

6-Acetyl-2-(*N*-methylamido)pyridineBi-Yun Su,<sup>a</sup> Jian-She Zhao<sup>a</sup> and  
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## Key indicators

Single-crystal X-ray study  
*T* = 295 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$   
*R* factor = 0.046  
*wR* factor = 0.165  
Data-to-parameter ratio = 8.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

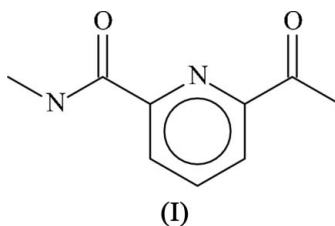
In the title crystal structure,  $\text{C}_9\text{H}_{10}\text{N}_2\text{O}_2$ , a weak  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond between the amide group of the methylamide substituent and the acetyl substituent of an adjacent molecule gives rise to a helical chain which propagates along the *c*-axis direction of the orthorhombic unit cell. The molecule is essentially flat.

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## Comment

Ethyl 6-acetyl-2-pyridinecarboxylate condenses with 2,6-disubstituted anilines to afford the Schiff bases,  $2-(\text{EtO}_2\text{C})-\text{C}_5\text{H}_3\text{N}-6-\text{C}(\text{Me})=\text{NC}_6\text{H}_3\text{X}_2$ , which can form complexes with transition metals in which the Schiff base *N,N'*-chelates to the metal atom. These metal complexes have been studied for their activity as olefin polymerization catalysts (Sun *et al.*, 2004). When ethyl 6-acetyl-2-pyridinecarboxylate is treated with methylamine under microwave radiation, a reaction occurs through the carboethoxy group instead of through the acetyl group, yielding 2-methylamido-6-acetylpyridine. The molecule is nearly planar. Adjacent molecules are linked by a weak  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond (Table 1) into a helical chain.



## Experimental

2,6-Pyridinedicarboxylic acid was converted to the diethyl ester (Levkoeva *et al.*, 1976); the diethyl ester was dissolved in freshly distilled ethyl acetate and then treated with sodium ethoxide followed by hydrolysis with hydrochloric acid to yield ethyl 6-acetyl-2-pyridinecarboxylate (Sun *et al.*, 2004). Ethyl 6-acetyl-2-pyridinecarboxylate (0.078 g, 0.4 mmol) and methylamine (0.114 g, 1.04 mmol) were placed in a 25-ml flask; the mixture was subjected to radiation in a 800 W microwave oven for 10 minutes on a medium-high setting (Yang *et al.*, 2002). The product was purified using column chromatography to yield pure 2-methylamido-6-acetylpyridine in about 60% yield; m.p. 371–373 K. Recrystallization was effected with a mixture of diethyl ether and *n*-pentane (1:1 *v/v*). The formulation was confirmed by <sup>1</sup>H-NMR and mass spectroscopic analysis.

Crystal data

C<sub>9</sub>H<sub>10</sub>N<sub>2</sub>O<sub>2</sub>  
*M<sub>r</sub>* = 178.19  
 Orthorhombic, *Pca*2<sub>1</sub>  
*a* = 22.784 (1) Å  
*b* = 4.3504 (2) Å  
*c* = 9.2672 (6) Å  
*V* = 918.56 (8) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.289 Mg m<sup>-3</sup>

Mo *K*α radiation  
 Cell parameters from 756 reflections  
 $\theta$  = 2.8–21.3°  
 $\mu$  = 0.09 mm<sup>-1</sup>  
*T* = 295 (2) K  
 Block, colorless  
 0.40 × 0.19 × 0.18 mm

Data collection

Bruker APEX-II area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: none  
 4164 measured reflections  
 1061 independent reflections

621 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.036  
 $\theta_{\text{max}}$  = 27.0°  
*h* = -29 → 20  
*k* = -5 → 5  
*l* = -9 → 11

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.046  
*wR*(*F*<sup>2</sup>) = 0.165  
*S* = 0.96  
 1061 reflections  
 121 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.1074P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 ( $\Delta/\sigma$ )<sub>max</sub> = 0.001  
 $\Delta\rho_{\text{max}}$  = 0.13 e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}}$  = -0.18 e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

O1—C2	1.219 (6)	C2—C3	1.517 (5)
O2—C8	1.207 (5)	C3—C4	1.388 (6)
N1—C3	1.329 (5)	C4—C5	1.331 (6)
N1—C7	1.345 (5)	C5—C6	1.376 (7)
N2—C8	1.334 (6)	C6—C7	1.367 (7)
N2—C9	1.461 (7)	C7—C8	1.502 (6)
C1—C2	1.441 (7)		
C3—N1—C7	116.9 (3)	C4—C5—C6	119.0 (4)
C8—N2—C9	121.5 (4)	C7—C6—C5	119.7 (4)
O1—C2—C1	122.0 (4)	N1—C7—C6	122.1 (4)
O1—C2—C3	119.8 (4)	N1—C7—C8	118.2 (3)
C1—C2—C3	118.2 (4)	C6—C7—C8	119.7 (4)
N1—C3—C4	123.2 (4)	O2—C8—N2	123.0 (4)
N1—C3—C2	116.1 (4)	O2—C8—C7	121.6 (4)
C4—C3—C2	120.7 (4)	N2—C8—C7	115.5 (3)
C5—C4—C3	119.1 (4)		

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>
N2—H2 <i>n</i> ...O1 <sup>i</sup>	0.86	2.41	3.152 (5)	145

Symmetry code: (i) -*x* + 1, -*y* + 2, *z* - ½.

All H atoms were positioned geometrically [*C*—H = 0.96 Å, *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(C) for the methyl H atoms, *C*—H = 0.93 Å, *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C) for the aromatic H atoms and N—H = 0.86 Å, *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(N)] and allowed to ride on their parent atoms in the riding-model approximation. The methyl groups were rotated to fit the electron density. In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve

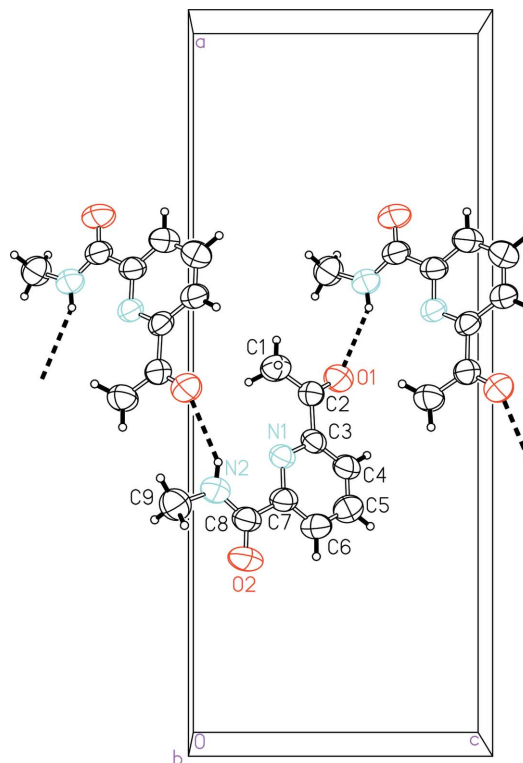


Figure 1

Partial packing plot (Johnson, 1976) of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii and hydrogen bonds are shown as dashed lines.

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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