Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Glycine ethyl ester hydrochloride

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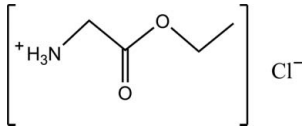
Received 12 May 2010; accepted 20 July 2010

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.027; wR factor = 0.064; data-to-parameter ratio = 17.1.

In the crystal structure of the title compound, $C_4H_{10}NO_2^+\cdot Cl^-$ (systematic name: 3-ethoxy-3-oxopropan-1-aminium chloride), there are strong intermolecular N—H···Cl, C—H···Cl and C—H···O hydrogen-bonding interactions between the free chloride anion and the organic cation, resulting in a two-dimensional supramolecular network in the *ab* plane.

Related literature

The title compound is an intermediate in the synthesis of dichlorovinylcyclopropane carboxylic acid, see: Xue (1995). For related structures, see: Taubald *et al.* (1984); Gainsford *et al.* (1986); Eduok *et al.* (1994).



Experimental

Crystal data

$C_4H_{10}NO_2^+\cdot Cl^-$
M_r = 139.58
Monoclinic, $P2_1/c$
a = 8.965 (3) Å
b = 12.543 (4) Å
c = 5.972 (2) Å
β = 103.630 (5)°
V = 652.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.50 mm^{-1}
T = 123 K
0.33 × 0.33 × 0.23 mm

Data collection

Rigaku SPIDER diffractometer
4996 measured reflections
1489 independent reflections
1294 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.064
S = 1.00
1489 reflections
87 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H0A···Cl1	0.904 (17)	2.300 (17)	3.1845 (16)	166.1 (12)
N1—H0B···Cl1^{i}	0.906 (18)	2.386 (18)	3.1658 (16)	144.3 (15)
N1—H0C···Cl1	0.890 (19)	2.435 (19)	3.2566 (16)	153.7 (15)
C1—H1A···O2	0.99	2.47	2.9072 (18)	106
C3—H3B···Cl1^{ii}	0.99	2.79	3.7529 (18)	164

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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: *SHELXTL*.

This work was supported by the Science Foundation of the Health Department of Jiangsu Province (No. H200934).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2143).

References

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supporting information

Acta Cryst. (2010). E**66**, o2115 [https://doi.org/10.1107/S1600536810028849]

Glycine ethyl ester hydrochloride

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S1. Comment

The title compound, glycine ethyl ester hydrochloride is used in the preparation of dichlorovinylcyclopropane carboxylic acid, an important pesticide intermediate (Xue,1995).It is also used in the preparation of function material, the crystal structures of dichloro-bis(glycine ethyl ester)-palladium(II) (Taubald, *et al.*, 1984),*p,p*-(μ_2-peroxo) -bis(tris(2-amino-ethyl)-amine-*N*,*N*′,*N*″,*N*‴)-bis(ethylglycinate-N)-cobalt(II) tetraperchlorate (Gainsford *et al.*, 1986),cis-β_2-((s,s)-chloro-(glycine ethyl ester-*N*)-(triethylenetetramine)-cobalt(III) dichloride trihydrate (Eduok *et al.*, 1994) have been reported. The molecular structure of(I) is shown in Fig.1. The three crystallographically independent N—H moieties are engaged in highly directional N^+—H···Cl^- hydrogen bonds with three symmetry-related Cl^- anions. These interactions promote the formation of a tape of $C_4H_{10}NO_2^+$.Cl^- moieties running parallel to the c axis.

S2. Experimental

Glycine ethyl ester hydrochloride (0.1 mmol, Sigma Aldrich at 99% purity) was dissolved methanol (20 ml) and gently heated under reflux for 1 h. After cooling the solution to ambient temperature, crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of the solvent after few days.

S3. Refinement

Hydrogen atoms bound to nitrogen and carbon were located at their idealized positions and were included in the final structural model in riding-motion approximation with C—H = 0.98Å and N—H = 0.90 Å. The isotropic thermal displacement parameters for these atoms were fixed at 1.2 (for the -CH_2- and -CH_3 group) or 1.5 (for the pendant -NH_3^+ moieties) times U_{eq} of the atom to which they are attached.

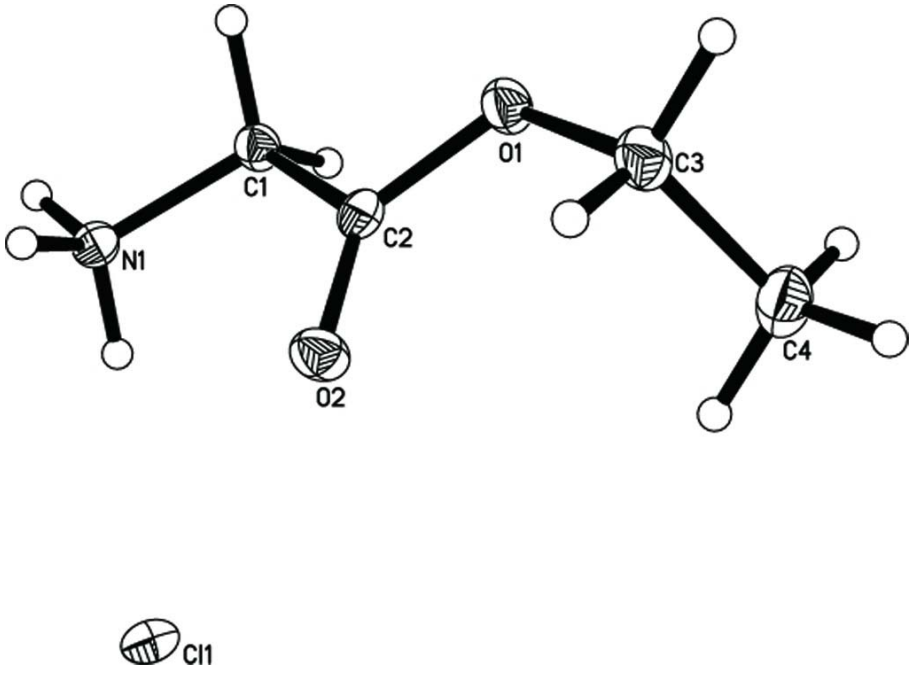

Figure 1

A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 50% probability level.

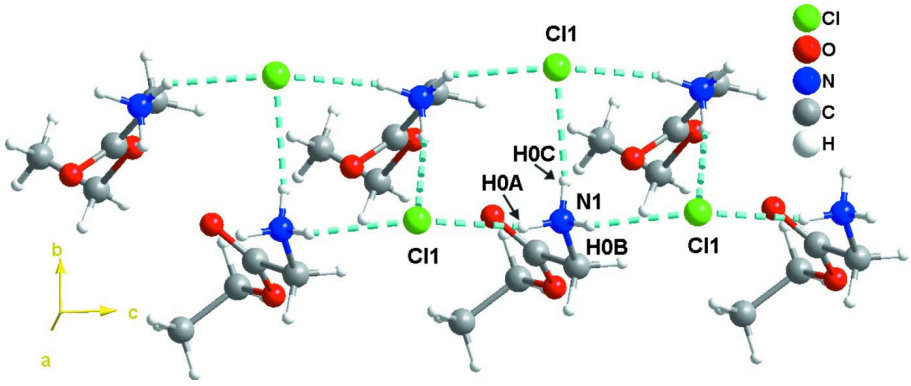

Figure 2

A view of the packing arrangement of the title compound. Hydogran bonds are shown by dashed lines.

3-ethoxy-3-oxopropan-1-aminium chloride

Crystal data

$C_4H_{10}NO_2^+\cdot Cl^-$
M_r = 139.58
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.965 (3) Å
b = 12.543 (4) Å
c = 5.972 (2) Å
β = 103.630 (5)°
V = 652.6 (4) Å^3
Z = 4
$F(000)$ = 296
D_x = 1.421 Mg m^{-3}
Melting point: 145(1) K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1964 reflections
θ = 3.3–27.5°
μ = 0.50 mm^{-1}
T = 123 K
Block, colorless
0.33 × 0.33 × 0.23 mm

Data collection

Rigaku SPIDER diffractometer
Radiation source: Rotating Anode
Graphite monochromator
ω scans
4996 measured reflections
1489 independent reflections
1294 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
$h = -10 \rightarrow 11$
$k = -16 \rightarrow 11$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.064$
$S = 1.00$
1489 reflections
87 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
$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 0.160P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.011 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.00205 (3)	0.38254 (2)	0.24012 (5)	0.01640 (11)
O1	0.52878 (10)	0.38513 (7)	0.85715 (16)	0.0168 (2)
O2	0.34886 (10)	0.29775 (7)	0.59414 (15)	0.0171 (2)
N1	0.11868 (13)	0.36318 (9)	0.7845 (2)	0.0144 (2)
C2	0.38589 (14)	0.35635 (9)	0.7575 (2)	0.0132 (3)
C1	0.27318 (14)	0.40847 (10)	0.8745 (2)	0.0136 (3)
H1A	0.3056	0.3965	1.0429	0.016*
H1B	0.2709	0.4863	0.8461	0.016*
C3	0.64973 (15)	0.34018 (11)	0.7579 (2)	0.0184 (3)
H3A	0.6205	0.2672	0.7010	0.022*
H3B	0.7464	0.3354	0.8786	0.022*
C4	0.67496 (16)	0.40810 (11)	0.5624 (2)	0.0222 (3)
H4A	0.5809	0.4096	0.4392	0.027*
H4B	0.7589	0.3782	0.5029	0.027*
H4C	0.7015	0.4808	0.6179	0.027*

H0A	0.0807 (19)	0.3806 (11)	0.635 (3)	0.022 (4)*
H0B	0.054 (2)	0.3873 (13)	0.869 (3)	0.035 (5)*
H0C	0.1184 (19)	0.2925 (15)	0.797 (3)	0.033 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01827 (17)	0.01928 (19)	0.01196 (16)	0.00511 (12)	0.00419 (11)	0.00078 (11)
O1	0.0131 (4)	0.0206 (5)	0.0171 (5)	−0.0019 (4)	0.0045 (4)	−0.0039 (4)
O2	0.0163 (4)	0.0191 (5)	0.0157 (5)	−0.0003 (4)	0.0033 (4)	−0.0049 (4)
N1	0.0149 (5)	0.0166 (6)	0.0128 (5)	−0.0010 (4)	0.0055 (4)	−0.0022 (4)
C2	0.0150 (6)	0.0120 (6)	0.0131 (6)	−0.0006 (5)	0.0043 (5)	0.0027 (4)
C1	0.0136 (6)	0.0132 (6)	0.0141 (6)	−0.0010 (5)	0.0038 (5)	−0.0020 (5)
C3	0.0130 (6)	0.0224 (7)	0.0202 (7)	0.0013 (5)	0.0046 (5)	−0.0017 (5)
C4	0.0216 (7)	0.0228 (7)	0.0252 (7)	−0.0032 (5)	0.0115 (6)	−0.0029 (6)

Geometric parameters (Å, º)

O1—C2	1.3290 (15)	C1—H1A	0.9900
O1—C3	1.4654 (16)	C1—H1B	0.9900
O2—C2	1.2040 (15)	C3—C4	1.505 (2)
N1—C1	1.4762 (16)	C3—H3A	0.9900
N1—H0A	0.902 (17)	C3—H3B	0.9900
N1—H0B	0.906 (19)	C4—H4A	0.9800
N1—H0C	0.890 (18)	C4—H4B	0.9800
C2—C1	1.5065 (18)	C4—H4C	0.9800
C2—O1—C3	116.20 (10)	C2—C1—H1B	109.7
C1—N1—H0A	111.7 (10)	H1A—C1—H1B	108.2
C1—N1—H0B	109.8 (12)	O1—C3—C4	110.89 (11)
H0A—N1—H0B	109.0 (16)	O1—C3—H3A	109.5
C1—N1—H0C	111.9 (11)	C4—C3—H3A	109.5
H0A—N1—H0C	108.6 (14)	O1—C3—H3B	109.5
H0B—N1—H0C	105.6 (15)	C4—C3—H3B	109.5
O2—C2—O1	125.54 (12)	H3A—C3—H3B	108.0
O2—C2—C1	123.62 (12)	C3—C4—H4A	109.5
O1—C2—C1	110.83 (11)	C3—C4—H4B	109.5
N1—C1—C2	109.79 (10)	H4A—C4—H4B	109.5
N1—C1—H1A	109.7	C3—C4—H4C	109.5
C2—C1—H1A	109.7	H4A—C4—H4C	109.5
N1—C1—H1B	109.7	H4B—C4—H4C	109.5
C3—O1—C2—O2	−0.45 (18)	O1—C2—C1—N1	−171.55 (10)
C3—O1—C2—C1	−179.62 (10)	C2—O1—C3—C4	86.87 (14)
O2—C2—C1—N1	9.27 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H0*A*···Cl1	0.904 (17)	2.300 (17)	3.1845 (16)	166.1 (12)
N1—H0*B*···Cl1^{i}	0.906 (18)	2.386 (18)	3.1658 (16)	144.3 (15)
N1—H0*C*···Cl1	0.890 (19)	2.435 (19)	3.2566 (16)	153.7 (15)
C1—H1*A*···O2	0.99	2.47	2.9072 (18)	106
C3—H3*B*···Cl1^{ii}	0.99	2.79	3.7529 (18)	164

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) *x*+1, *y*, *z*+1.