

Yu-Cheng Wang, Xiang-Shan Wang,* Zhao-Sen Zeng and Da-Qing Shi

Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China

Correspondence e-mail:
xswang1974@yahoo.com

Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.047
 wR factor = 0.137
Data-to-parameter ratio = 15.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

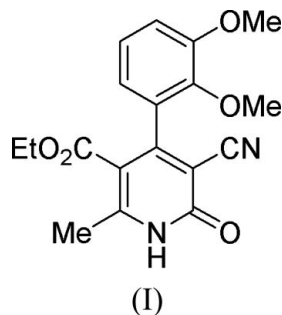
Ethyl 5-cyano-4-(2,3-dimethoxyphenyl)-2-methyl-6-oxopyridine-3-carboxylate

The title compound, $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_5$, was synthesized from 2,3-dimethoxyphenylmethylidenemalononitrile and ethyl acetate in the presence of triethylbenzylammonium chloride in an aqueous medium. The pyridone and benzene rings make a dihedral angle of $63.8(1)^\circ$. There are intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

It is known that many pyridine derivatives exhibit a wide spectrum of pharmacological activities and biological activities, such as inhibitory activity (Liu *et al.*, 2002), antimicrobial activity (Aytemir *et al.*, 2003) and anti-inflammatory activity (Ozturk *et al.*, 2002). We report here the crystal structure of the title compound, (I). Its aqueous synthesis (see *Experimental*) was inspired by the work of Breslow & Rideout (1980), who promoted the use of water as a solvent in organic chemistry.



The pyridone ring (C1—C5/N1) is essentially planar (Fig. 1), with a maximum deviation of $0.026(1)$ Å for C2. This ring forms a dihedral angle of $63.8(1)^\circ$ with the benzene ring (C11—C16). Molecules form centrosymmetric dimers by $\text{N1}-\text{H1A}\cdots\text{O1}$ ($1-x, 3-y, 1-z$) hydrogen bonds (Table 2), and they are further linked *via* $\text{C16}-\text{H16A}\cdots\text{O1}$ ($x, y-1, z$) interactions (Fig. 2).

Experimental

Compound (I) was prepared by the reaction of 2,3-dimethoxyphenylmethylidenemalononitrile (0.42 g, 2 mmol) and ethyl acetate (0.39 g, 3 mmol) in the presence of triethylbenzylammonium chloride (0.1 g) in water at 363 K for 8 h (yield 93%, mp. 513–515 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a DMF solution. ^1H NMR (CDCl_3): 0.89 (*t*, $J = 7.2$ Hz, 3H, CH_3), 2.68 (*s*, 3H, CH_3), 3.86 (*s*, 3H, OCH_3), 3.92 (*s*, 3H, OCH_3), 4.00 (*q*, $J = 7.2$ Hz, 2H, CH_2), 6.77 (*dd*, $J = 1.2$ Hz, $J' = 7.6$ Hz, 1H, ArH), 7.04 (*dd*, $J = 1.2$ Hz, $J' = 8.0$ Hz, 1H, ArH), 6.14 (*dd*, $J = 7.6$ Hz,

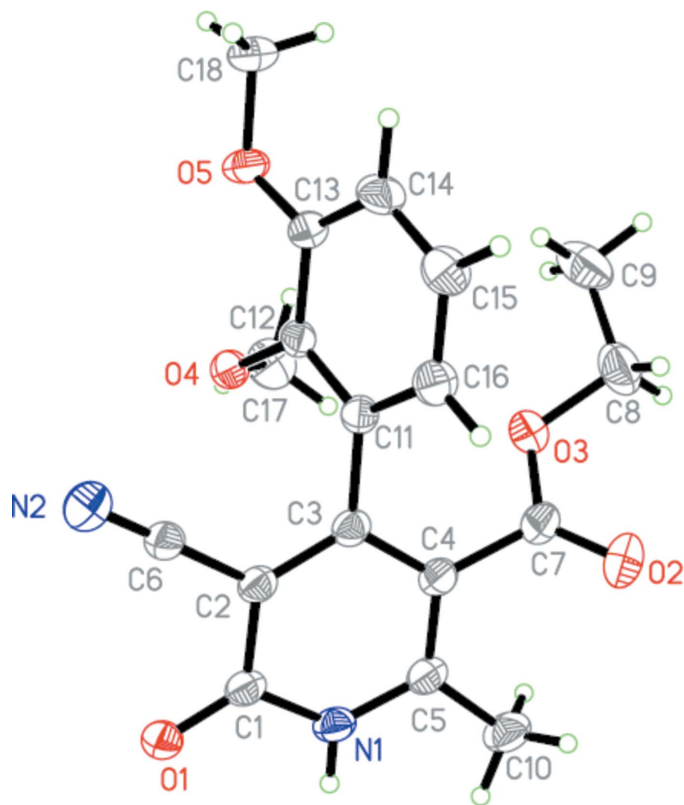


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

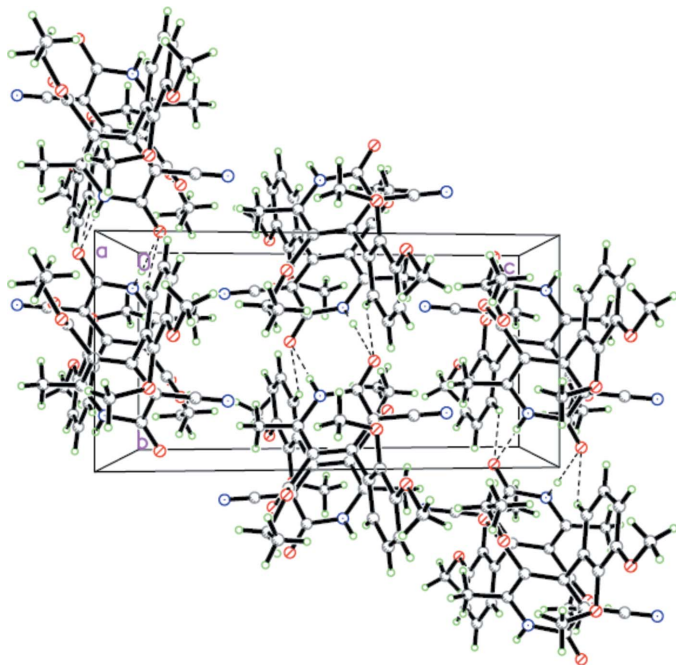


Figure 2
A packing diagram of (I), viewed along *c*. Dashed lines indicate hydrogen bonds.

$J' = 8.0$ Hz, 1H, ArH), IR (cm^{-1}): 3052 (ArH), 2852 (C–H), 2224 (CN), 1722, 1661 (C=O), 1592, 1470, 1430 (benzene ring).

Crystal data

$\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_5$
 $M_r = 342.34$
Monoclinic, $P2_1/c$
 $a = 15.2031$ (14) Å
 $b = 7.5946$ (7) Å
 $c = 15.1270$ (14) Å
 $\beta = 100.143$ (2)°
 $V = 1719.3$ (3) Å³
 $Z = 4$

$D_x = 1.323$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 2339 reflections
 $\theta = 2.7$ – 25.0°
 $\mu = 0.10$ mm⁻¹
 $T = 292$ (2) K
Block, colorless
 $0.2 \times 0.1 \times 0.1$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: none
8803 measured reflections
3379 independent reflections

2559 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -18 \rightarrow 18$
 $k = -8 \rightarrow 9$
 $l = -15 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.137$
 $S = 1.07$
3379 reflections
226 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 0.1609P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C1–N1	1.374 (2)	C3–C4	1.419 (2)
C1–C2	1.435 (2)	C4–C5	1.376 (2)
C2–C3	1.376 (2)	C5–N1	1.356 (2)
N1–C1–C2	114.33 (15)	C5–C4–C3	119.53 (16)
C3–C2–C1	121.70 (15)	N1–C5–C4	118.67 (15)
C2–C3–C4	119.42 (14)	C5–N1–C1	126.15 (14)
N1–C1–C2–C3	3.4 (2)	C3–C4–C7–O3	–52.4 (2)
C1–C2–C3–C4	–5.2 (2)	C2–C3–C11–C12	–65.9 (2)
C2–C3–C4–C5	2.8 (2)	C4–C5–N1–C1	–2.9 (3)
C3–C4–C5–N1	1.1 (2)	C2–C1–N1–C5	0.7 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C16–H16A...O1 ⁱ	0.93	2.48	3.263 (1)	141
N1–H1A...O1 ⁱⁱ	0.86	1.92	2.780 (2)	177

Symmetry codes: (i) $x, y - 1, z$; (ii) $-x + 1, -y + 3, -z + 1$.

The H atoms were positioned geometrically and refined as riding, with C–H = 0.93–0.97 Å and N–H = 0.86 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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