

4-Acetylpyridinium iodide

Jie Xu* and Xue-qun Fu

Ordered Matter Science Research Center, Southeast University, Nanjing 210096, People's Republic of China

Correspondence e-mail: fuxuequn222@163.com

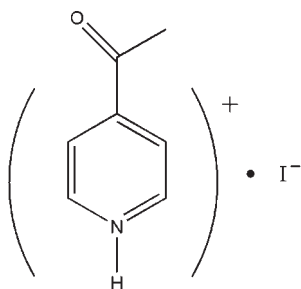
Received 24 May 2010; accepted 8 June 2010

 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.010$ Å; R factor = 0.040; wR factor = 0.124; data-to-parameter ratio = 22.0.

In the title compound, $\text{C}_7\text{H}_8\text{NO}^+\cdot\text{I}^-$, $\text{N}-\text{H}\cdots\text{I}$ hydrogen bonding and $\pi-\pi$ stacking interactions [centroid-centroid distance = 5.578 (4) Å] stabilize the structure.

Related literature

For background to phase transition materials, see: Li *et al.* (2008); Zhang *et al.* (2009). For 4-acetylpyridine as a ligand in coordination compounds, see: Steffen & Palenik (1977); Pang *et al.* (1994). For other structures involving 4-acetylpyridine, see: Fu (2009*a,b*); Majerz *et al.* (1991).



Experimental

Crystal data

$\text{C}_7\text{H}_8\text{NO}^+\cdot\text{I}^-$
 $M_r = 249.04$
 Monoclinic, $P2_1/c$
 $a = 8.5144$ (17) Å
 $b = 5.0926$ (10) Å

$c = 21.714$ (6) Å
 $\beta = 111.37$ (3)°
 $V = 876.8$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 3.59$ mm⁻¹
 $T = 298$ K

0.40 × 0.30 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.286$, $T_{\max} = 0.488$

8420 measured reflections
 2006 independent reflections
 1805 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.124$
 $S = 0.90$
 2006 reflections

91 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.70$ e Å⁻³
 $\Delta\rho_{\min} = -0.62$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{I1}^{\text{i}}$	0.86	2.67	3.456 (6)	153

 Symmetry code: (i) $-x + 1, -y, -z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

The authors are grateful to the starter fund of Southeast University for financial support to purchase the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2163).

References

- Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
 Fu, X. (2009*a*). *Acta Cryst.* **E65**, o1804.
 Fu, X. (2009*b*). *Acta Cryst.* **E65**, o2385.
 Li, X. Z., Qu, Z. R. & Xiong, R. G. (2008). *Chin. J. Chem.* **11**, 1959–1962.
 Majerz, I., Malarski, Z. & Sawka-Dobrowolska, W. (1991). *J. Mol. Struct.* **249**, 109–116.
 Pang, L., Whitehead, M. A., Bermardinelli, G. & Lucken, E. A. C. (1994). *J. Chem. Crystallogr.* **24**, 203–211.
 Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Steffen, W. L. & Palenik, G. J. (1977). *Inorg. Chem.* **16**, 1119–1128.
 Zhang, W., Chen, L. Z., Xiong, R. G., Nakamura, T. & Huang, S. D. (2009). *J. Am. Chem. Soc.* **131**, 12544–12545.

supplementary materials

Acta Cryst. (2010). E66, o1628 [doi:10.1107/S1600536810021860]

4-Acetylpyridinium iodide

J. Xu and X. Fu

Comment

As a continuation of our study of phase transition materials, including organic ligands (Li *et al.*, 2008), metal-organic coordination compounds (Zhang *et al.*, 2009), organic-inorganic hybrids, we studied the dielectric properties of the title compound, unfortunately, there was no distinct anomaly observed from 93 K to 350 K, (subliming above 388 K), suggesting that this compound should be not a real ferroelectrics or there may be no distinct phase transition occurred within the measured temperature range. In this article, the crystal structure of the title compound has been presented.

4-Acetylpyridine may be used as a ligand in coordination compounds *e.g.* with Zn (Steffen & Palenik, 1977) or Ni (Pang *et al.*, 1994). The crystal structures of 4-acetylpyridine together with pentachlorophenol (Majerz *et al.* 1991) and inorganic acids are also known *e.g.* with sulfuric acid (Fu, 2009b) and perchloric acid (Fu, 2009a).

The asymmetric unit of the title compound is built up from an protonated 4-acetylpyridinium cation wherein the acetyl group deviates 28.0 (5)° from the plane formed by the non-hydrogen atoms of the pyridine ring and a Γ anion (Fig. 1). The C1—C2—O1 bond angle and O1—C2—C3—C4 torsion angle are 122.6 (8)° and 27.8 (9)°, respectively. N—H...I hydrogen bonding (N...I distance 3.456 (6) Å) and π - π stacking interaction with the adjacent interplanar spacing of 5.578 (4) Å make great contribution to the stability of the crystal structure.

Experimental

1.19 g (10 mmol) 4-acetylpyridine was firstly dissolved in 50 ml ethanol, to which hydroiodic acid aqueous solution (40%, w/w) was then added until the solution became acidic under stirring. Single crystals of (I) were prepared by slow evaporation at room temperature of the acidic solution after 3 days.

Refinement

Positional parameters of all the H atoms were calculated geometrically and were allowed to ride on the C and N atoms to which they are bonded, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl group and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Figures

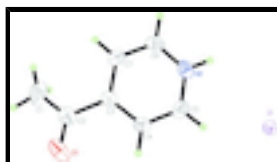


Fig. 1. The molecular structure of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level, and all H atoms have been omitted for clarity.

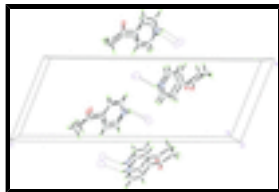


Fig. 2. A view of the packing of the title compound, stacking along the *b* axis. Dashed lines indicate hydrogen bonds.

4-acetylpyridinium iodide

Crystal data

$C_7H_8NO^+I^-$

$M_r = 249.04$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.5144$ (17) Å

$b = 5.0926$ (10) Å

$c = 21.714$ (6) Å

$\beta = 111.37$ (3)°

$V = 876.8$ (3) Å³

$Z = 4$

$F(000) = 472$

$D_x = 1.887$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4122 reflections

$\theta = 3.0$ – 27.6 °

$\mu = 3.59$ mm⁻¹

$T = 298$ K

Prism, colourless

$0.40 \times 0.30 \times 0.20$ mm

Data collection

Rigaku SCXmini
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: 13.6612 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)

$T_{\min} = 0.286$, $T_{\max} = 0.488$

8420 measured reflections

2006 independent reflections

1805 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.8$ °

$h = -11 \rightarrow 11$

$k = -6 \rightarrow 6$

$l = -28 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.124$

$S = 0.90$

2006 reflections

91 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 6.0704P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.70$ e Å⁻³

0 restraints

$$\Delta\rho_{\min} = -0.62 \text{ e } \text{\AA}^{-3}$$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
II	0.78163 (5)	0.05660 (9)	0.07248 (2)	0.05081 (18)
O1	0.3686 (9)	1.1056 (11)	0.2117 (3)	0.0769 (18)
N1	0.3294 (8)	0.3813 (11)	0.0580 (3)	0.0531 (13)
H1A	0.3405	0.2717	0.0297	0.064*
C2	0.2855 (10)	0.9025 (15)	0.2014 (3)	0.0546 (16)
C7	0.1674 (9)	0.5509 (14)	0.1144 (3)	0.0512 (15)
H7A	0.0675	0.5531	0.1226	0.061*
C5	0.4569 (9)	0.5415 (15)	0.0887 (4)	0.0552 (16)
H5A	0.5549	0.5357	0.0792	0.066*
C6	0.1850 (9)	0.3817 (13)	0.0688 (4)	0.0528 (16)
H6A	0.0978	0.2686	0.0455	0.063*
C3	0.2973 (8)	0.7194 (12)	0.1488 (3)	0.0412 (12)
C4	0.4428 (8)	0.7145 (13)	0.1342 (3)	0.0478 (14)
H4A	0.5307	0.8293	0.1556	0.057*
C1	0.1781 (13)	0.832 (3)	0.2385 (4)	0.093 (3)
H1B	0.1832	0.9683	0.2697	0.140*
H1C	0.2171	0.6700	0.2617	0.140*
H1D	0.0638	0.8111	0.2085	0.140*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.0462 (3)	0.0495 (3)	0.0573 (3)	-0.00662 (19)	0.01939 (19)	-0.00989 (18)
O1	0.122 (5)	0.044 (3)	0.059 (3)	-0.005 (3)	0.026 (3)	-0.008 (2)
N1	0.071 (4)	0.041 (3)	0.049 (3)	0.000 (3)	0.024 (3)	-0.001 (2)
C2	0.062 (4)	0.054 (4)	0.042 (3)	0.007 (3)	0.012 (3)	0.005 (3)
C7	0.044 (3)	0.055 (4)	0.056 (4)	-0.002 (3)	0.020 (3)	0.004 (3)
C5	0.054 (4)	0.057 (4)	0.063 (4)	0.001 (3)	0.031 (3)	0.005 (3)
C6	0.052 (4)	0.039 (3)	0.061 (4)	-0.008 (3)	0.013 (3)	-0.002 (3)
C3	0.049 (3)	0.036 (3)	0.035 (3)	0.003 (2)	0.010 (2)	0.006 (2)
C4	0.047 (3)	0.045 (3)	0.049 (3)	-0.010 (3)	0.014 (3)	0.000 (3)

supplementary materials

C1 0.094 (7) 0.140 (10) 0.057 (5) 0.014 (7) 0.040 (5) -0.003 (6)

Geometric parameters (\AA , $^\circ$)

O1—C2	1.227 (9)	C5—C4	1.362 (10)
N1—C5	1.327 (9)	C5—H5A	0.9300
N1—C6	1.332 (10)	C6—H6A	0.9300
N1—H1A	0.8600	C3—C4	1.385 (9)
C2—C1	1.468 (11)	C4—H4A	0.9300
C2—C3	1.506 (9)	C1—H1B	0.9600
C7—C6	1.363 (10)	C1—H1C	0.9600
C7—C3	1.383 (9)	C1—H1D	0.9600
C7—H7A	0.9300		
C5—N1—C6	123.3 (6)	C7—C6—H6A	120.6
C5—N1—H1A	118.3	C7—C3—C4	118.1 (6)
C6—N1—H1A	118.3	C7—C3—C2	122.1 (6)
O1—C2—C1	122.6 (8)	C4—C3—C2	119.8 (6)
O1—C2—C3	117.9 (7)	C5—C4—C3	120.0 (6)
C1—C2—C3	119.5 (8)	C5—C4—H4A	120.0
C6—C7—C3	120.4 (6)	C3—C4—H4A	120.0
C6—C7—H7A	119.8	C2—C1—H1B	109.5
C3—C7—H7A	119.8	C2—C1—H1C	109.5
N1—C5—C4	119.3 (6)	H1B—C1—H1C	109.5
N1—C5—H5A	120.3	C2—C1—H1D	109.5
C4—C5—H5A	120.3	H1B—C1—H1D	109.5
N1—C6—C7	118.8 (6)	H1C—C1—H1D	109.5
N1—C6—H6A	120.6		
C6—N1—C5—C4	0.8 (11)	C1—C2—C3—C7	27.6 (10)
C5—N1—C6—C7	-1.3 (11)	O1—C2—C3—C4	27.8 (9)
C3—C7—C6—N1	0.2 (10)	C1—C2—C3—C4	-151.5 (7)
C6—C7—C3—C4	1.4 (10)	N1—C5—C4—C3	0.9 (10)
C6—C7—C3—C2	-177.8 (6)	C7—C3—C4—C5	-2.0 (9)
O1—C2—C3—C7	-153.0 (7)	C2—C3—C4—C5	177.2 (6)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots I1 ⁱ	0.86	2.67	3.456 (6)	153.

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

Fig. 1

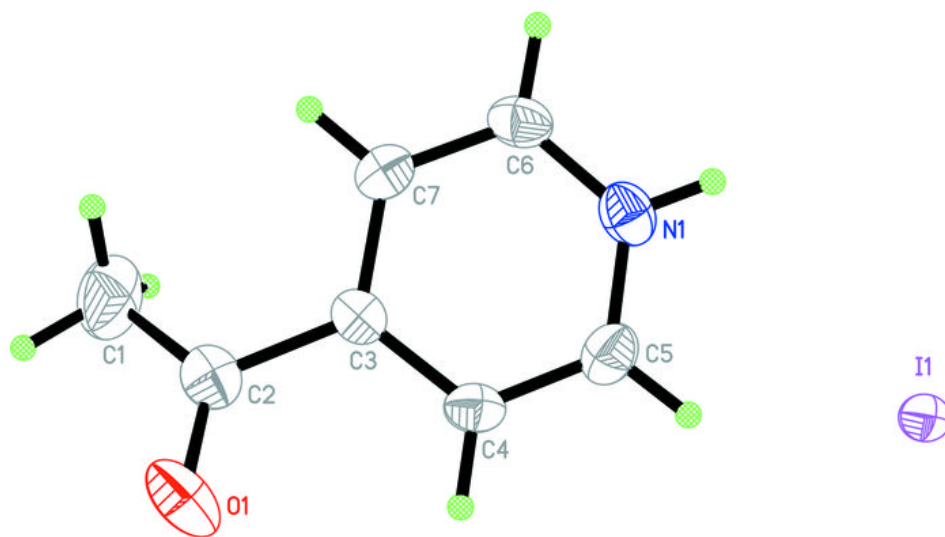


Fig. 2

