

β -Alaninium perchlorateS. Pandiarajan, B. Sridhar and
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Key indicators

Single-crystal X-ray study

T = 293 K

Mean σ (C–C) = 0.005 Å

Disorder in solvent or counterion

R factor = 0.062

wR factor = 0.216

Data-to-parameter ratio = 9.5

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

In the title compound, $C_3H_8NO_2^+ \cdot ClO_4^-$, a normal O–H...O hydrogen bond is observed which forms a dimer between the carboxylic acid groups of two β -alaninium residues related by an inversion center. An intramolecular hydrogen bond is observed between the amino-N and carboxyl-O atoms. The amino-N atom is also involved in a three-centered hydrogen bond with O atoms of the perchlorate anion across the center of inversion, forming infinite chains.

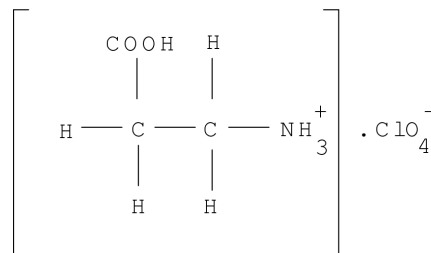
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Comment

Alanine is the second simplest amino acid, but the most common in proteins. β -Alanine is the only naturally occurring β -amino acid. The crystal structure of L-alanine (Lehmann *et al.*, 1972), L-alanine hydrochloride (Di Blasio *et al.*, 1977), β -alanine (Papavinasam *et al.*, 1986), bis(DL-alanine) phosphate (Averbuch-Pouchot *et al.*, 1988), DL-alanine nitrate (Asath Bahadur & Rajaram, 1995) and bis(β -alanine) hydrogen nitrate (Sridhar *et al.*, 2001) have been reported. In the present investigation, β -alanine was reacted with perchloric acid to produce the title compound (I) which was investigated to study the conformation and hydrogen bonds in the presence of an inorganic acid.



(I)

The asymmetric unit of (I) consists of one β -alaninium residue and a perchlorate anion. The backbone conformation angles ψ^1 and ψ^2 are 8.0 (4) and -171.5 (3) $^\circ$, respectively, for the alaninium residue. The straight-chain conformation angle χ^1 is in the *gauche* II form [-65.0 (3) $^\circ$].

In the perchlorate anion, all the O atoms are found to have orientational disorder. This leads to considerable variations in the Cl–O bond distances and the tetrahedral symmetry of the anion.

In biological molecules, such as amino acids, hydrogen bonds play an important role. In the present structure, a normal O–H...O hydrogen bond (2.726 Å) is observed, which forms a dimer between the carboxylic acid groups of two β -alaninium residues related by an inversion center. The

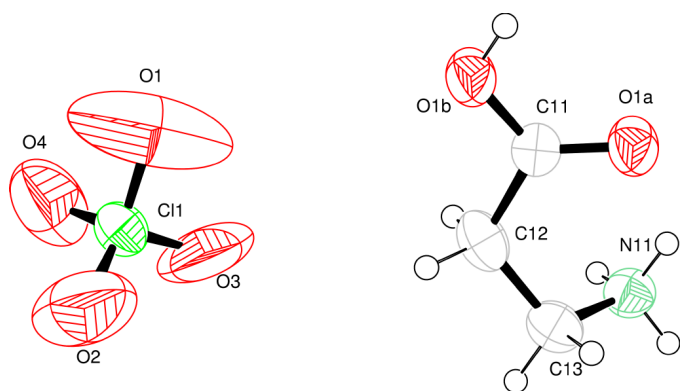


Figure 1

The molecular structures of the cation and anion of (I), showing the atomic numbering scheme and 50% probability displacement ellipsoids (Johnson, 1976). The minor-site disordered O atoms have been omitted.

hydrogen bonds that exist between the perchlorate anion and the alanine residue play an important role in stabilizing the structure. The amino-N atom is also involved in a chelated three-centered hydrogen bond with acceptor O atoms (O2 and O3) of the perchlorate anion. The amino-N atom is also found to be engaged in a three-centered hydrogen bond, with (i) the carboxyl atom O1A (intramolecular hydrogen bond) and atom O4 of the perchlorate anion and (ii) two O atoms of the perchlorate anions across a center of inversion, forming infinite chains. The presence of the three-centered hydrogen bond is due to an excess of acceptors over donors or proton deficiency (Jeffrey & Saenger, 1991).

Experimental

The title compound was crystallized from an aqueous solution of β -alanine and perchloric acid in a 1:1 stoichiometric ratio by slow evaporation.

Crystal data

$C_3H_8NO_2^+ \cdot ClO_4^-$
 $M_r = 189.55$
 Monoclinic, $P2_1/n$
 $a = 7.024$ (3) Å
 $b = 7.556$ (4) Å
 $c = 14.102$ (4) Å
 $\beta = 97.52$ (4)°
 $V = 742.0$ (5) Å³
 $Z = 4$
 $D_x = 1.697$ Mg m⁻³
 $D_m = 1.690$ Mg m⁻³

D_m measured by flotation using a mixture of carbon tetrachloride and xylene
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 6.4$ – 13.6 °
 $\mu = 0.50$ mm⁻¹
 $T = 293$ (2) K
 Needle, colorless
 $0.3 \times 0.2 \times 0.1$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.886$, $T_{\max} = 0.951$
 1521 measured reflections
 1297 independent reflections
 1049 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$
 $\theta_{\text{max}} = 24.8$ °
 $h = -8 \rightarrow 8$
 $k = 0 \rightarrow 8$
 $l = 0 \rightarrow 16$
 3 standard reflections
 frequency: 60 min
 intensity decay: none

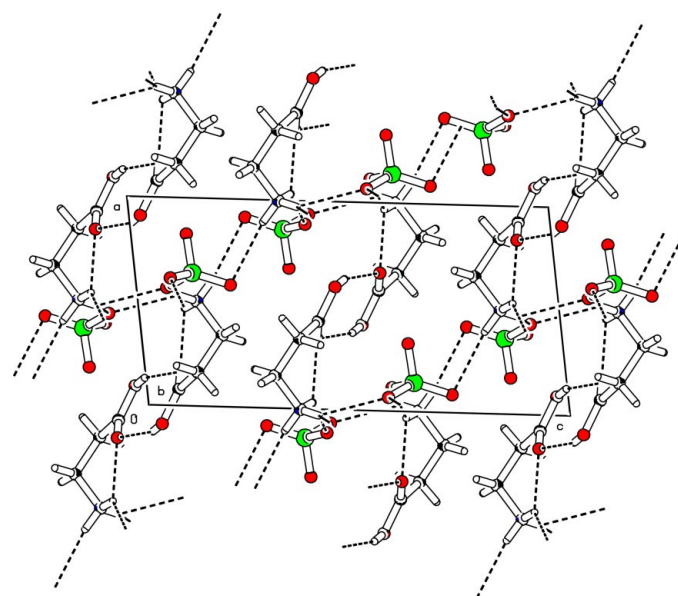


Figure 2

Packing diagram of the structure viewed down the b axis. The minor-site disordered O atoms have been omitted.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.216$
 $S = 1.12$
 1297 reflections
 136 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1724P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.48$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1A—C11	1.213 (4)	O1B—C11	1.287 (4)
O1A—C11—C12—C13	8.0 (4)	C11—C12—C13—N11	−65.0 (3)
O1B—C11—C12—C13	−171.5 (3)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1B—H12 \cdots O1A ⁱ	0.82	1.94	2.726 (3)	161
N11—H11A \cdots O2 ⁱⁱ	0.89	2.37	3.243 (12)	165
N11—H11A \cdots O2 ⁱⁱ	0.89	2.06	2.939 (10)	172
N11—H11A \cdots O3 ⁱⁱ	0.89	2.60	3.288 (9)	135
N11—H11A \cdots O3 ⁱⁱ	0.89	2.55	3.19 (2)	130
N11—H11B \cdots O4 ⁱⁱⁱ	0.89	2.24	2.941 (9)	135
N11—H11B \cdots O4 ⁱⁱⁱ	0.89	2.46	3.19 (3)	139
N11—H11B \cdots O1A	0.89	2.27	2.875 (3)	125
N11—H11C \cdots O1 ^{iv}	0.89	2.19	2.971 (15)	146
N11—H11C \cdots O1 ^{iv}	0.89	2.17	2.97 (2)	149
N11—H11C \cdots O1 ^v	0.89	2.42	3.064 (17)	129
N11—H11C \cdots O1 ^v	0.89	2.49	3.10 (2)	127

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

The perchlorate anion exhibits orientational disorder. The site-occupation factors for O1/O2/O3/O4 and O1'/O2'/O3'/O4' are 0.57 and 0.43, respectively. These O atoms were refined anisotropically with fixed site-occupation factors. All H atoms were fixed by geometric constraints using HFIX and allowed to ride on the attached atom.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL97*.

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