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Methyl 1-methyl-8-nitro-5-oxo-1,2,3,5-tetrahydroimidazo[1,2-*a*]pyridine-7-carboxylate

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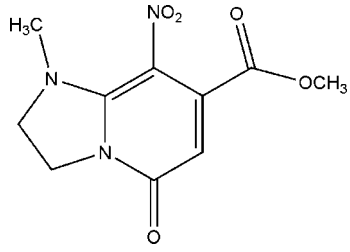
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.126; data-to-parameter ratio = 12.2.

In the title compound, $C_{10}H_{11}N_3O_5$, the dihedral angle between the fused pyridine and imidazolidine ring planes is 4.88 (12)°. The crystal structure is stabilized by weak C—H···O intermolecular interactions.

Related literature

For the synthesis, see: Huang & Tzai (1986).



Experimental

Crystal data

$C_{10}H_{11}N_3O_5$
M_r = 253.22
Monoclinic, $P2_1/n$
a = 9.848 (5) Å
b = 10.738 (5) Å
c = 11.318 (6) Å
β = 108.165 (8)°
V = 1137.2 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.14 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.974, T_{max} = 0.983
5593 measured reflections
2008 independent reflections
1428 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.126
S = 1.04
2008 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O2^{i}	0.97	2.54	3.106 (4)	118
C2—H2B···O5^{ii}	0.97	2.51	3.209 (3)	129
C3—H3B···O5^{iii}	0.97	2.52	3.295 (4)	137

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2590).

References

Bruker (1997). *SMART* (Version 5.611), *SAINT* (Version 5.01) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Huang, Z. T. & Tzai, L. H. (1986). *Chem. Ber.* **119**, 2208–2219.
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Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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Methyl 1-methyl-8-nitro-5-oxo-1,2,3,5-tetrahydroimidazo[1,2-*a*]pyridine-7-carboxylate

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Comment

Kentene aminals with an imidazolidine ring can act as nucleophiles and they are useful synthons, especially for the synthesis of fused heterocycles (Huang & Tzai, 1986). The title compound, (I), (Fig. 1), is an important representative of such reagents.

As shown in Fig. 1, the pyridine ring and the imidazolidine ring are nearly in the same plane [dihedral angle = 4.88 (12)°]. The crystal structure is stabilized by weak C—H···O intermolecular interactions (Table 1).

Experimental

The title compound was prepared according to the method of Huang & Tzai (1986). Colourless prism of (I) were obtained by recrystallization from ethyl acetate.

Refinement

All the H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

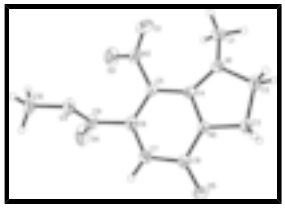

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Methyl 1-methyl-8-nitro-5-oxo-1,2,3,5-tetrahydroimidazo[1,2-*a*]pyridine-7-carboxylate

Crystal data

$C_{10}H_{11}N_3O_5$	F_{000} = 528
M_r = 253.22	D_x = 1.479 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2092 reflections
a = 9.848 (5) Å	θ = 2.7–25.7°
b = 10.738 (5) Å	μ = 0.12 mm^{-1}
c = 11.318 (6) Å	T = 294 (2) K

β = 108.165 (8)º	Prism, colorless
V = 1137.2 (10) Å^3	0.22 × 0.20 × 0.14 mm
Z = 4	

Data collection

Bruker SMART 1000 CCD diffractometer	2008 independent reflections
Radiation source: fine-focus sealed tube	1428 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
T = 294(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 2.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −9→11
T_{min} = 0.974, T_{max} = 0.983	k = −10→12
5593 measured reflections	l = −13→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H-atom parameters constrained
$wR(F^2)$ = 0.126	$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 0.3776P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.008
2008 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
165 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.4168 (2)	0.36676 (18)	0.40827 (18)	0.0638 (6)
O2	0.5293 (2)	0.2252 (2)	0.33881 (18)	0.0691 (6)

O3	0.30450 (17)	0.10139 (14)	0.15771 (13)	0.0430 (4)
O4	0.4560 (2)	−0.0553 (2)	0.23765 (17)	0.0732 (7)
O5	0.12662 (17)	−0.10383 (14)	0.53454 (15)	0.0464 (5)
N1	0.34353 (19)	0.26613 (16)	0.62403 (16)	0.0347 (4)
N2	0.23172 (18)	0.08686 (16)	0.55925 (15)	0.0322 (4)
N3	0.4424 (2)	0.2564 (2)	0.39231 (17)	0.0432 (5)
C1	0.4703 (3)	0.3461 (2)	0.6653 (2)	0.0476 (6)
H1A	0.5492	0.3056	0.6483	0.071*
H1B	0.4937	0.3610	0.7530	0.071*
H1C	0.4510	0.4239	0.6215	0.071*
C2	0.2644 (3)	0.2399 (2)	0.7117 (2)	0.0550 (7)
H2A	0.1934	0.3039	0.7071	0.066*
H2B	0.3289	0.2361	0.7962	0.066*
C3	0.1939 (3)	0.1156 (2)	0.6725 (2)	0.0439 (6)
H3A	0.2313	0.0531	0.7362	0.053*
H3B	0.0912	0.1213	0.6546	0.053*
C4	0.3190 (2)	0.17757 (19)	0.53658 (18)	0.0282 (5)
C5	0.3675 (2)	0.16054 (19)	0.43311 (19)	0.0317 (5)
C6	0.3348 (2)	0.0470 (2)	0.36515 (19)	0.0343 (5)
C7	0.2556 (3)	−0.0434 (2)	0.3964 (2)	0.0389 (6)
H7	0.2375	−0.1171	0.3510	0.047*
C8	0.1991 (2)	−0.0271 (2)	0.49896 (19)	0.0348 (5)
C9	0.3759 (2)	0.0253 (2)	0.2483 (2)	0.0398 (6)
C10	0.3298 (3)	0.0872 (3)	0.0386 (2)	0.0587 (8)
H10A	0.4256	0.1126	0.0464	0.088*
H10B	0.2634	0.1380	−0.0225	0.088*
H10C	0.3171	0.0015	0.0133	0.088*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0873 (14)	0.0420 (11)	0.0701 (13)	−0.0070 (10)	0.0361 (11)	0.0132 (10)
O2	0.0657 (12)	0.0934 (15)	0.0666 (12)	−0.0229 (11)	0.0473 (11)	−0.0131 (11)
O3	0.0539 (10)	0.0478 (10)	0.0311 (8)	0.0149 (8)	0.0187 (8)	0.0060 (7)
O4	0.0920 (15)	0.0826 (14)	0.0534 (12)	0.0525 (12)	0.0349 (11)	0.0129 (10)
O5	0.0532 (10)	0.0394 (9)	0.0463 (10)	−0.0144 (8)	0.0150 (8)	0.0045 (7)
N1	0.0362 (10)	0.0341 (10)	0.0381 (10)	−0.0070 (8)	0.0176 (8)	−0.0048 (8)
N2	0.0365 (10)	0.0342 (10)	0.0290 (9)	−0.0074 (8)	0.0147 (8)	−0.0019 (8)
N3	0.0441 (12)	0.0528 (14)	0.0368 (10)	−0.0067 (10)	0.0186 (9)	0.0060 (10)
C1	0.0478 (15)	0.0433 (15)	0.0534 (15)	−0.0141 (11)	0.0183 (12)	−0.0086 (12)
C2	0.0684 (17)	0.0590 (17)	0.0527 (15)	−0.0210 (14)	0.0408 (14)	−0.0200 (13)
C3	0.0510 (14)	0.0535 (15)	0.0349 (12)	−0.0144 (12)	0.0247 (11)	−0.0079 (11)
C4	0.0271 (11)	0.0282 (11)	0.0291 (10)	0.0016 (8)	0.0086 (9)	0.0032 (9)
C5	0.0320 (12)	0.0349 (12)	0.0306 (11)	0.0006 (9)	0.0132 (9)	0.0056 (10)
C6	0.0351 (12)	0.0390 (13)	0.0289 (11)	0.0107 (10)	0.0103 (10)	0.0046 (10)
C7	0.0518 (14)	0.0315 (12)	0.0322 (12)	0.0015 (10)	0.0113 (10)	−0.0030 (10)
C8	0.0373 (12)	0.0331 (12)	0.0309 (11)	−0.0019 (10)	0.0058 (10)	0.0030 (10)
C9	0.0448 (13)	0.0429 (14)	0.0342 (12)	0.0125 (11)	0.0159 (11)	0.0024 (11)

C10	0.080 (2)	0.0701 (19)	0.0337 (13)	0.0084 (15)	0.0291 (14)	0.0059 (13)

Geometric parameters (Å, °)

O1—N3	1.237 (3)	C1—H1C	0.9600
O2—N3	1.239 (2)	C2—C3	1.506 (3)
O3—C9	1.328 (3)	C2—H2A	0.9700
O3—C10	1.453 (3)	C2—H2B	0.9700
O4—C9	1.203 (3)	C3—H3A	0.9700
O5—C8	1.236 (2)	C3—H3B	0.9700
N1—C4	1.339 (3)	C4—C5	1.408 (3)
N1—C1	1.466 (3)	C5—C6	1.424 (3)
N1—C2	1.467 (3)	C6—C7	1.359 (3)
N2—C4	1.375 (3)	C6—C9	1.516 (3)
N2—C8	1.390 (3)	C7—C8	1.446 (3)
N2—C3	1.474 (3)	C7—H7	0.9300
N3—C5	1.424 (3)	C10—H10A	0.9600
C1—H1A	0.9600	C10—H10B	0.9600
C1—H1B	0.9600	C10—H10C	0.9600
C9—O3—C10	116.69 (18)	C2—C3—H3B	111.2
C4—N1—C1	126.18 (18)	H3A—C3—H3B	109.1
C4—N1—C2	110.63 (17)	N1—C4—N2	110.13 (17)
C1—N1—C2	118.42 (18)	N1—C4—C5	132.64 (19)
C4—N2—C8	126.48 (17)	N2—C4—C5	117.20 (19)
C4—N2—C3	110.84 (17)	C4—C5—C6	118.85 (19)
C8—N2—C3	121.94 (17)	C4—C5—N3	121.3 (2)
O1—N3—O2	122.2 (2)	C6—C5—N3	119.75 (18)
O1—N3—C5	119.69 (19)	C7—C6—C5	121.50 (19)
O2—N3—C5	118.0 (2)	C7—C6—C9	116.6 (2)
N1—C1—H1A	109.5	C5—C6—C9	121.67 (19)
N1—C1—H1B	109.5	C6—C7—C8	121.2 (2)
H1A—C1—H1B	109.5	C6—C7—H7	119.4
N1—C1—H1C	109.5	C8—C7—H7	119.4
H1A—C1—H1C	109.5	O5—C8—N2	119.68 (19)
H1B—C1—H1C	109.5	O5—C8—C7	125.9 (2)
N1—C2—C3	105.29 (17)	N2—C8—C7	114.39 (18)
N1—C2—H2A	110.7	O4—C9—O3	124.8 (2)
C3—C2—H2A	110.7	O4—C9—C6	124.0 (2)
N1—C2—H2B	110.7	O3—C9—C6	111.00 (18)
C3—C2—H2B	110.7	O3—C10—H10A	109.5
H2A—C2—H2B	108.8	O3—C10—H10B	109.5
N2—C3—C2	102.93 (17)	H10A—C10—H10B	109.5
N2—C3—H3A	111.2	O3—C10—H10C	109.5
C2—C3—H3A	111.2	H10A—C10—H10C	109.5
N2—C3—H3B	111.2	H10B—C10—H10C	109.5
C4—N1—C2—C3	4.1 (3)	O2—N3—C5—C6	−31.4 (3)
C1—N1—C2—C3	−153.0 (2)	C4—C5—C6—C7	1.5 (3)
C4—N2—C3—C2	2.8 (3)	N3—C5—C6—C7	−175.7 (2)
C8—N2—C3—C2	173.6 (2)	C4—C5—C6—C9	176.32 (19)

N1—C2—C3—N2	−4.0 (3)	N3—C5—C6—C9	−0.9 (3)
C1—N1—C4—N2	152.4 (2)	C5—C6—C7—C8	1.0 (3)
C2—N1—C4—N2	−2.4 (2)	C9—C6—C7—C8	−174.04 (19)
C1—N1—C4—C5	−25.7 (4)	C4—N2—C8—O5	175.04 (19)
C2—N1—C4—C5	179.4 (2)	C3—N2—C8—O5	5.8 (3)
C8—N2—C4—N1	−170.60 (19)	C4—N2—C8—C7	−5.3 (3)
C3—N2—C4—N1	−0.3 (2)	C3—N2—C8—C7	−174.6 (2)
C8—N2—C4—C5	7.9 (3)	C6—C7—C8—O5	−179.8 (2)
C3—N2—C4—C5	178.12 (19)	C6—C7—C8—N2	0.6 (3)
N1—C4—C5—C6	172.5 (2)	C10—O3—C9—O4	−2.0 (4)
N2—C4—C5—C6	−5.5 (3)	C10—O3—C9—C6	−177.7 (2)
N1—C4—C5—N3	−10.3 (4)	C7—C6—C9—O4	−66.8 (3)
N2—C4—C5—N3	171.63 (18)	C5—C6—C9—O4	118.1 (3)
O1—N3—C5—C4	−30.2 (3)	C7—C6—C9—O3	108.9 (2)
O2—N3—C5—C4	151.5 (2)	C5—C6—C9—O3	−66.1 (3)
O1—N3—C5—C6	146.9 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2A···O2^{i}	0.97	2.54	3.106 (4)	118
C2—H2B···O5^{ii}	0.97	2.51	3.209 (3)	129
C3—H3B···O5^{iii}	0.97	2.52	3.295 (4)	137

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

Fig. 1