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8-(4-Methoxybenzoyl)-2,3,6,7-tetrahydroimidazo[1,2-*a*]pyridin-5(1*H*)-one

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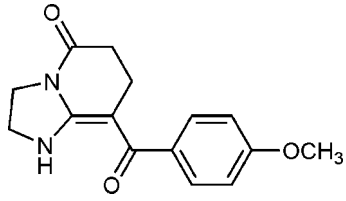
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.067; wR factor = 0.153; data-to-parameter ratio = 16.3.

In the title compound, $C_{15}H_{16}N_2O_3$, the crystal packing is consolidated by bifurcated intra- and intermolecular N—H···(O,O) hydrogen bonding. The dihydropyridinone ring adopts a twist sofa conformation.

Related literature

For the synthesis, see: Zhao & Huang (1993).



Experimental

Crystal data

$C_{15}H_{16}N_2O_3$
M_r = 272.30
Triclinic, $P\overline{1}$
a = 7.1455 (3) Å
b = 9.4356 (3) Å
c = 10.5282 (3) Å
α = 80.565 (1)°
β = 79.468 (1)°
γ = 69.568 (1)°
V = 650.10 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.28 × 0.26 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.973, T_{max} = 0.985
5329 measured reflections
2958 independent reflections
2314 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.153
S = 1.15
2958 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 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
N1—H1···O2	0.86	2.18	2.711 (2)	120
N1—H1···O2^{i}	0.86	2.26	2.982 (2)	142

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

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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: HB2659).

References

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Rigaku (2001). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zhao, Y. W. & Huang, Z. T. (1993). *Synth. Commun.* **23**, 1039–1046.

supplementary materials

Acta Cryst. (2007). E**63**, o4935 [**doi:10.1107/S1600536807061326**]

8-(4-Methoxybenzoyl)-2,3,6,7-tetrahydroimidazo[1,2-*a*]pyridin-5(1*H*)-one

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Comment

Heterocyclic ketene aminals can react with wide variety of organic compounds to afford different kinds of heterocyclic or fused heterocyclic compounds, therefore, they may be used as versatile synthetic inter-mediates in heterocyclic synthesis (Zhao & Huang, 1993).

The title compound is a representative of such reagents. As shown in Figs. 1 and 2, the packing is consolidated by intermolecular and intermolecular N—H···O hydrogen bonds (Table 1).

Experimental

The title compound was prepared according to the method of Zhao & Huang (1993) and colourless blocks of (I) were recrystallized from ethyl acetate in 90% yield (m.p. 474–475 K). IR: *v*= 3296 (NH), 1679 (lactam CO), 1627 (CO), 1599, 1521 $cm^{-1.}$ ^{1}H-NMR:*δ*= 9.46 (s, 1H), 7.42 (d, 2H), 6.87 (d, 2H), 3.94 (t, 2H), 3.80 (t, 2H), 3.81 (s, 3H), 2.74 (t, 2H), 2.50 p.p.m. (t, 2H). ^{13}C-NMR: *δ*= 190.1, 169.3, 160.5, 156.8, 133.5, 127.4, 128.9, 113.1, 85.2, 55.2, 42.6, 41.7, 32.5, 22.5 p.p.m.. MS: *m/z* = 272 (*M*+, 35), 271 (80), 244 (32), 229 (13), 215 (5), 163 (10), 135 (100) Anal. Calcd. for $C_{15}H_{16}N_2O_3$: C 66.16, H 5.92, *N* 10.29%; found: C 66.74, H 5.73, N 10.40%.

Refinement

The H atoms were positioned geometrically (C—H = 0.93–0.97 Å, N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(methyl C).

Figures

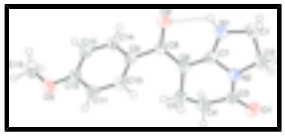

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radius.

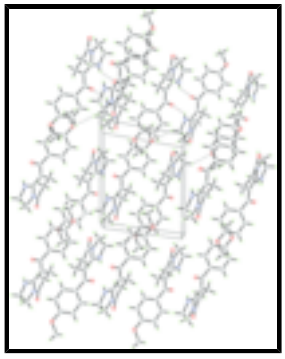

Fig. 2. The crystal packing for (I), with N—H···O interactions shown as dashed lines.

8-(4-Methoxybenzoyl)-2,3,6,7-tetrahydroimidazo[1,2-*a*]pyridin-5(1*H*)-one

Crystal data

$C_{15}H_{16}N_2O_3$	$Z = 2$
$M_r = 272.30$	$F_{000} = 288$
Triclinic, $P\bar{1}$	$D_x = 1.391$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$a = 7.1455$ (3) Å	Cell parameters from 5329 reflections
$b = 9.4356$ (3) Å	$\theta = 2.3$–27.5º
$c = 10.5282$ (3) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 80.565$ (1)º	$T = 296$ (2) K
$\beta = 79.468$ (1)º	Block, colorless
$\gamma = 69.568$ (1)º	0.28 × 0.26 × 0.15 mm
$V = 650.10$ (4) Å^3	

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	2958 independent reflections
Radiation source: rotating anode	2314 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 296(2)$ K	$\theta_{max} = 27.5$º
ω scans at fixed $\chi = 45°$	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$h = -9 \rightarrow 9$
$T_{min} = 0.973$, $T_{max} = 0.985$	$k = -11 \rightarrow 12$
5329 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.067$	H-atom parameters constrained

$wR(F^2) = 0.153$	$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.282P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} = <0.001$
2958 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
181 parameters	$\Delta\rho_{min} = -0.20$ 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}
C1	0.8836 (5)	1.0184 (3)	0.1877 (3)	0.0592 (8)
H1A	1.0187	0.9874	0.1403	0.071*
H1B	0.8321	1.1285	0.1832	0.071*
C2	0.7469 (4)	0.9591 (3)	0.1340 (2)	0.0501 (6)
H2A	0.6389	1.0419	0.0977	0.060*
H2B	0.8215	0.8928	0.0674	0.060*
C3	0.4947 (3)	0.8389 (2)	0.2575 (2)	0.0383 (5)
C4	0.4227 (4)	0.7760 (3)	0.3901 (2)	0.0457 (6)
H4A	0.3499	0.7095	0.3810	0.055*
H4B	0.3292	0.8594	0.4362	0.055*
C5	0.5916 (4)	0.6877 (3)	0.4709 (2)	0.0430 (6)
H5A	0.6686	0.5921	0.4358	0.052*
H5B	0.5341	0.6649	0.5595	0.052*
C6	0.7309 (3)	0.7784 (2)	0.4708 (2)	0.0350 (5)
C7	0.7621 (3)	0.8663 (2)	0.3560 (2)	0.0345 (5)
C8	0.8298 (3)	0.7768 (3)	0.5773 (2)	0.0366 (5)
C9	0.8096 (3)	0.6717 (2)	0.6990 (2)	0.0344 (5)
C10	0.8432 (3)	0.5168 (3)	0.6992 (2)	0.0374 (5)
H10	0.8755	0.4751	0.6207	0.045*
C11	0.8293 (3)	0.4245 (3)	0.8146 (2)	0.0393 (5)
H11	0.8545	0.3211	0.8133	0.047*
C12	0.7775 (3)	0.4859 (3)	0.9326 (2)	0.0367 (5)
C13	0.7434 (4)	0.6399 (3)	0.9343 (2)	0.0412 (5)
H13	0.7079	0.6820	1.0127	0.049*
C14	0.7629 (4)	0.7300 (3)	0.8180 (2)	0.0401 (5)

H14	0.7441	0.8324	0.8197	0.048*
C15	0.7128 (4)	0.4473 (3)	1.1643 (2)	0.0548 (7)
H15A	0.7077	0.3675	1.2329	0.082*
H15B	0.5835	0.5257	1.1671	0.082*
H15C	0.8128	0.4891	1.1751	0.082*
N1	0.8829 (3)	0.9503 (2)	0.32159 (18)	0.0438 (5)
H1	0.9539	0.9629	0.3739	0.053*
N2	0.6675 (3)	0.8741 (2)	0.24873 (17)	0.0378 (4)
O1	0.4075 (3)	0.8629 (2)	0.16296 (16)	0.0515 (5)
O2	0.9317 (3)	0.8615 (2)	0.57509 (16)	0.0530 (5)
O3	0.7638 (3)	0.38697 (19)	1.04175 (16)	0.0501 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.082 (2)	0.0592 (17)	0.0498 (15)	−0.0444 (16)	−0.0190 (14)	0.0165 (13)
C2	0.0537 (15)	0.0629 (17)	0.0365 (13)	−0.0280 (13)	−0.0069 (11)	0.0079 (11)
C3	0.0376 (12)	0.0355 (12)	0.0425 (12)	−0.0105 (9)	−0.0103 (9)	−0.0046 (9)
C4	0.0395 (13)	0.0593 (16)	0.0455 (13)	−0.0253 (12)	−0.0079 (10)	−0.0027 (11)
C5	0.0494 (14)	0.0503 (14)	0.0373 (12)	−0.0292 (11)	−0.0080 (10)	0.0045 (10)
C6	0.0367 (11)	0.0380 (12)	0.0329 (11)	−0.0165 (9)	−0.0047 (9)	−0.0015 (9)
C7	0.0327 (11)	0.0362 (12)	0.0361 (11)	−0.0117 (9)	−0.0058 (9)	−0.0060 (9)
C8	0.0395 (12)	0.0369 (12)	0.0366 (11)	−0.0166 (10)	−0.0048 (9)	−0.0045 (9)
C9	0.0340 (11)	0.0367 (12)	0.0355 (11)	−0.0138 (9)	−0.0099 (9)	−0.0016 (9)
C10	0.0377 (12)	0.0405 (13)	0.0357 (11)	−0.0123 (10)	−0.0068 (9)	−0.0082 (9)
C11	0.0411 (12)	0.0301 (12)	0.0473 (13)	−0.0105 (10)	−0.0098 (10)	−0.0040 (9)
C12	0.0319 (11)	0.0387 (12)	0.0390 (12)	−0.0128 (9)	−0.0080 (9)	0.0030 (9)
C13	0.0495 (14)	0.0421 (13)	0.0338 (11)	−0.0165 (11)	−0.0050 (10)	−0.0066 (9)
C14	0.0497 (14)	0.0339 (12)	0.0393 (12)	−0.0167 (10)	−0.0077 (10)	−0.0031 (9)
C15	0.0595 (17)	0.0629 (18)	0.0367 (13)	−0.0204 (14)	−0.0013 (11)	0.0043 (12)
N1	0.0530 (12)	0.0529 (12)	0.0356 (10)	−0.0327 (10)	−0.0076 (9)	0.0028 (9)
N2	0.0418 (10)	0.0428 (11)	0.0312 (9)	−0.0187 (9)	−0.0077 (8)	0.0030 (8)
O1	0.0528 (10)	0.0615 (12)	0.0470 (10)	−0.0228 (9)	−0.0214 (8)	0.0010 (8)
O2	0.0710 (12)	0.0630 (12)	0.0447 (10)	−0.0466 (10)	−0.0173 (8)	0.0050 (8)
O3	0.0636 (11)	0.0465 (10)	0.0398 (9)	−0.0223 (9)	−0.0067 (8)	0.0052 (7)

Geometric parameters (Å, °)

C1—N1	1.450 (3)	C7—N2	1.398 (3)
C1—C2	1.508 (4)	C8—O2	1.251 (3)
C1—H1A	0.9700	C8—C9	1.504 (3)
C1—H1B	0.9700	C9—C14	1.389 (3)
C2—N2	1.474 (3)	C9—C10	1.395 (3)
C2—H2A	0.9700	C10—C11	1.384 (3)
C2—H2B	0.9700	C10—H10	0.9300
C3—O1	1.219 (3)	C11—C12	1.391 (3)
C3—N2	1.371 (3)	C11—H11	0.9300
C3—C4	1.498 (3)	C12—O3	1.369 (3)
C4—C5	1.521 (3)	C12—C13	1.389 (3)

C4—H4A	0.9700	C13—C14	1.386 (3)
C4—H4B	0.9700	C13—H13	0.9300
C5—C6	1.522 (3)	C14—H14	0.9300
C5—H5A	0.9700	C15—O3	1.430 (3)
C5—H5B	0.9700	C15—H15A	0.9600
C6—C7	1.379 (3)	C15—H15B	0.9600
C6—C8	1.426 (3)	C15—H15C	0.9600
C7—N1	1.332 (3)	N1—H1	0.8600
N1—C1—C2	104.15 (19)	O2—C8—C9	117.80 (19)
N1—C1—H1A	110.9	C6—C8—C9	119.50 (18)
C2—C1—H1A	110.9	C14—C9—C10	118.0 (2)
N1—C1—H1B	110.9	C14—C9—C8	118.47 (19)
C2—C1—H1B	110.9	C10—C9—C8	123.47 (19)
H1A—C1—H1B	108.9	C11—C10—C9	120.9 (2)
N2—C2—C1	103.22 (18)	C11—C10—H10	119.5
N2—C2—H2A	111.1	C9—C10—H10	119.5
C1—C2—H2A	111.1	C10—C11—C12	120.2 (2)
N2—C2—H2B	111.1	C10—C11—H11	119.9
C1—C2—H2B	111.1	C12—C11—H11	119.9
H2A—C2—H2B	109.1	O3—C12—C13	124.0 (2)
O1—C3—N2	120.5 (2)	O3—C12—C11	116.3 (2)
O1—C3—C4	124.4 (2)	C13—C12—C11	119.7 (2)
N2—C3—C4	115.00 (19)	C14—C13—C12	119.4 (2)
C3—C4—C5	113.69 (19)	C14—C13—H13	120.3
C3—C4—H4A	108.8	C12—C13—H13	120.3
C5—C4—H4A	108.8	C13—C14—C9	121.8 (2)
C3—C4—H4B	108.8	C13—C14—H14	119.1
C5—C4—H4B	108.8	C9—C14—H14	119.1
H4A—C4—H4B	107.7	O3—C15—H15A	109.5
C4—C5—C6	111.05 (19)	O3—C15—H15B	109.5
C4—C5—H5A	109.4	H15A—C15—H15B	109.5
C6—C5—H5A	109.4	O3—C15—H15C	109.5
C4—C5—H5B	109.4	H15A—C15—H15C	109.5
C6—C5—H5B	109.4	H15B—C15—H15C	109.5
H5A—C5—H5B	108.0	C7—N1—C1	113.64 (19)
C7—C6—C8	119.26 (19)	C7—N1—H1	123.2
C7—C6—C5	115.36 (19)	C1—N1—H1	123.2
C8—C6—C5	125.37 (19)	C3—N2—C7	123.54 (18)
N1—C7—C6	130.3 (2)	C3—N2—C2	123.97 (18)
N1—C7—N2	107.69 (18)	C7—N2—C2	110.89 (18)
C6—C7—N2	121.94 (19)	C12—O3—C15	117.26 (19)
O2—C8—C6	122.7 (2)		
N1—C1—C2—N2	5.2 (3)	C10—C11—C12—C13	−1.2 (3)
O1—C3—C4—C5	152.2 (2)	O3—C12—C13—C14	179.5 (2)
N2—C3—C4—C5	−29.9 (3)	C11—C12—C13—C14	−0.4 (3)
C3—C4—C5—C6	49.7 (3)	C12—C13—C14—C9	2.0 (3)
C4—C5—C6—C7	−35.5 (3)	C10—C9—C14—C13	−2.0 (3)
C4—C5—C6—C8	145.3 (2)	C8—C9—C14—C13	−179.5 (2)

C8—C6—C7—N1	3.8 (4)	C6—C7—N1—C1	175.9 (3)
C5—C6—C7—N1	−175.4 (2)	N2—C7—N1—C1	−1.4 (3)
C8—C6—C7—N2	−179.2 (2)	C2—C1—N1—C7	−2.6 (3)
C5—C6—C7—N2	1.6 (3)	O1—C3—N2—C7	172.3 (2)
C7—C6—C8—O2	6.0 (4)	C4—C3—N2—C7	−5.7 (3)
C5—C6—C8—O2	−174.9 (2)	O1—C3—N2—C2	8.0 (4)
C7—C6—C8—C9	−175.1 (2)	C4—C3—N2—C2	−170.0 (2)
C5—C6—C8—C9	4.0 (3)	N1—C7—N2—C3	−161.0 (2)
O2—C8—C9—C14	46.4 (3)	C6—C7—N2—C3	21.4 (3)
C6—C8—C9—C14	−132.5 (2)	N1—C7—N2—C2	5.1 (3)
O2—C8—C9—C10	−130.9 (2)	C6—C7—N2—C2	−172.5 (2)
C6—C8—C9—C10	50.1 (3)	C1—C2—N2—C3	159.5 (2)
C14—C9—C10—C11	0.4 (3)	C1—C2—N2—C7	−6.5 (3)
C8—C9—C10—C11	177.7 (2)	C13—C12—O3—C15	−0.5 (3)
C9—C10—C11—C12	1.2 (3)	C11—C12—O3—C15	179.4 (2)
C10—C11—C12—O3	178.94 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	2.18	2.711 (2)	120
N1—H1···O2^{i}	0.86	2.26	2.982 (2)	142

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

Fig. 1

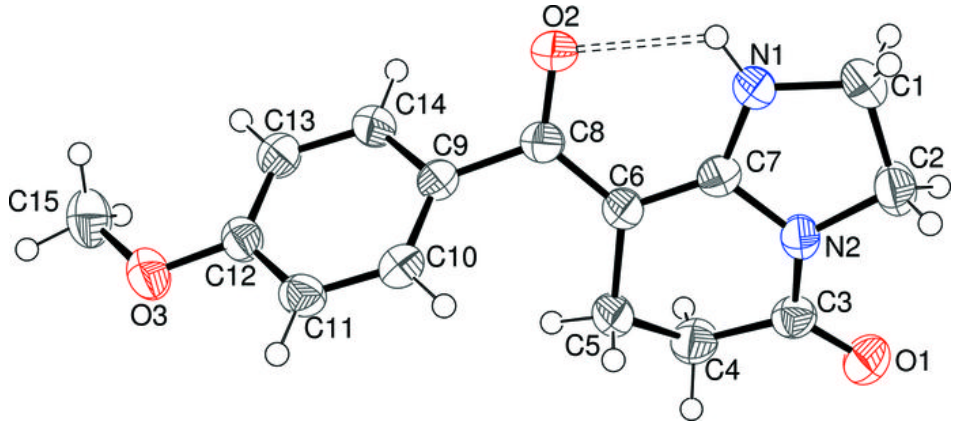

Fig. 2