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4-(Acetylaminomethyl)-1-methylpyridinium iodide

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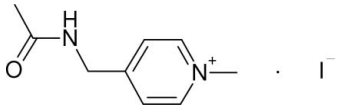
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.003 Å; R factor = 0.019; wR factor = 0.047; data-to-parameter ratio = 16.5.

In the title compound, $C_9H_{13}N_2O^+\cdot I^-$, the dihedral angle between the aromatic ring and the *N*-acetyl group is 73.93 (8)°. In the crystal structure, the cation and anion interact by way of an N—H···I hydrogen bond.

Related literature

For background, see: Javet *et al.* (2007). For reference structural data, see: Allen *et al.* (1995).



Experimental

Crystal data

$C_9H_{13}N_2O^+\cdot I^-$
M_r = 292.11
Monoclinic, $P2_1/c$
a = 8.597 (2) Å
b = 12.986 (3) Å
c = 10.335 (3) Å
β = 100.802 (5)°
V = 1133.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.79 mm^{-1}
T = 93 (2) K
0.10 × 0.10 × 0.10 mm

Data collection

Rigaku Mercury CCD diffractometer
6776 measured reflections
2035 independent reflections
1932 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.048
S = 1.04
2035 reflections
123 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···I1	0.79 (3)	2.81 (3)	3.603 (2)	174 (3)
C4—H4···O1^{i}	0.95	2.48	3.186 (3)	131

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank Lauren K. McDonald and M. John Plater for supplying the sample.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2511).

References

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supplementary materials

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Comment

The title compound, (I) (Fig. 1), is an intermediate in the synthesis of azacyanine dyes (Javet *et al.*, 2007) The dihedral angle between the C1—C5/N1 pyrininium ring and the C7/C8/N2/O1 grouping in (I) is 73.93 (8)°. Otherwise, the geometry of (I) may be regarded as normal (Allen *et al.*, 1995).

In the crystal of (I), an N—H···I hydrogen bond (Table 1) links the cation and the anion. A weak C—H···O interaction may also help to consolidate the packing, leading to [001] chains. There are no aromatic π-π stacking interactions in (I) as the closest centroid-centroid separation is greater than 4.37 Å.

Experimental

4-Aminomethylpyridine was aceylated with acetic anhydride in water, followed by methylation with CH_3I in CH_2Cl_2, yielding yellow cubic blocks of (I) after solvent removal.

Refinement

The N-bound hydrogen atom was located in a difference map and its position was freely refined with U_{iso}(H) = 1.2U_{eq}(N). The C-bound hydrogen atoms were geometrically placed (C—H = 0.95–0.99 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate, but not tip, to best fit the electron density.

Figures

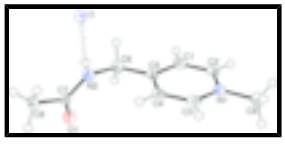

Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The hydrogen bond is indicated by a double-dashed line.

4-(Acetylaminomethyl)-1-methylpyridinium iodide

Crystal data

$C_9H_{13}N_2O^+\cdot I^-$	Z = 4
M_r = 292.11	F_{000} = 568
Monoclinic, $P2_1/c$	D_x = 1.712 Mg m^{-3}
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.597 (2) Å	μ = 2.79 mm^{-1}

b = 12.986 (3) Å	T = 93 (2) K
c = 10.335 (3) Å	Cube, yellow
β = 100.802 (5)º	0.10 × 0.10 × 0.10 mm
V = 1133.4 (5) Å^3	

Data collection

Rigaku Mercury CCD diffractometer	1932 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.023
Monochromator: graphite	θ_{max} = 25.3º
T = 93(2) K	θ_{min} = 2.4º
ω and φ scans	h = −9→10
Absorption correction: none	k = −15→14
6776 measured reflections	l = −11→12
2035 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difmap and geom
$R[F^2 > 2\sigma(F^2)]$ = 0.019	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.048	$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 0.8567P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.002
2035 reflections	$\Delta\rho_{max}$ = 0.82 e Å^{-3}
123 parameters	$\Delta\rho_{min}$ = −0.46 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1654 (3)	0.42190 (17)	0.5581 (2)	0.0205 (5)
H1	0.1710	0.3976	0.4724	0.025*

C2	0.0883 (3)	0.36517 (17)	0.6378 (2)	0.0196 (4)
H2A	0.0395	0.3020	0.6066	0.024*
C3	0.1502 (3)	0.48747 (17)	0.8068 (2)	0.0207 (5)
H3	0.1445	0.5094	0.8935	0.025*
C4	0.2286 (3)	0.54695 (17)	0.7301 (2)	0.0202 (5)
H4	0.2778	0.6093	0.7640	0.024*
C5	0.2361 (2)	0.51580 (16)	0.6022 (2)	0.0176 (4)
C6	0.3140 (3)	0.57832 (18)	0.5096 (2)	0.0226 (5)
H6A	0.3852	0.5326	0.4708	0.027*
H6B	0.2309	0.6033	0.4368	0.027*
C7	0.3626 (3)	0.76363 (17)	0.5334 (2)	0.0200 (5)
C8	0.4766 (3)	0.84538 (18)	0.5954 (3)	0.0278 (5)
H8A	0.5497	0.8161	0.6705	0.042*
H8B	0.4176	0.9023	0.6257	0.042*
H8C	0.5366	0.8711	0.5303	0.042*
C9	−0.0093 (3)	0.33746 (18)	0.8422 (2)	0.0255 (5)
H9A	−0.0177	0.2660	0.8113	0.038*
H9B	−0.1156	0.3668	0.8352	0.038*
H9C	0.0455	0.3394	0.9343	0.038*
N1	0.0809 (2)	0.39812 (12)	0.76054 (19)	0.0179 (4)
N2	0.4043 (2)	0.66579 (14)	0.5685 (2)	0.0218 (4)
H2	0.485 (3)	0.655 (2)	0.617 (3)	0.026*
O1	0.23978 (19)	0.78412 (13)	0.45526 (17)	0.0281 (4)
I1	0.753932 (15)	0.602150 (10)	0.806080 (13)	0.01921 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0250 (12)	0.0206 (10)	0.0147 (11)	0.0018 (9)	0.0008 (9)	−0.0032 (9)
C2	0.0237 (11)	0.0155 (10)	0.0178 (11)	0.0024 (9)	−0.0006 (9)	−0.0033 (9)
C3	0.0208 (11)	0.0238 (11)	0.0155 (11)	0.0028 (9)	−0.0016 (9)	−0.0045 (9)
C4	0.0203 (11)	0.0170 (10)	0.0212 (11)	0.0014 (8)	−0.0020 (9)	−0.0068 (9)
C5	0.0149 (10)	0.0173 (10)	0.0183 (11)	0.0051 (8)	−0.0031 (8)	0.0005 (8)
C6	0.0224 (12)	0.0222 (10)	0.0219 (12)	0.0002 (9)	0.0010 (10)	−0.0007 (9)
C7	0.0195 (11)	0.0221 (11)	0.0196 (11)	0.0011 (9)	0.0070 (9)	0.0024 (9)
C8	0.0269 (12)	0.0206 (11)	0.0360 (14)	−0.0033 (9)	0.0061 (11)	0.0019 (10)
C9	0.0291 (12)	0.0270 (12)	0.0194 (12)	−0.0006 (10)	0.0020 (10)	0.0044 (9)
N1	0.0176 (10)	0.0191 (9)	0.0150 (10)	0.0035 (7)	−0.0017 (8)	0.0010 (7)
N2	0.0178 (9)	0.0202 (9)	0.0243 (11)	0.0003 (7)	−0.0039 (8)	0.0027 (8)
O1	0.0246 (9)	0.0277 (9)	0.0300 (10)	0.0024 (7)	0.0002 (7)	0.0102 (7)
I1	0.01935 (11)	0.02071 (10)	0.01594 (11)	−0.00113 (5)	−0.00091 (7)	0.00050 (5)

Geometric parameters (Å, °)

C1—C2	1.365 (3)	C6—H6B	0.9900
C1—C5	1.400 (3)	C7—O1	1.232 (3)
C1—H1	0.9500	C7—N2	1.351 (3)
C2—N1	1.352 (3)	C7—C8	1.504 (3)
C2—H2A	0.9500	C8—H8A	0.9800

C3—N1	1.350 (3)	C8—H8B	0.9800
C3—C4	1.371 (3)	C8—H8C	0.9800
C3—H3	0.9500	C9—N1	1.476 (3)
C4—C5	1.395 (3)	C9—H9A	0.9800
C4—H4	0.9500	C9—H9B	0.9800
C5—C6	1.505 (3)	C9—H9C	0.9800
C6—N2	1.445 (3)	N2—H2	0.79 (3)
C6—H6A	0.9900		
C2—C1—C5	120.2 (2)	O1—C7—N2	122.1 (2)
C2—C1—H1	119.9	O1—C7—C8	122.4 (2)
C5—C1—H1	119.9	N2—C7—C8	115.5 (2)
N1—C2—C1	120.6 (2)	C7—C8—H8A	109.5
N1—C2—H2A	119.7	C7—C8—H8B	109.5
C1—C2—H2A	119.7	H8A—C8—H8B	109.5
N1—C3—C4	120.8 (2)	C7—C8—H8C	109.5
N1—C3—H3	119.6	H8A—C8—H8C	109.5
C4—C3—H3	119.6	H8B—C8—H8C	109.5
C3—C4—C5	120.0 (2)	N1—C9—H9A	109.5
C3—C4—H4	120.0	N1—C9—H9B	109.5
C5—C4—H4	120.0	H9A—C9—H9B	109.5
C4—C5—C1	117.8 (2)	N1—C9—H9C	109.5
C4—C5—C6	123.5 (2)	H9A—C9—H9C	109.5
C1—C5—C6	118.7 (2)	H9B—C9—H9C	109.5
N2—C6—C5	115.13 (19)	C3—N1—C2	120.60 (19)
N2—C6—H6A	108.5	C3—N1—C9	120.02 (19)
C5—C6—H6A	108.5	C2—N1—C9	119.34 (18)
N2—C6—H6B	108.5	C7—N2—C6	122.1 (2)
C5—C6—H6B	108.5	C7—N2—H2	120 (2)
H6A—C6—H6B	107.5	C6—N2—H2	118 (2)
C5—C1—C2—N1	−0.8 (3)	C4—C3—N1—C2	0.4 (3)
N1—C3—C4—C5	0.7 (3)	C4—C3—N1—C9	−177.2 (2)
C3—C4—C5—C1	−1.7 (3)	C1—C2—N1—C3	−0.3 (3)
C3—C4—C5—C6	177.5 (2)	C1—C2—N1—C9	177.3 (2)
C2—C1—C5—C4	1.8 (3)	O1—C7—N2—C6	4.6 (3)
C2—C1—C5—C6	−177.4 (2)	C8—C7—N2—C6	−174.9 (2)
C4—C5—C6—N2	9.8 (3)	C5—C6—N2—C7	−115.1 (2)
C1—C5—C6—N2	−171.05 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···I1	0.79 (3)	2.81 (3)	3.603 (2)	174 (3)
C4—H4···O1^{i}	0.95	2.48	3.186 (3)	131

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2.

Fig. 1