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4-Amino-(1-ethoxycarbonylmethyl)pyridinium iodide

T. Seethalakshmi,[a] P. Venkatesan,[b] F. R. Fronczek,[c] P. Kaliannan[a] and S. Thamotharan[d]*

[a]School of Physics, Bharathidasan University, Tiruchirappalli 620 024, India, [b]School of Chemistry, Bharathidasan University, Tiruchirappalli 620 024, India, [c]Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA, and [d]Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012, India

Correspondence e-mail: thamu_as@yahoo.com

Key indicators

Single-crystal X-ray study
T = 115 K
Mean σ(C–C) = 0.002 Å
R factor = 0.028
wR factor = 0.068
Data-to-parameter ratio = 41.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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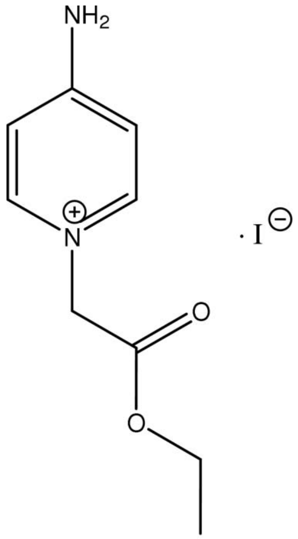
The crystal structure of the title compound, $C_9H_{13}N_2O_2^+\cdot I^-$, consists of pyridinium cations and iodide anions stabilized by intermolecular N—H⋯I hydrogen bonds, forming one-dimensional chains along [010].

Comment

Pyridinium derivatives often possess antibacterial and antifungal activities (Seethalakshmi *et al.*, 2006, and references therein). In continuation of our study of pyridinium derivatives, the crystal structure analysis of the title compound, (I), has been undertaken.



(I)

The structure of the asymmetric unit of (I), consisting of a pyridinium cation and an iodide anion, is shown in Fig. 1. The bond lengths and angles within the pyridinium ring are normal and comparable with those reported for related structures (Seethalakshmi *et al.*, 2006; Sundar *et al.*, 2004*a*,*b*, 2005; Sundar *et al.*, 2006, 2006*a*,*b*). The N1—C6—C7—O1, C6—C7—O1—C8 and C7—O1—C8—C9 torsion angles in (I) (Table 1) indicate that the ethoxycarbonylmethyl group is in an extended conformation. Atoms N1/C6/C7/O1/C8/C9 form an approximate plane with a maximum deviation of 0.193 (2) Å for C6; the dihedral angle between this plane and the pyridinium ring is 66.1 (1)°.

In the crystal structure of (I), neighbouring pyridinium cations are interconnected by iodide anions through intermolecular N—H⋯I hydrogen bonds (Fig. 2 and Table 2), leading to a one-dimensional chain along [010]. In addition to these interactions, a weak intermolecular C—H⋯I inter-

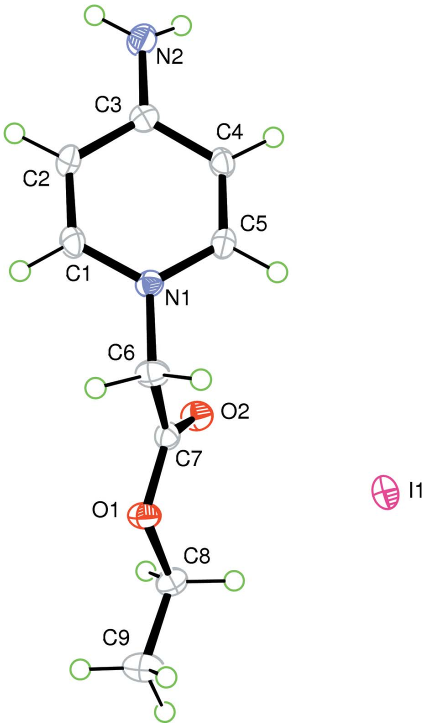


Figure 1
The asymmetric unit of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are represented by circles of arbitrary radii.

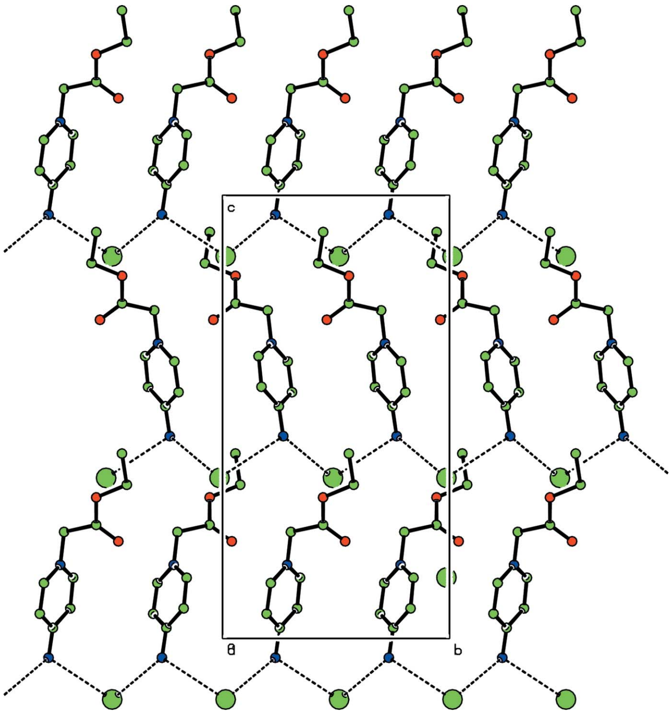


Figure 2
Part of the crystal structure of (I), viewed along the *a* axis. The intermolecular non-bonded N···I distances of the N—H···I hydrogen bonds are indicated by dashed lines. H atoms have been omitted.

action also is observed, involving the H atom bonded to C1 and I1 [C1···I1 = 3.773 (2) Å, H1···I1 = 2.92 Å and C1—H1···I1 = 150°].

Experimental

A solution of 4-aminopyridine (1 mol, 25 ml) and ethyl α-iodoacetate (1 mol, 25 ml) in acetone was stirred at room temperature (303 K) for 1–2 h. The solid that separated was filtered, washed with dry acetone and dried in vacuum to give the stable salt, (I), which recrystallized from an aqueous ethanol (80% *v*/*v*) solution (m.p. 449–451 K).

Crystal data

$C_9H_{13}N_2O_2^+\cdot I^-$
$M_r = 308.11$
Orthorhombic, *Pbcn*
$a = 12.5402$ (15) Å
$b = 9.8173$ (10) Å
$c = 19.135$ (2) Å
$V = 2355.7$ (4) Å^3
$Z = 8$
$D_x = 1.738$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.70$ mm^{-1}
$T = 115$ (2) K
Fragment, colorless
0.25 × 0.22 × 0.20 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
ω scans with κ offsets
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.524$, $T_{\max} = 0.583$
36052 measured reflections
5535 independent reflections
4300 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 36.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.068$
$S = 1.06$
5535 reflections
135 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0272P)^2 + 1.6703P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.96$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00249 (13)

Table 1
Selected torsion angles (°).

C8—O1—C7—C6	167.70 (14)	C7—O1—C8—C9	−177.72 (16)
N1—C6—C7—O1	161.57 (14)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H21···I1	0.85 (3)	2.79 (3)	3.6384 (17)	173 (2)
N2—H22···I1$^{\rm i}$	0.79 (3)	2.91 (2)	3.6527 (17)	159 (2)

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

The amino H atoms were located in a difference Fourier map and refined with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$. The methyl H atoms were constrained to an ideal geometry (C—H = 0.98 Å), with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$, but were allowed to rotate freely about the C—C bond. The remaining H atoms were placed in geometrically idealized positions (C—H = 0.95–0.99 Å) and were constrained to ride on their parent atoms with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The highest residual density peak is 0.68 Å from I1 and the deepest hole is 0.67 Å from I1.

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Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (version 1.07; Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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