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1-[6-(6-Acetylpyridin-2-yl)pyridin-2-yl]-ethanone

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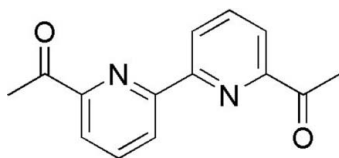
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.042; wR factor = 0.105; data-to-parameter ratio = 16.1.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_2$, the asymmetric unit comprises one half-molecule with an inversion center between the pyridine rings. The rings are *trans* coplanar with the acetyl groups deviating slightly from the mean planes, making a dihedral angle of $4.63(4)^\circ$. In the crystal, molecules are linked by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a supramolecular sheet parallel to (100).

Related literature

The compound is of interest with respect to supramolecular chemistry as a precursor for polypyridyl bridging ligands. For related structures, see: Parks *et al.* (1973); Potts *et al.* (1993); Zong *et al.* (2006); Şengül *et al.* (1998); Agac *et al.* (2010); Iyoda *et al.* (1990); Janiak *et al.* (1999); O'Donnell & Steel (2010); Kochel (2005). For applications of related structures, see: Parks *et al.* (1973); Iyoda *et al.* (1990); Şengül *et al.* (2009); Agac *et al.* (2010).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_2$	$V = 588.50(6) \text{ \AA}^3$
$M_r = 240.26$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 3.9338(2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 13.8005(8) \text{ \AA}$	$T = 120 \text{ K}$
$c = 10.8728(6) \text{ \AA}$	$0.50 \times 0.20 \times 0.20 \text{ mm}$
$\beta = 94.437(4)^\circ$	

Data collection

Bruker–Nonius KappaCCD diffractometer with APEXII area detector	10564 measured reflections 1336 independent reflections 1220 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2007)	$R_{\text{int}} = 0.034$
$T_{\text{min}} = 0.955$, $T_{\text{max}} = 0.982$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$	83 parameters
$wR(F^2) = 0.105$	H-atom parameters constrained
$S = 1.10$	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
1336 reflections	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C3}-\text{H3}\cdots\text{O1}^i$	0.95	2.56	3.2992 (16)	135

 Symmetry code: (i) $x + 1, -y + \frac{1}{2}, z + \frac{1}{2}$

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO (Otwinowski & Minor, 1997) and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: publCIF (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2286).

References

- Agac, C., Yilmaz, I., Sengul, A. & Coles, S. J. (2010). *Turk. J. Chem.* **34**, 781–791.
- Hooft, R. (1998). COLLECT. Nonius BV, Delft, The Netherlands.
- Iyoda, M., Otsuka, H., Sato, K., Nisato, N. & Oda, M. (1990). *Bull. Chem. Soc. Jpn.* **63**, 80–87.
- Janiak, C., Deblon, S., Wu, H.-P., Kolm, M. J., Klüfers, P., Piotrowski, H. & Mayer, P. (1999). *Eur. J. Inorg. Chem.* **1999**, 1507–1521.
- Kochel, A. (2005). *Acta Cryst.* **E61**, o926–o927.
- O'Donnell, M. A. & Steel, P. J. (2010). *Acta Cryst.* **E66**, m1630.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Parks, J. E., Wagner, B. E. & Holm, R. H. (1973). *J. Organomet. Chem.* **56**, 53–66.
- Potts, K. T., Raiford, K. A. G. & Keshavarz-K, M. (1993). *J. Am. Chem. Soc.* **115**, 2793–2807.
- Şengül, A., Hursthouse, M. B., Coles, S. J. & Gillard, R. D. (1998). *Acta Cryst.* **C54**, 661–662.
- Şengül, A., Wang, W.-J. & Coles, S. J. (2009). *Polyhedron*, **28**, 69–76.
- Sheldrick, G. M. (2007). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Zong, R., Wang, D., Hammit, R. & Thummel, R. P. (2006). *J. Org. Chem.* **71**, 167–175.

supporting information

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1-[6-(6-Acetylpyridin-2-yl)pyridin-2-yl]ethanone

Huseyin Zekeriya Dogan, Abdurrahman Sengul and Simon John Coles

S1. Comment

The principles of supramolecular chemistry provide guidelines for the construction of quite complex molecules or constructs from relatively simple components. In this respect, 6,6'-diacetyl-2,2'-bipyridine, acting as a diketone has been widely used as a precursor or building block for the construction of polypyridine bridging ligands [Şengül *et al.*, 2009; Agac *et al.*, 2010; Potts *et al.*, 1993; Zong *et al.*, 2006]. The well established coordination ability of 2,2'-bipyridine suggests that ligands containing multiple pyridine rings joined through their 2,6-positions would be ideal for the self-assembly of mono-, double-, or triple-stranded helicates containing one or more transition-metal cations and producing a variety of coordination geometries and architectures. This area is therefore of interest with respect to supramolecular chemistry as a precursor for polypyridyl bridging ligands (Janiak *et al.*, 1999; Potts *et al.*, 1993; Zong *et al.*, 2006) and derivatives are important materials for the preparation of oximes or other functionalities (Iyoda *et al.*, 1990; Parks *et al.*, 1973; Agac *et al.*, 2010).

As a continuation of work on the structures of such compounds (Şengül *et al.*, 1998) the title compound derived from the coupling of 6-bromo-2-acetylpyridine is reported herein. The molecule of the title compound (Fig. 1.) possesses a twofold symmetry where each of the pyridyl rings are *trans* to each other, forming an essentially planar structure. The bond lengths have normal values (Şengül *et al.*, 1998), and are comparable to those observed in similar compounds (Janiak *et al.*, 1999; O'Donnell & Steel, 2010; Kochel, 2005; Şengül *et al.* 2009).

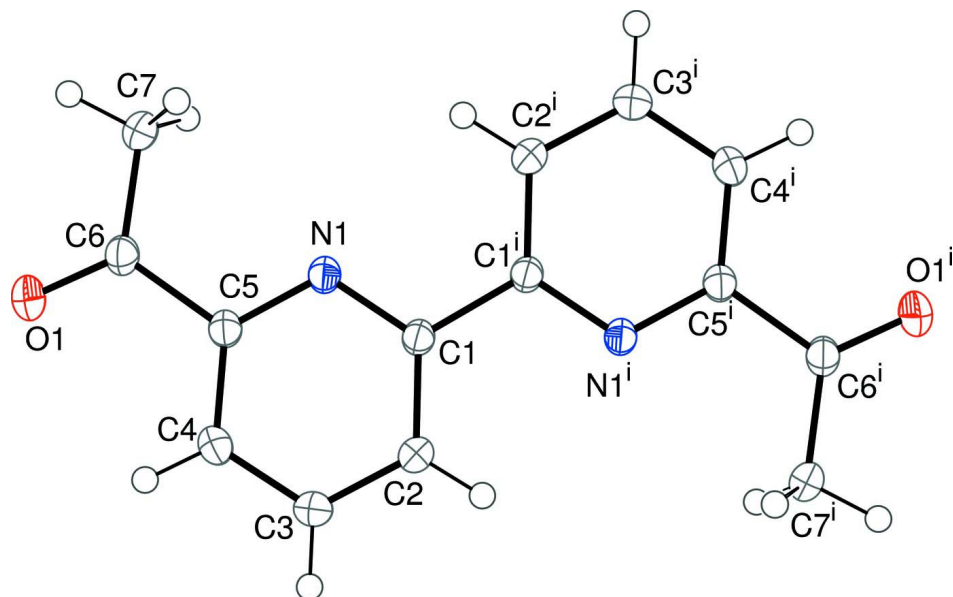
In the crystal, molecules are linked through intermolecular C-H...O H-bonds (Table 1) to form a supramolecular network parallel to (100) (Fig. 1).

S2. Experimental

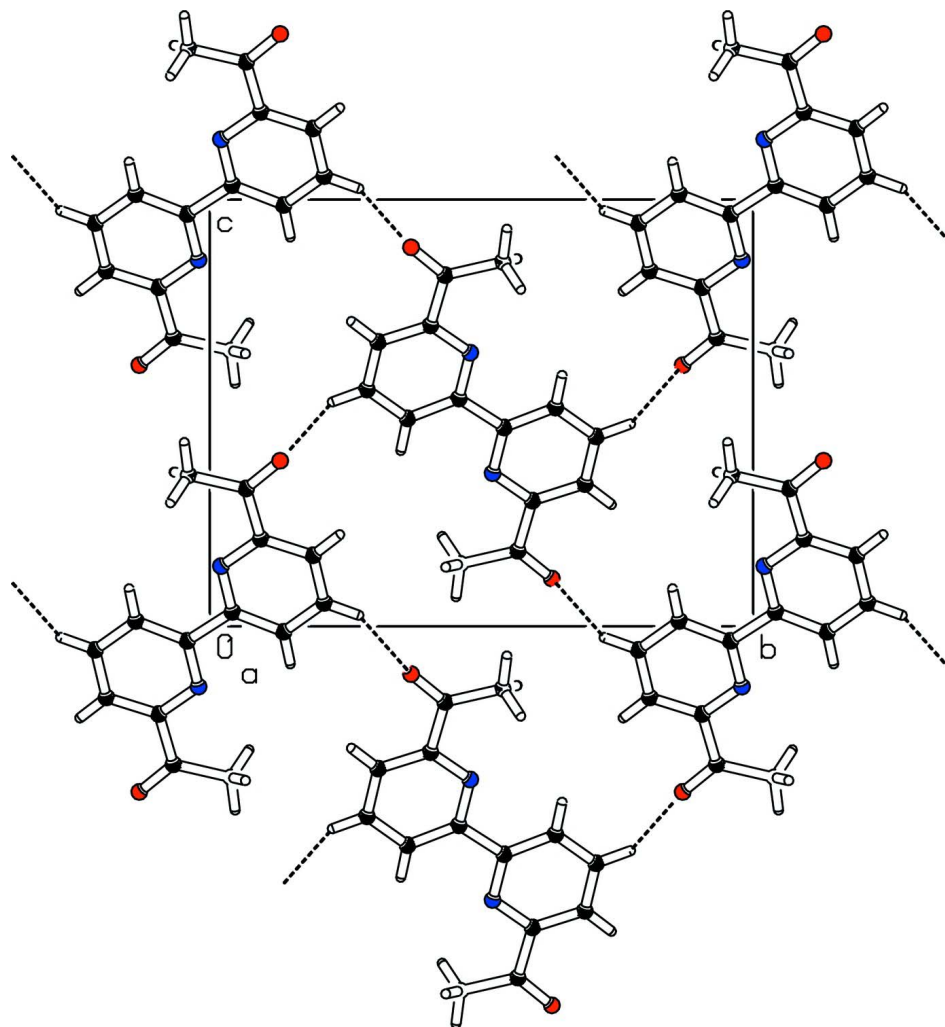
The title compound was synthesized by the reported method of homocoupling of aryl halides using Ni(II) complex and zinc in the presence of triphenylphosphine by Janiak *et al.* (1999). The spectroscopic and analytical data are in good agreement with the reported values in literature by Zong *et al.*, 2006; Potts *et al.*, 1993; Agac *et al.*, 2010 and Parks *et al.*, 1973. The solid was crystallized from dichloromethane to afford colourless needles suitable for X-ray diffraction. Mp.: 178.5–179.5 °C. ¹H-NMR (dms-*d*₆, δ_{p.p.m.}): 8.81(d, 2H, *J*_{3,4} = 8 Hz, H3,3'), 8.23(d, 2H, *J*_{5,4} = 7 Hz, H5,5'), 8.07(dd, 2H, *J*_{4,3} = 8.2 Hz, *J*_{4,5} = 1 Hz, H4,4'), 2.79(s, 6H, 2xCH₃). *Calc.* for C₁₄H₁₂N₂O₂: C, 69.99; H, 5.03; N, 11.66 Found: C, 62.54; H, 4.54; N, 11.68%. IR (ATR, ν cm⁻¹): 3056 (CH_{ar}), 2990 (CH_{al}), 1590 (C=O), 1487 and 1437 (C=N and C=C), 1311, 1182, 1120, 1094, 1071, 995, 861, 748, 720. UV-Vis (MeCN, λ_{max}/nm): 286, 258, 219.

S3. Refinement

Hydrogen atoms were fixed in idealized positions [0.98 Å (CH₃) & 0.95 Å (CH)] and refined using the riding model with *U*_{iso} (H) set to 1.5 and 1.2*U*_{eq}(carrier) respectively.

**Figure 1**

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Symmetry code: (i) -x, -y, -z

**Figure 2**

Intermolecular C=O...H contacts forming a supramolecular sheet along the *a* axis

1-[6-(6-Acetylpyridin-2-yl)pyridin-2-yl]ethanone

Crystal data

$C_{14}H_{12}N_2O_2$

$M_r = 240.26$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1bc$

$a = 3.9338\ (2)\ \text{\AA}$

$b = 13.8005\ (8)\ \text{\AA}$

$c = 10.8728\ (6)\ \text{\AA}$

$\beta = 94.437\ (4)^\circ$

$V = 588.50\ (6)\ \text{\AA}^3$

$Z = 2$

$F(000) = 252$

$D_x = 1.356\ \text{Mg m}^{-3}$

Melting point: 452 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 10564 reflections

$\theta = 2.9\text{--}27.5^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 120\ \text{K}$

Rod, colourless

$0.50 \times 0.20 \times 0.20\ \text{mm}$

Data collection

Bruker–Nonius Kappa CCD
 diffractometer with APEXII area detector
 Radiation source: Bruker–Nonius FR591
 rotating anode
 10cm confocal mirrors monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2007)
 $T_{\min} = 0.955$, $T_{\max} = 0.982$

10564 measured reflections
 1336 independent reflections
 1220 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.8^\circ$
 $h = -5 \rightarrow 4$
 $k = -17 \rightarrow 17$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.105$
 $S = 1.10$
 1336 reflections
 83 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 0.237P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4161 (3)	0.54168 (8)	0.46613 (10)	0.0185 (3)
C2	0.4431 (3)	0.63598 (9)	0.51269 (11)	0.0234 (3)
H2	0.5670	0.6485	0.5895	0.028*
C3	0.2863 (3)	0.71096 (9)	0.44508 (12)	0.0277 (3)
H3	0.3018	0.7757	0.4748	0.033*
C4	0.1063 (3)	0.69010 (9)	0.33338 (12)	0.0248 (3)
H4	-0.0011	0.7402	0.2846	0.030*
C5	0.0871 (3)	0.59397 (8)	0.29474 (10)	0.0197 (3)
C6	-0.1175 (3)	0.56675 (9)	0.17703 (11)	0.0216 (3)
C7	-0.1483 (3)	0.46102 (9)	0.14615 (11)	0.0247 (3)
H7A	-0.2969	0.4528	0.0703	0.037*
H7B	-0.2460	0.4266	0.2139	0.037*
H7C	0.0780	0.4346	0.1341	0.037*
N1	0.2396 (2)	0.52066 (7)	0.35884 (9)	0.0190 (2)
O1	-0.2556 (3)	0.62953 (7)	0.11205 (8)	0.0315 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0184 (5)	0.0192 (6)	0.0174 (5)	-0.0012 (4)	-0.0011 (4)	0.0013 (4)
C2	0.0270 (6)	0.0206 (6)	0.0215 (6)	-0.0012 (5)	-0.0047 (5)	-0.0012 (4)
C3	0.0333 (7)	0.0185 (6)	0.0297 (7)	0.0004 (5)	-0.0073 (5)	-0.0017 (5)
C4	0.0275 (6)	0.0199 (6)	0.0259 (6)	0.0018 (5)	-0.0053 (5)	0.0032 (5)
C5	0.0199 (6)	0.0200 (6)	0.0189 (5)	0.0005 (4)	-0.0012 (4)	0.0018 (4)
C6	0.0210 (6)	0.0234 (6)	0.0199 (6)	0.0019 (4)	-0.0019 (4)	0.0016 (4)

C7	0.0258 (6)	0.0245 (6)	0.0225 (6)	0.0006 (5)	-0.0063 (5)	-0.0017 (5)
N1	0.0191 (5)	0.0197 (5)	0.0178 (5)	0.0000 (4)	-0.0014 (4)	0.0016 (4)
O1	0.0384 (6)	0.0282 (5)	0.0259 (5)	0.0068 (4)	-0.0104 (4)	0.0033 (4)

Geometric parameters (Å, °)

C1—N1	1.3423 (15)	C4—H4	0.9500
C1—C2	1.3975 (16)	C5—N1	1.3433 (14)
C1—C1 ⁱ	1.492 (2)	C5—C6	1.5058 (16)
C2—C3	1.3861 (17)	C6—O1	1.2189 (15)
C2—H2	0.9500	C6—C7	1.5000 (17)
C3—C4	1.3878 (17)	C7—H7A	0.9800
C3—H3	0.9500	C7—H7B	0.9800
C4—C5	1.3919 (17)	C7—H7C	0.9800
N1—C1—C2	122.39 (11)	N1—C5—C6	116.16 (10)
N1—C1—C1 ⁱ	116.22 (12)	C4—C5—C6	120.48 (10)
C2—C1—C1 ⁱ	121.39 (13)	O1—C6—C7	122.48 (11)
C3—C2—C1	119.01 (11)	O1—C6—C5	120.02 (11)
C3—C2—H2	120.5	C7—C6—C5	117.49 (10)
C1—C2—H2	120.5	C6—C7—H7A	109.5
C2—C3—C4	119.03 (11)	C6—C7—H7B	109.5
C2—C3—H3	120.5	H7A—C7—H7B	109.5
C4—C3—H3	120.5	C6—C7—H7C	109.5
C3—C4—C5	118.28 (11)	H7A—C7—H7C	109.5
C3—C4—H4	120.9	H7B—C7—H7C	109.5
C5—C4—H4	120.9	C1—N1—C5	117.92 (10)
N1—C5—C4	123.35 (11)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C3—H3 \cdots O1 ⁱⁱ	0.95	2.56	3.2992 (16)	135

Symmetry code: (ii) $x+1, -y+3/2, z+1/2$.