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1-(2-Carboxyethyl)-5-ethyl-2-methylpyridinium chloride

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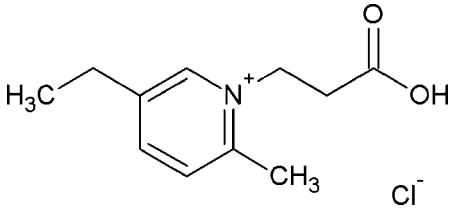
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.102; data-to-parameter ratio = 19.5.

In the crystal structure of the title salt, $C_{11}H_{16}NO_2^+\cdot Cl^-$, the cations and anions are linked by O—H···Cl hydrogen bonds. The structure is further stabilized by weak C—H···Cl hydrogen bonds.

Related literature

For the biological activity of 4-aminopyridine, see: Judge & Bever (2006); Schwid *et al.* (1997); Strupp *et al.* (2004). For related structures, see: Anderson *et al.* (2005); Fun *et al.* (2009).



Experimental

Crystal data

$C_{11}H_{16}NO_2^+\cdot Cl^-$
M_r = 229.70
Triclinic, $P\overline{1}$
a = 7.5013 (4) Å
b = 9.0509 (5) Å
c = 9.3452 (5) Å
α = 75.253 (2)°
β = 80.985 (2)°
γ = 72.047 (2)°
V = 581.59 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 293 K
0.32 × 0.20 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.972, T_{max} = 0.992
11668 measured reflections
2772 independent reflections
2363 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.102
S = 0.83
2772 reflections
142 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···Cl1	0.92 (3)	2.06 (3)	2.9749 (12)	170 (2)
C2—H2···Cl1^{i}	0.93	2.72	3.6249 (14)	166
C6—H6A···Cl1^{ii}	0.97	2.68	3.6261 (14)	166
C11—H11A···Cl1^{iii}	0.96	2.79	3.7410 (16)	170

Symmetry codes: (i) $x-1, y, z+1$; (ii) $x, y, z+1$; (iii) $-x+2, -y, -z+1$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6827).

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supplementary materials

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1-(2-Carboxyethyl)-5-ethyl-2-methylpyridinium chloride

V. Sabari, G. Kalaiselvi, S. Balasubramanian and S. Aravindhan

Comment

4-Aminopyridine (Fampridine) is used clinically in Lambert-Eaton myasthenic syndrome and multiple sclerosis because by blocking potassium channels it prolongs action potentials thereby increasing transmitter release at the neuromuscular junction (Judge & Bever *et al.*, 2006; Schwid *et al.*, 1997; Strupp *et al.*, 2004).

In the title compound (Fig. 1), the bond lengths and angles have normal values. The asymmetric unit is composed of one 1-(2-carboxy ethyl) 5-ethyl 2-methyl pyridinium cation and one Cl^- anion. The C1—N1—C5 angle in the pyridinium ring is widened to 121.20 (15) °, compared to 115.25 (13)° in 4-aminopyridine (Anderson *et al.*, 2005) and 120.7 (2)°, in Aminopyridinium (Fun *et al.*, 2009). In the crystal structure, anions and cations are connected by O—H···Cl and C—H···Cl hydrogen bonds.

Experimental

1g (8.3mmol) of freshly distilled 5-ethyl 2-methyl pyridine was dissolved in 10 ml of THF at -10°C under nitrogen atmosphere. To the above solution, 0.8 ml (8.0 mmol) of acrylic acid in 10 ml of THF was added drop wise with continuous stirring. After stirring for 20 min in an ice bath, 0.5 mL of HCl was added and stirred for 24 h. White solid formed after the completion of the reaction and the solid was filtered, washed with THF and dried in vacuum. The product was recrystallized from methanol Yield: 1.52g (80%)

Refinement

All H atoms on carbons were positioned geometrically with C—H distances ranging from 0.95 to 1.00 Å and refined as riding on their parent atoms, with U_{iso}(H) = 1.2Ueq(C) or U_{iso}(H) = 1.5Ueq(C_{methyl}) The hydroxyl H atom was freely refined.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

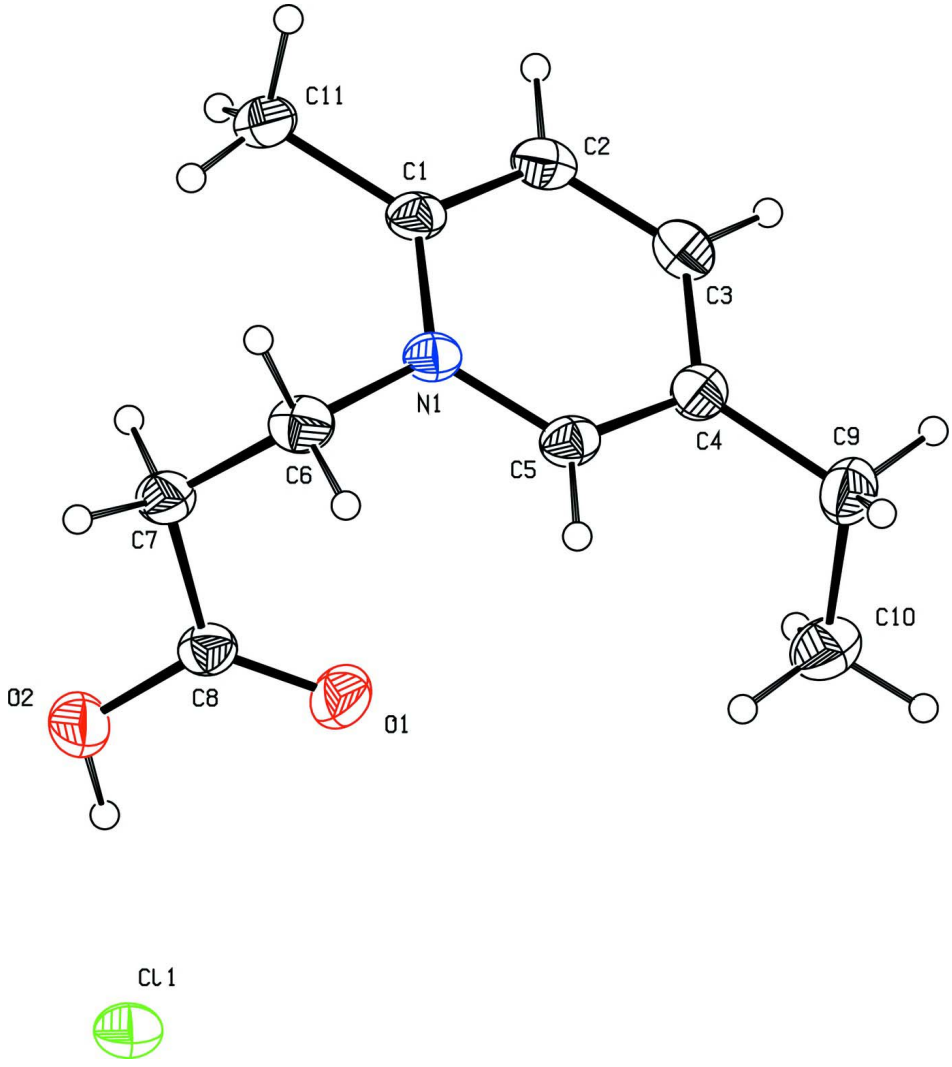

Figure 1

View of one molecule of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level (arbitrary spheres for the H atoms).

1-(2-Carboxyethyl)-5-ethyl-2-methylpyridinium chloride

Crystal data

$C_{11}H_{16}NO_2^+ \cdot Cl^-$
$M_r = 229.70$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.5013\ (4)$ Å
$b = 9.0509\ (5)$ Å
$c = 9.3452\ (5)$ Å
$\alpha = 75.253\ (2)°$
$\beta = 80.985\ (2)°$
$\gamma = 72.047\ (2)°$
$V = 581.59\ (5)$ Å^3
$Z = 2$
$F(000) = 244$
$D_x = 1.312$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5710 reflections
$\theta = 1.8–28.5°$
$\mu = 0.31$ mm^{-1}
$T = 293$ K
Triclinic, colourless
0.32 × 0.20 × 0.10 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.972, T_{max} = 0.992

11668 measured reflections
2772 independent reflections
2363 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.9°, θ_{min} = 2.3°
h = −9→9
k = −11→11
l = −10→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.102
S = 0.83
2772 reflections
142 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.2215P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.034 (6)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	1.11724 (18)	0.12433 (14)	0.49990 (13)	0.0607 (3)
Cl1	1.13273 (5)	0.19233 (4)	0.17024 (4)	0.04786 (14)
N1	0.73265 (15)	0.39471 (13)	0.81065 (11)	0.0344 (2)
C1	0.60912 (19)	0.31985 (15)	0.89780 (13)	0.0373 (3)
C8	1.01152 (18)	0.23770 (16)	0.56489 (14)	0.0388 (3)
C5	0.67204 (19)	0.53934 (15)	0.72045 (13)	0.0374 (3)
H5	0.7607	0.5877	0.6639	0.045*
O1	0.92281 (18)	0.36464 (13)	0.49974 (11)	0.0570 (3)
C4	0.4848 (2)	0.61779 (16)	0.70897 (14)	0.0390 (3)
C11	0.6787 (2)	0.16261 (18)	1.00116 (16)	0.0503 (4)
H11A	0.7271	0.0810	0.9453	0.075*
H11B	0.5770	0.1396	1.0707	0.075*
H11C	0.7768	0.1662	1.0539	0.075*
C3	0.3566 (2)	0.54196 (18)	0.79512 (16)	0.0441 (3)
H3	0.2282	0.5900	0.7897	0.053*

C2	0.4195 (2)	0.39556 (17)	0.88856 (15)	0.0430 (3)
H2	0.3323	0.3465	0.9468	0.052*
C6	0.93976 (19)	0.32296 (19)	0.81201 (15)	0.0436 (3)
H6A	0.9709	0.2834	0.9143	0.052*
H6B	1.0014	0.4055	0.7671	0.052*
C7	1.0172 (2)	0.18822 (18)	0.73100 (15)	0.0448 (3)
H7B	1.1466	0.1356	0.7534	0.054*
H7A	0.9463	0.1110	0.7692	0.054*
C9	0.4262 (2)	0.77820 (17)	0.60617 (17)	0.0510 (4)
H9B	0.4899	0.8477	0.6273	0.061*
H9A	0.2920	0.8242	0.6246	0.061*
C10	0.4713 (3)	0.7697 (2)	0.44375 (17)	0.0566 (4)
H10A	0.4327	0.8749	0.3825	0.085*
H10C	0.4054	0.7038	0.4214	0.085*
H10B	0.6042	0.7252	0.4247	0.085*
H2A	1.108 (3)	0.153 (3)	0.399 (3)	0.099 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0687 (7)	0.0570 (7)	0.0404 (6)	0.0055 (6)	−0.0043 (5)	−0.0115 (5)
Cl1	0.0537 (2)	0.0563 (2)	0.03510 (19)	−0.01908 (17)	0.00042 (14)	−0.01085 (14)
N1	0.0393 (5)	0.0394 (6)	0.0274 (5)	−0.0143 (4)	−0.0016 (4)	−0.0091 (4)
C1	0.0487 (7)	0.0390 (7)	0.0276 (5)	−0.0171 (6)	0.0032 (5)	−0.0114 (5)
C8	0.0357 (6)	0.0436 (7)	0.0364 (6)	−0.0143 (5)	−0.0016 (5)	−0.0048 (5)
C5	0.0470 (7)	0.0387 (7)	0.0306 (6)	−0.0185 (5)	−0.0004 (5)	−0.0088 (5)
O1	0.0768 (8)	0.0457 (6)	0.0376 (5)	−0.0042 (5)	−0.0076 (5)	−0.0044 (4)
C4	0.0499 (7)	0.0368 (6)	0.0329 (6)	−0.0116 (5)	−0.0040 (5)	−0.0124 (5)
C11	0.0660 (9)	0.0424 (8)	0.0364 (7)	−0.0154 (7)	0.0070 (6)	−0.0044 (6)
C3	0.0407 (7)	0.0496 (8)	0.0443 (7)	−0.0111 (6)	−0.0002 (6)	−0.0185 (6)
C2	0.0454 (7)	0.0486 (8)	0.0395 (7)	−0.0211 (6)	0.0081 (6)	−0.0148 (6)
C6	0.0391 (7)	0.0578 (8)	0.0350 (6)	−0.0148 (6)	−0.0088 (5)	−0.0070 (6)
C7	0.0382 (7)	0.0514 (8)	0.0350 (6)	−0.0061 (6)	−0.0022 (5)	−0.0010 (6)
C9	0.0661 (10)	0.0376 (7)	0.0460 (8)	−0.0078 (7)	−0.0099 (7)	−0.0087 (6)
C10	0.0693 (10)	0.0565 (9)	0.0428 (8)	−0.0199 (8)	−0.0084 (7)	−0.0039 (7)

Geometric parameters (Å, º)

O2—C8	1.3097 (18)	C11—H11C	0.9600
O2—H2A	0.92 (3)	C3—C2	1.377 (2)
N1—C5	1.3499 (17)	C3—H3	0.9300
N1—C1	1.3604 (16)	C2—H2	0.9300
N1—C6	1.4885 (17)	C6—C7	1.514 (2)
C1—C2	1.381 (2)	C6—H6A	0.9700
C1—C11	1.4938 (19)	C6—H6B	0.9700
C8—O1	1.1971 (17)	C7—H7B	0.9700
C8—C7	1.5058 (19)	C7—H7A	0.9700
C5—C4	1.372 (2)	C9—C10	1.519 (2)
C5—H5	0.9300	C9—H9B	0.9700
C4—C3	1.388 (2)	C9—H9A	0.9700

C4—C9	1.5009 (19)	C10—H10A	0.9600
C11—H11A	0.9600	C10—H10C	0.9600
C11—H11B	0.9600	C10—H10B	0.9600
C8—O2—H2A	110.8 (16)	C3—C2—H2	119.3
C5—N1—C1	121.15 (11)	C1—C2—H2	119.3
C5—N1—C6	116.99 (11)	N1—C6—C7	114.17 (11)
C1—N1—C6	121.85 (11)	N1—C6—H6A	108.7
N1—C1—C2	117.68 (12)	C7—C6—H6A	108.7
N1—C1—C11	120.43 (13)	N1—C6—H6B	108.7
C2—C1—C11	121.88 (12)	C7—C6—H6B	108.7
O1—C8—O2	123.99 (13)	H6A—C6—H6B	107.6
O1—C8—C7	124.60 (13)	C8—C7—C6	114.89 (12)
O2—C8—C7	111.41 (12)	C8—C7—H7B	108.5
N1—C5—C4	122.58 (12)	C6—C7—H7B	108.5
N1—C5—H5	118.7	C8—C7—H7A	108.5
C4—C5—H5	118.7	C6—C7—H7A	108.5
C5—C4—C3	117.08 (13)	H7B—C7—H7A	107.5
C5—C4—C9	120.07 (13)	C4—C9—C10	112.48 (12)
C3—C4—C9	122.85 (14)	C4—C9—H9B	109.1
C1—C11—H11A	109.5	C10—C9—H9B	109.1
C1—C11—H11B	109.5	C4—C9—H9A	109.1
H11A—C11—H11B	109.5	C10—C9—H9A	109.1
C1—C11—H11C	109.5	H9B—C9—H9A	107.8
H11A—C11—H11C	109.5	C9—C10—H10A	109.5
H11B—C11—H11C	109.5	C9—C10—H10C	109.5
C2—C3—C4	119.99 (13)	H10A—C10—H10C	109.5
C2—C3—H3	120.0	C9—C10—H10B	109.5
C4—C3—H3	120.0	H10A—C10—H10B	109.5
C3—C2—C1	121.50 (12)	H10C—C10—H10B	109.5
C5—N1—C1—C2	1.26 (17)	C4—C3—C2—C1	−0.9 (2)
C6—N1—C1—C2	−179.81 (11)	N1—C1—C2—C3	−0.25 (19)
C5—N1—C1—C11	−177.69 (11)	C11—C1—C2—C3	178.69 (13)
C6—N1—C1—C11	1.24 (18)	C5—N1—C6—C7	−105.73 (14)
C1—N1—C5—C4	−1.18 (18)	C1—N1—C6—C7	75.30 (15)
C6—N1—C5—C4	179.84 (11)	O1—C8—C7—C6	−14.0 (2)
N1—C5—C4—C3	0.01 (19)	O2—C8—C7—C6	165.58 (13)
N1—C5—C4—C9	−179.76 (11)	N1—C6—C7—C8	69.15 (16)
C5—C4—C3—C2	0.99 (19)	C5—C4—C9—C10	68.80 (18)
C9—C4—C3—C2	−179.24 (13)	C3—C4—C9—C10	−110.95 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1^{i}	0.93	2.72	3.6249 (14)	166
C6—H6*A*···Cl1^{ii}	0.97	2.68	3.6261 (14)	166

O2—H2*A*···Cl1	0.92 (3)	2.06 (3)	2.9749 (12)	170 (2)
C11—H11*A*···Cl1^{iii}	0.96	2.79	3.7410 (16)	170

Symmetry codes: (i) $x-1, y, z+1$; (ii) $x, y, z+1$; (iii) $-x+2, -y, -z+1$.