Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 5-methylimidazo[1,2-*a*]pyridine-2-carboxylate

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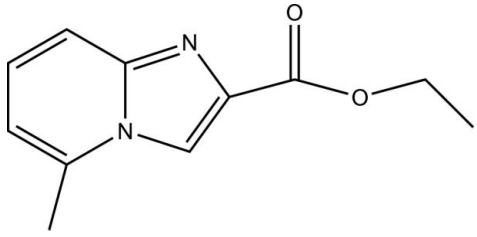
Received 18 June 2010; accepted 5 July 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.152; data-to-parameter ratio = 13.6.

The title compound, $C_{11}H_{12}N_2O_2$, was synthesized from the reaction of 6-methylpyridin-2-amine and ethyl 3-bromo-2-oxopropionate. In the molecular structure, the six- and five-membered rings are individually almost planar with r.m.s. deviations of 0.003 and 0.002 Å, respectively. The two rings are almost coplanar, the dihedral angle between their planes being 1.4 (3)°. Intermolecular C—H···O and C—H···N hydrogen bonds are present in the crystal structure.

Related literature

For the biological properties of related compounds, see: Xia *et al.* (2005); Warshakoon *et al.* (2006); Imaeda *et al.* (2008). For the synthetic procedure, see: Xia *et al.* (2005). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{11}H_{12}N_2O_2$
M_r = 204.23
Monoclinic, $C2/c$
a = 17.164 (3) Å
b = 10.521 (2) Å
c = 13.759 (3) Å
β = 124.77 (3)°
V = 2041 (1) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.973, T_{max} = 0.991
1923 measured reflections
1859 independent reflections
1360 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.152
S = 1.00
1859 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···N1^{i}	0.93	2.59	3.461 (4)	155
C3—H3A···O1^{i}	0.93	2.58	3.456 (4)	157

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *SHELXL97*.

The authors would like to thank Professor Hua-qin Wang of Nanjing University for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2214).

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supporting information

Acta Cryst. (2010). E**66**, o1999 [https://doi.org/10.1107/S1600536810026577]

Ethyl 5-methylimidazo[1,2-*a*]pyridine-2-carboxylate

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S1. Comment

Imidazo[1,2a]pyridine derivatives are of great interest because of their chemical and pharmaceutical properties. Some derivatives play a key role in preparing hypoxia inducible factor 1α prolyl hydroxylase inhibitors. And HIF-1α is of great potential value for treating ischemic diseases (Warshakoon *et al.*,2006). Some can be used as a material for preparing a series of FXa inhibitors, which can be used to cure various kinds of thromboembolic diseases (Imaeda *et al.*,2008). Herein we report the crystal structure of the title compound, (I). The molecular structure of (I) is shown in Fig.1. In this structure ring A (C1/C2/C3/C4/N2/C7) is a planar six-mermbered ring and the mean deviation from plane is 0.0027 Å, ring B(C4/N1/C5/C6/N2) is a planar five-mermbered ring with a mean deviation from planarity of 0.0018 Å. The dihedral angle between A and B ring is 1.4 (3)°. In the crystal structure, intermolecular C—H···O and C—H···N hydrogen bonds (Table 1) link the molecules to form a trimeric unit (Fig. 2).

S2. Experimental

6-Methyl-pyridin-2-ylamine (8 mmol) and 3-bromo-2-oxo-propionic acid ethyl ester (12 mmol) were added to 50 mL ethanol. The mixture was refluxed for 6 h and then the solvent was totally evaporated. Solid anhydrous $KHCO_3$ was added until pH=8 had been reached. Set aside for 3 h led to the observation of a white, flocculent precipitate, which was filtered and dried (yield ca. 52.6%, Xia *et al.* (2005)). Crystals suitable for X-ray analysis were obtained by dissolving (I) (0.5 g) in ethyl acetate (20 ml) and evaporating the solvent slowly at room temperature for about 10 d.

S3. Refinement

All H atoms were positioned geometrically, with C—H= 0.97, 0.96 and 0.93 Å for methylene, methyl and aromatic H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H)=xU_{eq}(C), where x=1.5 for methyl H atoms and x=1.2 for all other H atoms.

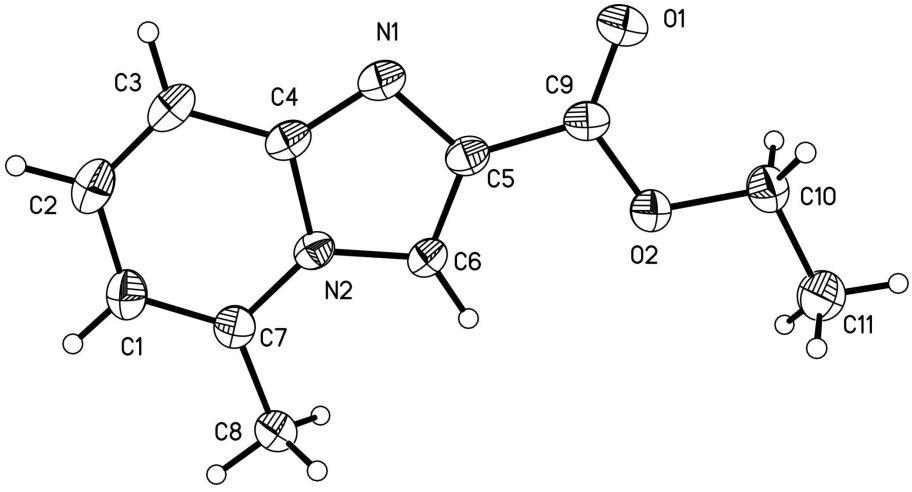

Figure 1

A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

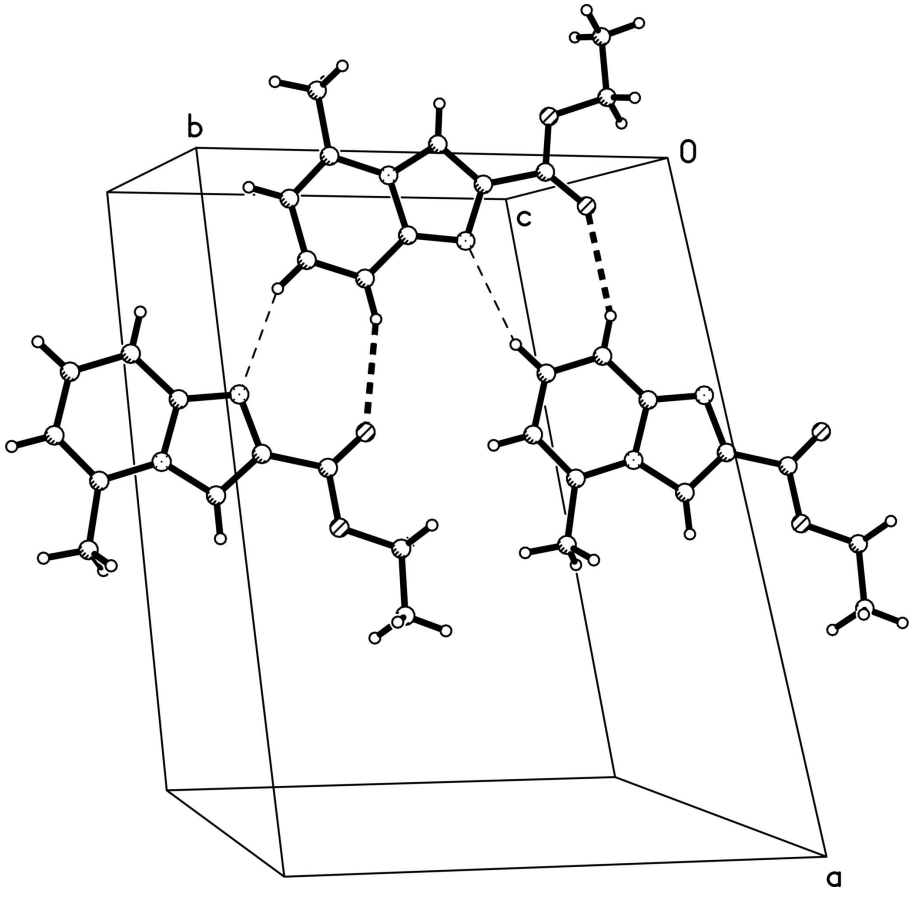

Figure 2

A packing diagram for (I). Dashed lines indicate an intermolecular C—H···N hydrogen bond and an intermolecular C—H···O hydrogen bond.

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Crystal data

$C_{11}H_{12}N_2O_2$
$M_r = 204.23$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 17.164$ (3) Å
$b = 10.521$ (2) Å
$c = 13.759$ (3) Å
$\beta = 124.77$ (3)°
$V = 2041$ (1) Å^3
$Z = 8$
$F(000) = 864$
$D_x = 1.329$ Mg m^{-3}
Melting point = 425–426 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.973$, $T_{max} = 0.991$
1923 measured reflections
1859 independent reflections
1360 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.4°$
$h = -20 \rightarrow 0$
$k = -12 \rightarrow 0$
$l = -13 \rightarrow 16$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.152$
$S = 1.00$
1859 reflections
137 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0117 (17)

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}
N1	0.12866 (11)	0.40243 (18)	0.19751 (15)	0.0466 (5)

O1	0.07064 (11)	0.13738 (15)	0.15389 (15)	0.0602 (5)
C1	0.06679 (17)	0.7803 (2)	0.15626 (19)	0.0523 (6)
H1B	0.0502	0.8658	0.1452	0.063*
N2	0.02673 (11)	0.56505 (15)	0.13920 (13)	0.0376 (4)
O2	−0.07427 (10)	0.20448 (14)	0.09371 (13)	0.0512 (5)
C2	0.16246 (17)	0.7466 (3)	0.2109 (2)	0.0582 (6)
H2A	0.2075	0.8102	0.2346	0.070*
C3	0.19016 (14)	0.6232 (2)	0.22978 (18)	0.0527 (6)
H3A	0.2537	0.6020	0.2665	0.063*
C4	0.12108 (13)	0.5273 (2)	0.19286 (17)	0.0422 (5)
C5	0.03813 (14)	0.3590 (2)	0.14702 (17)	0.0408 (5)
C6	−0.02457 (13)	0.45553 (19)	0.11134 (16)	0.0387 (5)
H6A	−0.0896	0.4487	0.0751	0.046*
C7	−0.00174 (15)	0.6916 (2)	0.11927 (17)	0.0432 (5)
C8	−0.10473 (15)	0.7154 (2)	0.05724 (19)	0.0509 (6)
H8A	−0.1164	0.8053	0.0480	0.076*
H8B	−0.1260	0.6807	0.1028	0.076*
H8C	−0.1385	0.6757	−0.0192	0.076*
C9	0.01645 (14)	0.2218 (2)	0.13352 (17)	0.0432 (5)
C10	−0.10735 (16)	0.0748 (2)	0.0699 (2)	0.0560 (6)
H10A	−0.0701	0.0241	0.1412	0.067*
H10B	−0.1011	0.0386	0.0098	0.067*
C11	−0.20816 (17)	0.0755 (3)	0.0283 (2)	0.0727 (8)
H11A	−0.2321	−0.0099	0.0114	0.109*
H11B	−0.2443	0.1261	−0.0421	0.109*
H11C	−0.2134	0.1109	0.0887	0.109*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0347 (9)	0.0568 (12)	0.0450 (10)	0.0026 (8)	0.0208 (8)	0.0007 (8)
O1	0.0514 (10)	0.0524 (10)	0.0734 (11)	0.0127 (8)	0.0335 (9)	0.0054 (8)
C1	0.0589 (14)	0.0472 (13)	0.0525 (13)	−0.0098 (11)	0.0328 (11)	−0.0024 (10)
N2	0.0335 (9)	0.0460 (10)	0.0347 (9)	−0.0019 (7)	0.0202 (7)	−0.0025 (7)
O2	0.0443 (9)	0.0417 (9)	0.0654 (10)	−0.0026 (6)	0.0301 (8)	−0.0048 (7)
C2	0.0540 (14)	0.0666 (16)	0.0554 (13)	−0.0196 (12)	0.0320 (11)	−0.0058 (12)
C3	0.0362 (11)	0.0717 (17)	0.0488 (13)	−0.0106 (10)	0.0234 (10)	−0.0027 (11)
C4	0.0330 (10)	0.0564 (14)	0.0360 (10)	−0.0003 (9)	0.0188 (8)	−0.0004 (9)
C5	0.0365 (10)	0.0492 (12)	0.0365 (10)	0.0012 (9)	0.0207 (9)	0.0003 (9)
C6	0.0327 (10)	0.0457 (12)	0.0372 (10)	−0.0037 (9)	0.0196 (8)	−0.0020 (9)
C7	0.0474 (12)	0.0466 (13)	0.0387 (11)	−0.0031 (10)	0.0264 (9)	−0.0024 (9)
C8	0.0498 (13)	0.0447 (13)	0.0588 (13)	0.0022 (10)	0.0313 (11)	−0.0019 (10)
C9	0.0397 (11)	0.0491 (13)	0.0401 (11)	0.0056 (9)	0.0223 (9)	0.0022 (9)
C10	0.0557 (14)	0.0427 (13)	0.0662 (15)	−0.0034 (10)	0.0327 (12)	−0.0038 (11)
C11	0.0557 (15)	0.0608 (17)	0.092 (2)	−0.0087 (12)	0.0369 (15)	−0.0109 (14)

Geometric parameters (Å, º)

N1—C4	1.318 (3)	C3—H3A	0.9300
N1—C5	1.369 (2)	C5—C6	1.352 (3)
O1—C9	1.198 (2)	C5—C9	1.475 (3)
C1—C7	1.353 (3)	C6—H6A	0.9300
C1—C2	1.407 (3)	C7—C8	1.482 (3)
C1—H1B	0.9300	C8—H8A	0.9600
N2—C6	1.366 (2)	C8—H8B	0.9600
N2—C7	1.391 (3)	C8—H8C	0.9600
N2—C4	1.401 (2)	C10—C11	1.480 (3)
O2—C9	1.336 (2)	C10—H10A	0.9700
O2—C10	1.442 (3)	C10—H10B	0.9700
C2—C3	1.357 (4)	C11—H11A	0.9600
C2—H2A	0.9300	C11—H11B	0.9600
C3—C4	1.412 (3)	C11—H11C	0.9600
C4—N1—C5	104.84 (16)	C1—C7—C8	126.6 (2)
C7—C1—C2	121.8 (2)	N2—C7—C8	116.43 (18)
C7—C1—H1B	119.1	C7—C8—H8A	109.5
C2—C1—H1B	119.1	C7—C8—H8B	109.5
C6—N2—C7	130.90 (17)	H8A—C8—H8B	109.5
C6—N2—C4	105.97 (16)	C7—C8—H8C	109.5
C7—N2—C4	123.11 (16)	H8A—C8—H8C	109.5
C9—O2—C10	116.01 (17)	H8B—C8—H8C	109.5
C3—C2—C1	121.2 (2)	O1—C9—O2	124.2 (2)
C3—C2—H2A	119.4	O1—C9—C5	126.1 (2)
C1—C2—H2A	119.4	O2—C9—C5	109.67 (17)
C2—C3—C4	119.0 (2)	O2—C10—C11	107.8 (2)
C2—C3—H3A	120.5	O2—C10—H10A	110.2
C4—C3—H3A	120.5	C11—C10—H10A	110.2
N1—C4—N2	111.15 (17)	O2—C10—H10B	110.2
N1—C4—C3	130.93 (19)	C11—C10—H10B	110.2
N2—C4—C3	117.91 (19)	H10A—C10—H10B	108.5
C6—C5—N1	111.79 (19)	C10—C11—H11A	109.5
C6—C5—C9	126.71 (19)	C10—C11—H11B	109.5
N1—C5—C9	121.48 (18)	H11A—C11—H11B	109.5
C5—C6—N2	106.24 (17)	C10—C11—H11C	109.5
C5—C6—H6A	126.9	H11A—C11—H11C	109.5
N2—C6—H6A	126.9	H11B—C11—H11C	109.5
C1—C7—N2	117.0 (2)		
C7—C1—C2—C3	−0.6 (3)	C4—N2—C6—C5	−0.42 (19)
C1—C2—C3—C4	0.4 (3)	C2—C1—C7—N2	0.9 (3)
C5—N1—C4—N2	−0.5 (2)	C2—C1—C7—C8	−177.8 (2)
C5—N1—C4—C3	−179.0 (2)	C6—N2—C7—C1	−178.96 (19)
C6—N2—C4—N1	0.6 (2)	C4—N2—C7—C1	−1.2 (3)
C7—N2—C4—N1	−177.71 (17)	C6—N2—C7—C8	−0.2 (3)

C6—N2—C4—C3	179.30 (17)	C4—N2—C7—C8	177.64 (16)
C7—N2—C4—C3	1.0 (3)	C10—O2—C9—O1	−2.8 (3)
C2—C3—C4—N1	177.8 (2)	C10—O2—C9—C5	176.75 (17)
C2—C3—C4—N2	−0.6 (3)	C6—C5—C9—O1	171.5 (2)
C4—N1—C5—C6	0.2 (2)	N1—C5—C9—O1	−6.9 (3)
C4—N1—C5—C9	178.87 (19)	C6—C5—C9—O2	−8.0 (3)
N1—C5—C6—N2	0.2 (2)	N1—C5—C9—O2	173.49 (16)
C9—C5—C6—N2	−178.43 (18)	C9—O2—C10—C11	179.40 (18)
C7—N2—C6—C5	177.67 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*A*···N1^{i}	0.93	2.59	3.461 (4)	155
C3—H3*A*···O1^{i}	0.93	2.58	3.456 (4)	157

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+1/2.