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9-(4-Methyoxylbenzoyl)-1,2,3,4,7,8-hexahydropyrido[1,2-*a*]pyrimidin-6-one

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In the title compound, $C_{16}H_{18}N_2O_3$, there is an intramolecular N—H···O hydrogen bond. Intermolecular C—H···O bonds stabilize the crystal packing.

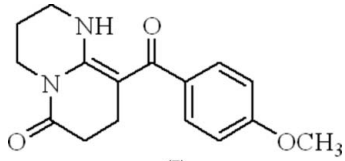
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.111
Data-to-parameter ratio = 12.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Heterocyclic ketene aminals (HKAs) (Huang & Wang, 1994), also known as cyclic 1,1-enediamines, are versatile synthons for heterocyclic synthesis. The title compound, (I) (Fig. 1), which possesses a pyrimidine ring, is a member of this family. The crystallographic data for (I) will provide valuable information for assessing its electronic conjugation properties and a possible intramolecular hydrogen bond (Wang *et al.*, 1987), which may be correlated with the reactivity of the secondary amine and the α-C atom (Huang & Wamhoff, 1984).



(I)

The C9═O2 double bond [1.2710 (19) Å] is longer than C8═O1 [1.2162 (18) Å]. The nominal C4═C5 double bond [1.402 (2) Å] has almost the same length as the nominal single bond C5—C9 [1.413 (2) Å]. On the other hand, the C4—N2 bond [1.3266 (19) Å] is shorter than a normal C—N single bond. The molecular conformation is stabilized by an intramolecular hydrogen bond [N2—H2 = 0.86 Å, H2···O2 = 1.87 Å, N2···O2 = 2.569 (1) Å and N2—H2···O2 = 137°]. The crystal packing of (I) is stabilized by intermolecular C—H···O hydrogen bonds (Fig. 2).

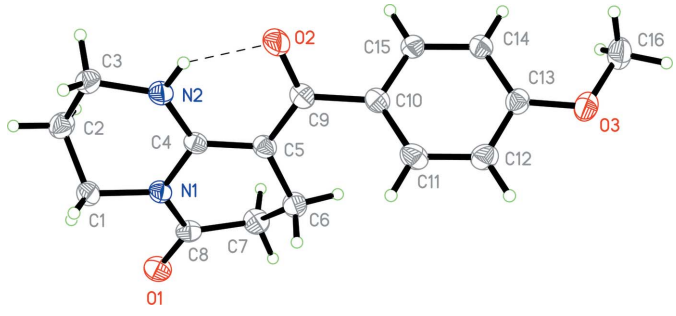


Figure 1
View of (I) showing 30% displacement ellipoids (spheres of arbitrary radius for the H atoms). The intramolecular hydrogen bond is indicated by a dashed line.

Experimental

Compound (I) was prepared according to the procedure reported by Zhao *et al.* (1993) and recrystallized from ethyl acetate in 91% yield (m.p. 456 K). Analysis calculated for $C_{16}H_{18}N_2O_3$: C 67.11, H 6.34, N 9.79%; found: C 67.08, H 6.35, N 9.76%.

Crystal data

$C_{16}H_{18}N_2O_3$
$M_r = 286.32$
Triclinic, $P\overline{1}$
$a = 7.5696$ (17) Å
$b = 9.725$ (2) Å
$c = 10.446$ (2) Å
$\alpha = 88.964$ (4)°
$\beta = 77.971$ (4)°
$\gamma = 68.169$ (3)°
$V = 696.6$ (3) Å^3
$Z = 2$
$D_x = 1.365$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 294$ (2) K
Prism, yellow
0.20 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.678$, $T_{max} = 1.000$
3539 measured reflections
2435 independent reflections
1890 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.111$
$S = 1.01$
2435 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.1302P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.052 (7)

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

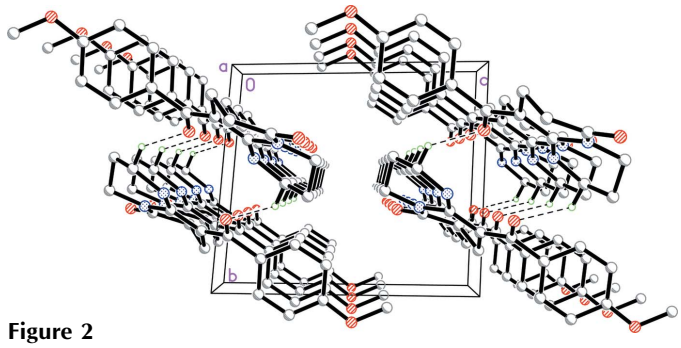


Figure 2
The molecular packing of (I), viewed along the *c* axis. H atoms bonded to C atoms have been omitted for clarity. Dashed lines indicate the hydrogen-bonding interactions.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2002) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXTL*.

Dr Hai-Bing Song of Nankai University collected the data and is gratefully acknowledged.

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