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9-(4-Methoxybenzoyl)-1,2,3,4-tetrahydro-6*H*-pyrido[1,2-*a*]pyrimidin-6-one

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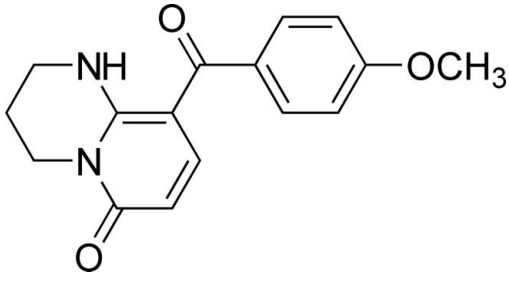
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.111; data-to-parameter ratio = 12.4.

The title compound, $C_{16}H_{16}N_2O_3$, contains an intramolecular N—H···O hydrogen bond. The six-membered 1,3-diazo-heterocyclic ring adopts a twisted conformation and the methoxybenzene group forms a dihedral angle of 54.1 (1)° with the pyrimidinone ring.

Related literature

The title compound is a heterocyclic ketene aminal; compounds of this type are useful building blocks in organic synthesis, especially for the synthesis of heterocycles. See: Huang & Liu (1986); Huang & Wang (1994).



Experimental

Crystal data

$C_{16}H_{16}N_2O_3$
M_r = 284.12
Triclinic, $P\overline{1}$
a = 7.9544 (18) Å
b = 8.3320 (19) Å
c = 11.063 (3) Å
α = 77.913 (3)°
β = 73.384 (4)°
γ = 77.641 (4)°
V = 677.7 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
3460 measured reflections
2370 independent reflections
1814 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.111
S = 1.00
2370 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.86	1.94	2.624 (2)	135

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*.

The authors thank Mr Haibin Song at Nankai University for the X-ray structure determination.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2189).

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Huang, Z.-T. & Liu, Z.-R. (1986). *Heterocycles*, **24**, 2247–2254.
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supplementary materials

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9-(4-Methoxybenzoyl)-1,2,3,4-tetrahydro-6*H*-pyrido[1,2-*a*]pyrimidin-6-one

C.-Y. Yu, X.-N. Yuan and Z.-T. Huang

Comment

Heterocyclic ketene aminals, also named cyclic 1,1-enediamines, are useful building blocks in organic synthesis, especially for the synthesis of heterocycles (Huang & Wang, 1994). The title compound (Fig. 1) was synthesized through consecutive nucleophilic addition and cyclocondensation reactions. The crystal structure was determined to provide unambiguous evidence for the molecular configuration.

The six-membered 1,3-diazoheterocyclic ring C1/C2/C3/N2/C4/N1 adopts a twisted conformation, in which the C1/C2/C3/N2 and N1/C1/C2/C3 torsion angles are −52.5 (2) and 54.2 (2)°, respectively. The phenyl ring C10–C15 forms a dihedral angle of 54.1 (1)° to the pyrimidinone ring.

Experimental

The title compound was prepared according to the procedure of Huang & Liu (1986) and recrystallized from ethyl acetate in 80% yield.

Refinement

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

Figures

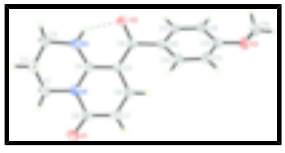

Fig. 1. The molecular structure of the title compound showing 30% displacement ellipsoids for non-H atoms. The intramolecular hydrogen bond is indicated by a dashed line.

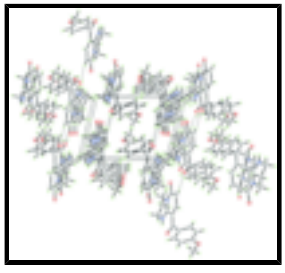

Fig. 2. Packing diagram of the title compound viewed down the *b*-axis.

9-(4-Methoxybenzoyl)-1,2,3,4-tetrahydro-6*H*-pyrido[1,2-*a*]pyrimidin-6-one

Crystal data

$C_{16}H_{16}N_2O_3$	$Z = 2$
$M_r = 284.12$	$F_{000} = 300$
Triclinic, $P\overline{1}$	$D_x = 1.393$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 437-439 K
$a = 7.9544$ (18) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 8.3320$ (19) Å	Cell parameters from 1694 reflections
$c = 11.063$ (3) Å	$\theta = 2.5$–26.4º
$\alpha = 77.913$ (3)º	$\mu = 0.10$ mm^{-1}
$\beta = 73.384$ (4)º	$T = 294$ (2) K
$\gamma = 77.641$ (4)º	Prism, colorless
$V = 677.7$ (3) Å^3	0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD diffractometer	1814 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.017$
Monochromator: graphite	$\theta_{max} = 25.0$º
$T = 294$(2) K	$\theta_{min} = 2.0$º
ω and φ scans	$h = -9\rightarrow4$
Absorption correction: none	$k = -9\rightarrow9$
3460 measured reflections	$l = -13\rightarrow12$
2370 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.111$	$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.1309P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
2370 reflections	$\Delta\rho_{max} = 0.12$ e Å^{-3}
191 parameters	$\Delta\rho_{min} = -0.16$ 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	0.58919 (16)	0.28590 (17)	0.11492 (13)	0.0664 (4)
O2	−0.22334 (15)	0.58455 (15)	0.22135 (13)	0.0572 (4)
O3	−0.29821 (17)	1.13578 (14)	0.55187 (12)	0.0580 (4)
N1	0.29385 (17)	0.30686 (16)	0.13068 (13)	0.0438 (3)
N2	−0.00321 (18)	0.32891 (16)	0.13146 (13)	0.0470 (4)
H2	−0.1095	0.3840	0.1478	0.056*
C1	0.3389 (3)	0.1542 (2)	0.0723 (2)	0.0635 (5)
H1A	0.4377	0.0815	0.1006	0.076*
H1B	0.3757	0.1833	−0.0199	0.076*
C2	0.1820 (3)	0.0633 (2)	0.10810 (19)	0.0610 (5)
H2A	0.2112	−0.0306	0.0625	0.073*
H2B	0.1541	0.0213	0.1990	0.073*
C3	0.0250 (2)	0.1795 (2)	0.07471 (17)	0.0510 (4)
H3A	0.0460	0.2086	−0.0174	0.061*
H3B	−0.0798	0.1262	0.1069	0.061*
C4	0.1214 (2)	0.38749 (18)	0.16018 (14)	0.0385 (4)
C5	0.0801 (2)	0.53028 (18)	0.22182 (14)	0.0382 (4)
C6	0.2259 (2)	0.5872 (2)	0.23969 (16)	0.0452 (4)
H6	0.2029	0.6824	0.2771	0.054*
C7	0.3953 (2)	0.5109 (2)	0.20547 (16)	0.0499 (4)
H7	0.4854	0.5546	0.2190	0.060*
C8	0.4387 (2)	0.3644 (2)	0.14882 (15)	0.0477 (4)
C9	−0.0996 (2)	0.61628 (19)	0.25642 (16)	0.0416 (4)
C10	−0.1457 (2)	0.75394 (18)	0.33420 (15)	0.0400 (4)
C11	−0.2538 (2)	0.8991 (2)	0.29682 (15)	0.0438 (4)
H11	−0.2930	0.9084	0.2237	0.053*
C12	−0.3050 (2)	1.03035 (19)	0.36520 (16)	0.0459 (4)
H12	−0.3734	1.1283	0.3362	0.055*
C13	−0.2537 (2)	1.01488 (19)	0.47683 (15)	0.0434 (4)
C14	−0.1506 (2)	0.8687 (2)	0.51784 (16)	0.0509 (4)
H14	−0.1187	0.8564	0.5943	0.061*
C15	−0.0949 (2)	0.7414 (2)	0.44651 (16)	0.0482 (4)
H15	−0.0221	0.6456	0.4738	0.058*

C16	−0.3885 (3)	1.2929 (2)	0.5050 (2)	0.0648 (5)
H16A	−0.3154	1.3387	0.4253	0.097*
H16B	−0.4122	1.3671	0.5658	0.097*
H16C	−0.4986	1.2790	0.4923	0.097*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0416 (7)	0.0836 (10)	0.0730 (9)	0.0115 (6)	−0.0199 (6)	−0.0244 (7)
O2	0.0383 (6)	0.0571 (7)	0.0841 (9)	−0.0064 (5)	−0.0148 (6)	−0.0305 (7)
O3	0.0664 (8)	0.0503 (7)	0.0611 (8)	0.0042 (6)	−0.0215 (6)	−0.0239 (6)
N1	0.0419 (8)	0.0427 (7)	0.0465 (8)	0.0027 (6)	−0.0141 (6)	−0.0123 (6)
N2	0.0410 (8)	0.0434 (8)	0.0598 (9)	−0.0053 (6)	−0.0114 (6)	−0.0184 (6)
C1	0.0541 (11)	0.0619 (12)	0.0782 (13)	0.0136 (9)	−0.0207 (10)	−0.0369 (10)
C2	0.0821 (14)	0.0440 (10)	0.0607 (11)	−0.0035 (9)	−0.0211 (10)	−0.0192 (8)
C3	0.0549 (11)	0.0496 (10)	0.0513 (10)	−0.0113 (8)	−0.0082 (8)	−0.0185 (8)
C4	0.0397 (9)	0.0363 (8)	0.0384 (8)	−0.0061 (6)	−0.0104 (7)	−0.0027 (6)
C5	0.0383 (8)	0.0343 (8)	0.0427 (9)	−0.0063 (6)	−0.0114 (7)	−0.0055 (6)
C6	0.0453 (9)	0.0415 (9)	0.0518 (10)	−0.0076 (7)	−0.0158 (8)	−0.0087 (7)
C7	0.0395 (9)	0.0584 (10)	0.0567 (10)	−0.0091 (8)	−0.0174 (8)	−0.0113 (8)
C8	0.0406 (9)	0.0562 (10)	0.0434 (9)	0.0022 (8)	−0.0156 (7)	−0.0054 (8)
C9	0.0392 (9)	0.0365 (8)	0.0497 (9)	−0.0101 (7)	−0.0093 (7)	−0.0067 (7)
C10	0.0365 (8)	0.0360 (8)	0.0459 (9)	−0.0074 (6)	−0.0069 (7)	−0.0063 (7)
C11	0.0425 (9)	0.0463 (9)	0.0434 (9)	−0.0036 (7)	−0.0129 (7)	−0.0095 (7)
C12	0.0454 (9)	0.0385 (9)	0.0507 (10)	0.0021 (7)	−0.0139 (8)	−0.0072 (7)
C13	0.0433 (9)	0.0395 (9)	0.0461 (9)	−0.0057 (7)	−0.0068 (7)	−0.0114 (7)
C14	0.0613 (11)	0.0482 (10)	0.0441 (9)	−0.0032 (8)	−0.0191 (8)	−0.0072 (8)
C15	0.0549 (10)	0.0357 (8)	0.0511 (10)	−0.0006 (7)	−0.0169 (8)	−0.0024 (7)
C16	0.0742 (13)	0.0444 (10)	0.0762 (13)	0.0039 (9)	−0.0202 (11)	−0.0230 (9)

Geometric parameters (Å, °)

O1—C8	1.2298 (19)	C5—C9	1.438 (2)
O2—C9	1.2495 (19)	C6—C7	1.344 (2)
O3—C13	1.3633 (18)	C6—H6	0.930
O3—C16	1.423 (2)	C7—C8	1.421 (2)
N1—C4	1.3714 (19)	C7—H7	0.930
N1—C8	1.416 (2)	C9—C10	1.498 (2)
N1—C1	1.476 (2)	C10—C11	1.386 (2)
N2—C4	1.332 (2)	C10—C15	1.390 (2)
N2—C3	1.457 (2)	C11—C12	1.384 (2)
N2—H2	0.860	C11—H11	0.930
C1—C2	1.512 (3)	C12—C13	1.381 (2)
C1—H1A	0.970	C12—H12	0.930
C1—H1B	0.970	C13—C14	1.384 (2)
C2—C3	1.493 (3)	C14—C15	1.376 (2)
C2—H2A	0.970	C14—H14	0.930
C2—H2B	0.970	C15—H15	0.930
C3—H3A	0.970	C16—H16A	0.960

C3—H3B	0.970	C16—H16B	0.960
C4—C5	1.426 (2)	C16—H16C	0.960
C5—C6	1.420 (2)		
C13—O3—C16	117.28 (14)	C6—C7—C8	120.88 (16)
C4—N1—C8	123.72 (13)	C6—C7—H7	119.6
C4—N1—C1	120.34 (14)	C8—C7—H7	119.6
C8—N1—C1	115.91 (13)	O1—C8—N1	118.55 (16)
C4—N2—C3	125.61 (14)	O1—C8—C7	125.50 (16)
C4—N2—H2	117.2	N1—C8—C7	115.94 (14)
C3—N2—H2	117.2	O2—C9—C5	122.69 (14)
N1—C1—C2	111.30 (15)	O2—C9—C10	116.66 (14)
N1—C1—H1A	109.4	C5—C9—C10	120.62 (14)
C2—C1—H1A	109.4	C11—C10—C15	117.62 (14)
N1—C1—H1B	109.4	C11—C10—C9	118.49 (14)
C2—C1—H1B	109.4	C15—C10—C9	123.77 (14)
H1A—C1—H1B	108.0	C12—C11—C10	121.86 (15)
C3—C2—C1	109.44 (16)	C12—C11—H11	119.1
C3—C2—H2A	109.8	C10—C11—H11	119.1
C1—C2—H2A	109.8	C13—C12—C11	119.49 (14)
C3—C2—H2B	109.8	C13—C12—H12	120.3
C1—C2—H2B	109.8	C11—C12—H12	120.3
H2A—C2—H2B	108.2	O3—C13—C12	124.20 (14)
N2—C3—C2	108.93 (14)	O3—C13—C14	116.40 (15)
N2—C3—H3A	109.9	C12—C13—C14	119.40 (15)
C2—C3—H3A	109.9	C15—C14—C13	120.56 (16)
N2—C3—H3B	109.9	C15—C14—H14	119.7
C2—C3—H3B	109.9	C13—C14—H14	119.7
H3A—C3—H3B	108.3	C14—C15—C10	120.98 (15)
N2—C4—N1	118.91 (14)	C14—C15—H15	119.5
N2—C4—C5	121.85 (14)	C10—C15—H15	119.5
N1—C4—C5	119.23 (13)	O3—C16—H16A	109.5
C6—C5—C4	116.56 (13)	O3—C16—H16B	109.5
C6—C5—C9	122.47 (14)	H16A—C16—H16B	109.5
C4—C5—C9	120.86 (13)	O3—C16—H16C	109.5
C7—C6—C5	123.47 (15)	H16A—C16—H16C	109.5
C7—C6—H6	118.3	H16B—C16—H16C	109.5
C5—C6—H6	118.3		
C4—N1—C1—C2	−26.8 (2)	C6—C7—C8—O1	−179.74 (17)
C8—N1—C1—C2	155.27 (15)	C6—C7—C8—N1	0.5 (2)
N1—C1—C2—C3	54.2 (2)	C6—C5—C9—O2	167.23 (15)
C4—N2—C3—C2	26.0 (2)	C4—C5—C9—O2	−8.9 (2)
C1—C2—C3—N2	−52.5 (2)	C6—C5—C9—C10	−10.8 (2)
C3—N2—C4—N1	2.6 (2)	C4—C5—C9—C10	173.00 (14)
C3—N2—C4—C5	−176.66 (15)	O2—C9—C10—C11	−43.4 (2)
C8—N1—C4—N2	175.55 (14)	C5—C9—C10—C11	134.74 (16)
C1—N1—C4—N2	−2.2 (2)	O2—C9—C10—C15	132.67 (17)
C8—N1—C4—C5	−5.1 (2)	C5—C9—C10—C15	−49.2 (2)
C1—N1—C4—C5	177.15 (15)	C15—C10—C11—C12	2.4 (2)

N2—C4—C5—C6	−175.92 (14)	C9—C10—C11—C12	178.70 (14)
N1—C4—C5—C6	4.8 (2)	C10—C11—C12—C13	−2.7 (2)
N2—C4—C5—C9	0.5 (2)	C16—O3—C13—C12	−6.4 (2)
N1—C4—C5—C9	−178.82 (14)	C16—O3—C13—C14	173.73 (16)
C4—C5—C6—C7	−2.1 (2)	C11—C12—C13—O3	−179.36 (15)
C9—C5—C6—C7	−178.48 (15)	C11—C12—C13—C14	0.5 (2)
C5—C6—C7—C8	−0.5 (3)	O3—C13—C14—C15	−178.17 (15)
C4—N1—C8—O1	−177.41 (14)	C12—C13—C14—C15	1.9 (3)
C1—N1—C8—O1	0.4 (2)	C13—C14—C15—C10	−2.3 (3)
C4—N1—C8—C7	2.4 (2)	C11—C10—C15—C14	0.2 (2)
C1—N1—C8—C7	−179.77 (15)	C9—C10—C15—C14	−175.97 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.86	1.94	2.624 (2)	135

Fig. 1

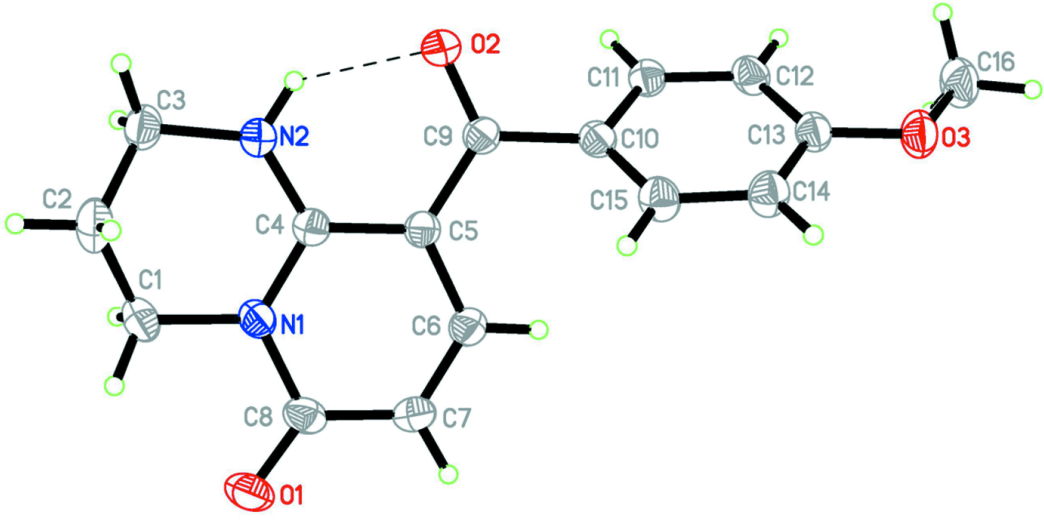

Fig. 2