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3-Methoxy-3-oxopropanaminium chloride

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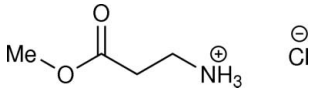
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.080; data-to-parameter ratio = 21.4.

In the title compound, $C_4H_{10}NO_2^+\cdot Cl^-$, the central ethylene bond of the cation adopts a *gauche* conformation. The three H atoms of the $-NH_3^+$ group are engaged in strong and highly directional intermolecular N—H···Cl hydrogen bonds, which result in a tape-like arrangement along [010] of the respective ion pairs. In addition, weak intermolecular C—H···Cl and C—H···O interactions are present.

Related literature

For the synthesis of the title compound, see: Hansen (1963). For related structures, see: Akkerman *et al.* (2003); Robinson *et al.* (2004); Vilela *et al.* (2009); Tarafdar & Swamy (2010); Gossage *et al.* (2010); He *et al.* (2010). For information on the *gauche* effect, see: Amos *et al.* (1992). For details of the H-atom treatment, see: Cooper *et al.* (2010). For the weighting scheme used in the refinement, see: Watkin (1994); Prince (1982).



Experimental

Crystal data

$C_4H_{10}NO_2^+\cdot Cl^-$
M_r = 139.58
Monoclinic, $P2_1/c$
a = 9.8469 (2) Å
b = 5.3263 (1) Å
c = 13.2804 (2) Å
β = 99.4638 (10)°
V = 687.04 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 150 K
0.28 × 0.13 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan *DENZO/SCALEPACK* (Otwinowski & Minor, 1997)
T_{min} = 0.94, T_{max} = 0.96
14336 measured reflections
1563 independent reflections
1413 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.080
S = 0.93
1563 reflections
73 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N8—H81···Cl1^{i}	0.90	2.26	3.1456 (12)	171 (1)
N8—H82···Cl1	0.92	2.29	3.1910 (12)	171 (1)
N8—H83···Cl1^{ii}	0.90	2.35	3.1923 (12)	157 (1)
C5—H53···O4^{iii}	0.96	2.67	3.5965 (18)	163 (1)
C7—H72···Cl1^{iv}	0.96	2.84	3.4708 (14)	124 (1)

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, -y+2, -z+1$; (iv) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 2001).; cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5384).

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supplementary materials

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Comment

The asymmetic unit of the title compound, (**I**), consists of a 2-acetoxy-ethyl-ammonium cation and a chloride ion as shown in Figure 1. The ester motif [atoms C6/O2/C3/O4/C5] is approximately planar with the largest deviation from the mean plane for O2 (d = 0.029 Å). The central —CH_2—CH_2— unit is not in the often favoured antiperiplanar conformation, instead adopting a *gauche* conformation with a torsion angle of 57.42 (14)° for atoms O2—C6—C7—N8. This may be attributed to the stereoselective *gauche* effect (Amos *et al.*, 1992), though an influence of the crystal packing on the molecular conformation of (**I**) cannot be ruled out. For comparison, the observed torsion angle is 67.6° in 1,2-difluoroethane (Akkerman *et al.*, 2003), 73.7° for *O*-stearoylethanolamine hydrochloride (Tarafdar & Swamy, 2010) and 71.7° in 2-(benzoyloxy)ethanaminium nitrate (Gossage *et al.*, 2010).

The three N—H units of (**I**) are engaged in apparently strong and highly directional N^+—H···Cl^- hydrogen bonds with three symmetry-related Cl^- ions (Table 1). These interactions result in a tape-like arrangement of the respective ion pairs parallel to the crystallographic *b* axis (Figure 2). In the packing, the corrugated two dimensional supramolecular network defined by the N—H···Cl interactions is connected with neighbouring strands *via* weak C—H···Cl and C—H···O contacts (Table 1) in the direction of the crystallographic *c* and *a* axes, respectively. Interestingly, the observed packing behaviour is very similar to the structure of glycine ethyl ester hydrochloride (He *et al.*, 2010), an isomer of (**I**), and the analogous glycine methyl ester (Vilela *et al.*, 2009).

Experimental

The title compound was prepared from 2-aminoethanol and acetyl chloride according to the literature (Hansen, 1963). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of (**I**) in chloroform.

Refinement

The structure was refined freely, except for the hydrogen atoms which were refined prior to the generation of the riding model (Cooper *et al.*, 2010). Weights were applied using a five parameter Chebychev polynomial (Watkin, 1994, Prince, 1982).

Dihedral angles calculated with *PLATON* (Spek, 2009); all other standard uncertainties calculated from the full variance co-variance matrix within *CRYSTALS* (Betteridge *et al.*, 2003).

Computing details

Data collection: *COLLECT* (Nonius, 2001).; cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003) and *PLATON* (Spek, 2009).

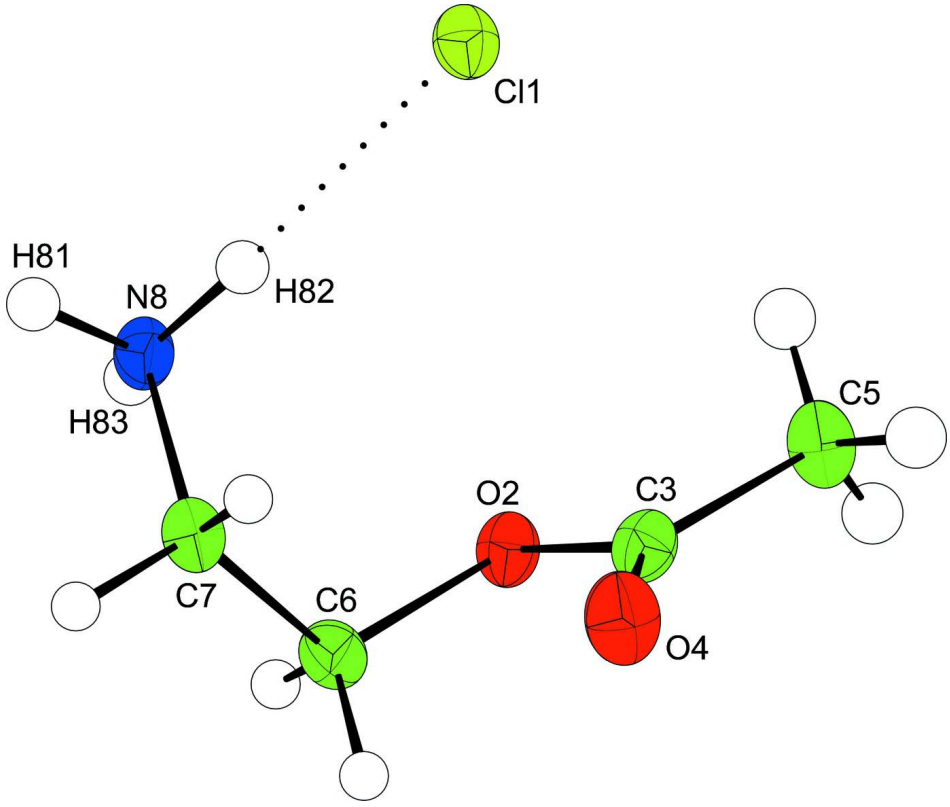

Figure 1

Molecular structure of (**I**) with displacement ellipsoids drawn at 50% probability. The dotted line indicates a hydrogen bond.

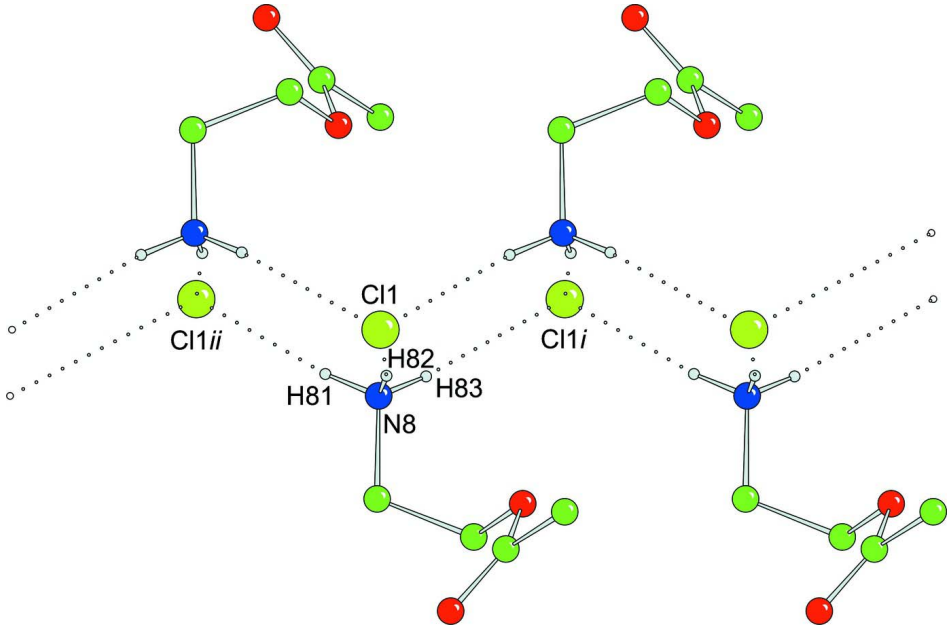

Figure 2

The corrugated two dimensional supramolecular network defined by the N—H···Cl interactions forming tapes [*i*: 2 - *x*,1/2 + *y*,3/2 - *z*; *ii*: 2 - *x*,-1/2 + *y*,3/2 - *z*].

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Crystal data

$C_4H_{10}NO_2^+ \cdot Cl^-$
$M_r = 139.58$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.8469$ (2) Å
$b = 5.3263$ (1) Å
$c = 13.2804$ (2) Å
$\beta = 99.4638$ (10)°
$V = 687.04$ (2) Å^3
$Z = 4$
$F(000) = 296$
$D_x = 1.349$ Mg m^{-3}
Melting point: not measured K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1729 reflections
$\theta = 5$–27°
$\mu = 0.47$ mm^{-1}
$T = 150$ K
Block, clear_pale_colourless
0.28 × 0.13 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997)
$T_{min} = 0.94$, $T_{max} = 0.96$
14336 measured reflections
1563 independent reflections
1413 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
$h = -12 \rightarrow 12$
$k = -6 \rightarrow 6$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.080$
$S = 0.93$
1563 reflections
73 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H-atom parameters constrained
Method, part 1, Chebychev polynomial, (Watkin, 1994; *P*rince, 1982) [*w*eight] = $1.0/[A_0*T_0(x) + A_1*T_1(x) \cdots + A_{n-1}]*T_{n-1}(x)]$ where A_i are the Chebychev coefficients listed below and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 37.6 62.5 38.0 16.9 4.31
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.92411 (3)	0.72604 (6)	0.59816 (2)	0.0244
O2	0.72167 (10)	1.11170 (19)	0.75243 (7)	0.0236
C3	0.62577 (14)	1.0662 (3)	0.66935 (11)	0.0241
O4	0.53381 (11)	0.9165 (2)	0.66871 (8)	0.0335
C5	0.64993 (16)	1.2257 (3)	0.58178 (12)	0.0307
C6	0.70489 (15)	0.9789 (3)	0.84455 (10)	0.0257
C7	0.76188 (14)	0.7166 (3)	0.84666 (10)	0.0240
N8	0.91018 (12)	0.7208 (2)	0.83652 (9)	0.0235
H51	0.7403	1.1944	0.5697	0.0451*
H52	0.6422	1.3972	0.6003	0.0448*
H53	0.5842	1.1883	0.5224	0.0448*
H61	0.7569	1.0756	0.8995	0.0291*

H62	0.6067	0.9749	0.8523	0.0288*
H71	0.7136	0.6156	0.7909	0.0287*
H72	0.7551	0.6399	0.9112	0.0286*
H81	0.9506	0.5763	0.8594	0.0342*
H82	0.9161	0.7420	0.7690	0.0341*
H83	0.9517	0.8494	0.8726	0.0346*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02820 (19)	0.02258 (18)	0.02136 (18)	−0.00083 (12)	0.00108 (12)	−0.00105 (12)
O2	0.0248 (5)	0.0223 (5)	0.0222 (5)	−0.0002 (4)	−0.0009 (4)	0.0002 (4)
C3	0.0238 (6)	0.0228 (6)	0.0243 (6)	0.0016 (5)	−0.0005 (5)	−0.0012 (5)
O4	0.0303 (5)	0.0348 (6)	0.0329 (6)	−0.0080 (5)	−0.0025 (4)	0.0039 (5)
C5	0.0332 (8)	0.0305 (8)	0.0268 (7)	−0.0027 (6)	0.0000 (6)	0.0047 (6)
C6	0.0292 (7)	0.0278 (7)	0.0200 (6)	0.0021 (6)	0.0041 (5)	−0.0010 (5)
C7	0.0268 (7)	0.0229 (6)	0.0215 (6)	−0.0016 (5)	0.0017 (5)	0.0021 (5)
N8	0.0287 (6)	0.0202 (5)	0.0208 (5)	0.0032 (4)	0.0015 (4)	0.0012 (4)

Geometric parameters (Å, º)

O2—C3	1.3509 (16)	C6—H61	0.968
O2—C6	1.4459 (17)	C6—H62	0.989
C3—O4	1.2057 (18)	C7—N8	1.4886 (18)
C3—C5	1.490 (2)	C7—H71	0.973
C5—H51	0.945	C7—H72	0.961
C5—H52	0.952	N8—H81	0.896
C5—H53	0.955	N8—H82	0.915
C6—C7	1.504 (2)	N8—H83	0.895
C3—O2—C6	116.20 (11)	C7—C6—H62	110.2
O2—C3—O4	123.21 (13)	H61—C6—H62	109.8
O2—C3—C5	110.81 (12)	C6—C7—N8	110.65 (11)
O4—C3—C5	125.98 (13)	C6—C7—H71	111.4
C3—C5—H51	107.9	N8—C7—H71	107.7
C3—C5—H52	108.4	C6—C7—H72	109.3
H51—C5—H52	109.3	N8—C7—H72	107.5
C3—C5—H53	110.6	H71—C7—H72	110.2
H51—C5—H53	110.7	C7—N8—H81	110.1
H52—C5—H53	109.9	C7—N8—H82	108.2
O2—C6—C7	112.08 (11)	H81—N8—H82	110.0
O2—C6—H61	104.9	C7—N8—H83	109.3
C7—C6—H61	109.3	H81—N8—H83	109.7
O2—C6—H62	110.3	H82—N8—H83	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H81···Cl1^{i}	0.90	2.26	3.1456 (12)	171 (1)
N8—H82···Cl1	0.92	2.29	3.1910 (12)	171 (1)

N8—H83···Cl1^{ii}	0.90	2.35	3.1923 (12)	157 (1)
C5—H53···O4^{iii}	0.96	2.67	3.5965 (18)	163 (1)
C7—H72···Cl1^{iv}	0.96	2.84	3.4708 (14)	124 (1)

Symmetry codes: (i) $-x+2, y-1/2, -z+3/2$; (ii) $-x+2, y+1/2, -z+3/2$; (iii) $-x+1, -y+2, -z+1$; (iv) $x, -y+3/2, z+1/2$.