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

Hend Bel Ghacham, Youssef Kandri Rodi, Frédéric Capet, El Mokhtar Essassi and Seik Weng Ng

S1. Comment

6-Bromo-1*H*-imidazo[4,5-*b*]pyridine-2(3*H*)-one reacts with organic compounds to form pharmaceutical active compounds. It is easily methylated; in this study, it is methylated by methyl iodide under catalytic conditions. The mono *N*-methylated compound (Scheme I) is planar [r.m.s 0.017 (1) Å]. The amino group is hydrogen-bond donor to the carbonyl group of an inversion-related molecule to generate a hydrogen-bonded dimer (Fig. 1).

S2. Experimental

6-Bromo-1*H*-imidazo[4,5-*b*]pyridine-2(3*H*)-thione (1 mmol), potassium carbonate (4 mmol), tetra-*n*-butylammonium bromide (0.1 mmol) and methyl iodide (2.5 mmol) in DMF (15 ml) were stirred for 48 hours. After completion of reaction (as monitored by TLC), the salt was filtered and the solvent removed under reduced pressure. The resulting residue was purified by column chromatography on silica gel using ethyl acetate/hexane (1/2) as eluent. Yellow crystals was isolated when the solvent was allowed to evaporate.

S3. Refinement

Carbon-bound H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C). The amino H atom was located in a difference Fourier map, and was refined isotropically with a distance restraint of N—H = 0.86 (1) Å.

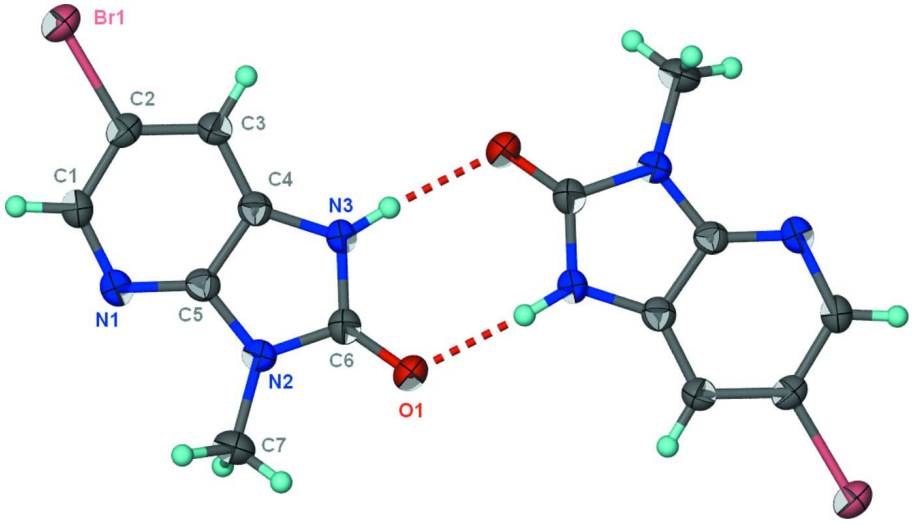


Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of the title compound forming a centrosymmetric dimer at the 50% probability level; H atoms are drawn as spheres of arbitrary radii.

6-Bromo-3-methyl-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

Crystal data

$C_7H_6BrN_3O$
$M_r = 228.06$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.4151$ (1) Å
$b = 9.6004$ (2) Å
$c = 10.5330$ (3) Å
$\alpha = 116.248$ (1)°
$\beta = 93.074$ (2)°
$\gamma = 91.687$ (1)°
$V = 399.14$ (2) Å^3
$Z = 2$
$F(000) = 224$
$D_x = 1.898$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2054 reflections
$\theta = 2.4$–$24.1°$
$\mu = 5.10$ mm^{-1}
$T = 293$ K
Prism, yellow
0.36 × 0.17 × 0.10 mm

Data collection

Bruker X8 APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.478$, $T_{max} = 0.630$
4790 measured reflections
1401 independent reflections
1199 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 24.9°$, $\theta_{min} = 2.2°$
$h = -5 \rightarrow 4$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.077$
$S = 1.05$
1401 reflections
114 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
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.0118P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.91934 (8)	0.96903 (4)	0.29671 (4)	0.04832 (17)
O1	0.0726 (5)	0.3085 (3)	0.3562 (3)	0.0478 (6)
N1	0.7410 (6)	0.4941 (3)	0.1298 (3)	0.0393 (6)
N2	0.4091 (6)	0.3574 (3)	0.2169 (3)	0.0368 (6)
N3	0.2638 (6)	0.5575 (3)	0.4045 (3)	0.0373 (6)
H3	0.165 (7)	0.603 (4)	0.478 (2)	0.052 (11)*
C1	0.8436 (8)	0.6389 (4)	0.1562 (3)	0.0395 (8)
H1	0.9737	0.6506	0.0946	0.047*
C2	0.7621 (7)	0.7715 (4)	0.2724 (3)	0.0362 (7)
C3	0.5682 (7)	0.7635 (3)	0.3680 (3)	0.0357 (7)
H3A	0.5157	0.8517	0.4462	0.043*
C4	0.4584 (7)	0.6171 (4)	0.3399 (3)	0.0332 (7)

C5	0.5521 (7)	0.4890 (4)	0.2200 (3)	0.0326 (7)
C6	0.2306 (7)	0.3988 (4)	0.3293 (3)	0.0359 (7)
C7	0.4409 (9)	0.1982 (4)	0.1123 (4)	0.0532 (10)
H7A	0.6141	0.1577	0.1406	0.080*
H7B	0.4681	0.1960	0.0216	0.080*
H7C	0.2613	0.1357	0.1055	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0551 (3)	0.0388 (2)	0.0537 (3)	−0.00649 (16)	0.00846 (17)	0.02302 (18)
O1	0.0560 (15)	0.0356 (12)	0.0502 (14)	−0.0057 (11)	0.0173 (12)	0.0168 (11)
N1	0.0399 (15)	0.0385 (15)	0.0382 (15)	0.0040 (12)	0.0119 (12)	0.0146 (13)
N2	0.0420 (15)	0.0298 (13)	0.0356 (14)	0.0018 (11)	0.0087 (12)	0.0112 (11)
N3	0.0406 (16)	0.0336 (14)	0.0361 (15)	0.0011 (12)	0.0140 (13)	0.0131 (12)
C1	0.0404 (18)	0.0410 (18)	0.0380 (18)	0.0024 (14)	0.0119 (15)	0.0175 (16)
C2	0.0372 (17)	0.0328 (16)	0.0416 (19)	−0.0016 (13)	0.0022 (15)	0.0194 (15)
C3	0.0367 (17)	0.0305 (16)	0.0381 (17)	0.0040 (13)	0.0074 (14)	0.0131 (14)
C4	0.0294 (16)	0.0336 (16)	0.0353 (17)	0.0032 (13)	0.0029 (13)	0.0139 (14)
C5	0.0305 (16)	0.0308 (15)	0.0344 (16)	0.0030 (13)	0.0015 (13)	0.0126 (13)
C6	0.0349 (17)	0.0367 (17)	0.0357 (17)	0.0011 (14)	0.0064 (14)	0.0155 (14)
C7	0.067 (3)	0.0341 (18)	0.049 (2)	0.0033 (17)	0.0166 (19)	0.0079 (16)

Geometric parameters (Å, º)

Br1—C2	1.902 (3)	C1—C2	1.394 (5)
O1—C6	1.233 (4)	C1—H1	0.9300
N1—C5	1.313 (4)	C2—C3	1.382 (4)
N1—C1	1.349 (4)	C3—C4	1.369 (4)
N2—C6	1.373 (4)	C3—H3A	0.9300
N2—C5	1.382 (4)	C4—C5	1.411 (4)
N2—C7	1.452 (4)	C7—H7A	0.9600
N3—C6	1.370 (4)	C7—H7B	0.9600
N3—C4	1.381 (4)	C7—H7C	0.9600
N3—H3	0.856 (10)		
C5—N1—C1	114.5 (3)	C3—C4—N3	134.7 (3)
C6—N2—C5	109.8 (2)	C3—C4—C5	118.7 (3)
C6—N2—C7	124.2 (3)	N3—C4—C5	106.6 (3)
C5—N2—C7	126.1 (3)	N1—C5—N2	126.7 (3)
C6—N3—C4	110.0 (3)	N1—C5—C4	126.5 (3)
C6—N3—H3	119 (3)	N2—C5—C4	106.7 (3)
C4—N3—H3	131 (3)	O1—C6—N3	127.3 (3)
N1—C1—C2	122.5 (3)	O1—C6—N2	125.8 (3)
N1—C1—H1	118.7	N3—C6—N2	106.9 (3)
C2—C1—H1	118.7	N2—C7—H7A	109.5
C3—C2—C1	122.1 (3)	N2—C7—H7B	109.5
C3—C2—Br1	119.5 (2)	H7A—C7—H7B	109.5

C1—C2—Br1	118.4 (2)	N2—C7—H7C	109.5
C4—C3—C2	115.6 (3)	H7A—C7—H7C	109.5
C4—C3—H3A	122.2	H7B—C7—H7C	109.5
C2—C3—H3A	122.2		

C5—N1—C1—C2	1.8 (5)	C6—N2—C5—C4	0.2 (3)
N1—C1—C2—C3	−0.5 (5)	C7—N2—C5—C4	−179.4 (3)
N1—C1—C2—Br1	−179.4 (2)	C3—C4—C5—N1	0.6 (5)
C1—C2—C3—C4	−0.9 (5)	N3—C4—C5—N1	−179.2 (3)
Br1—C2—C3—C4	178.0 (2)	C3—C4—C5—N2	−179.9 (3)
C2—C3—C4—N3	−179.4 (3)	N3—C4—C5—N2	0.3 (3)
C2—C3—C4—C5	0.8 (4)	C4—N3—C6—O1	−179.5 (3)
C6—N3—C4—C3	179.6 (3)	C4—N3—C6—N2	0.7 (4)
C6—N3—C4—C5	−0.6 (3)	C5—N2—C6—O1	179.7 (3)
C1—N1—C5—N2	178.8 (3)	C7—N2—C6—O1	−0.8 (5)
C1—N1—C5—C4	−1.9 (5)	C5—N2—C6—N3	−0.5 (3)
C6—N2—C5—N1	179.6 (3)	C7—N2—C6—N3	179.0 (3)
C7—N2—C5—N1	0.1 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1^{i}	0.86 (1)	1.95 (1)	2.804 (3)	176 (4)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.