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β-Alaninium trichloroacetate at 105 K

K. Rajagopal,[a] R. V. Krishnakumar,[b] M. Subha Nandhini,[c] A. Mostad[d] and S. Natarajan[c]*

[a]Department of Physics, Saraswathi Narayanan College, Madurai 625 022, India, [b]Department of Physics, Thiagarajar College, Madurai 625 009, India, [c]Department of Physics, Madurai Kamaraj University, Madurai 625 021, India, and [d]Department of Chemistry, University of Oslo, PO Box 1033 Blindern, N-0315 Oslo 3, Norway

Correspondence e-mail: s_natarajan50@yahoo.com

Key indicators

Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.104
Data-to-parameter ratio = 20.5

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

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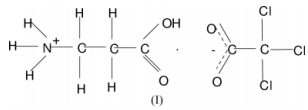
In the title compound, $C_3H_8NO_2^+\cdot C_2Cl_3O_2^-$, the β-alanine molecule exists in the cationic form, with a positively charged amino group and an uncharged carboxylic acid group. The trichloroacetic acid molecule exists in the anionic state. The structure is stabilized by a three-dimensional network of O—H···O, N—H···O and N—H···Cl interactions. There are no direct hydrogen-bonded interactions between the trichloroacetate anions. The nature of the interactions between individual molecules is similar to that in DL-valinium trichloroacetate.

Comment

Precise X-ray crystallographic investigations on amino acid–carboxylic acid complexes have provided a wealth of information regarding intermolecular interactions and biomolecular aggregation patterns that might well have occurred in prebiotic polymerization (Vijayan, 1988; Prasad & Vijayan, 1993). The crystal structures of β-alanine (Papavinasam *et al.*, 1986), β-alaninium maleate (Rajagopal *et al.*, 2001), bis(β-alanine) hydrogen nitrate (Sridhar *et al.*, 2001), β-alaninium perchlorate (Pandiarajan *et al.*, 2001), β-alaninium oxalate hemihydrate (Krishnakumar *et al.*, 2002), DL-valinium trichloroacetate (Rajagopal *et al.*, 2002) and DL-methioninium trichloroacetate (Rajagopal *et al.*, 2003) have already been reported. A brief survey of the Cambridge Structural Database (Allen, 2002) revealed a scarcity of precise crystallographic data on amino acid–halogenoacetic acid complexes. We report here the crystal structure of a complex of β-alanine with trichloroacetic acid, namely β-alaninium trichloroacetate, (I). β-Alanine (3-aminopropionic acid) is the only naturally occurring β-amino acid and is a component of the naturally occurring peptides carnosine and anserine, and also of pantothenic acid. Trichloroacetic acid is an excellent medicine for wrinkles formed in the skin.



(I)

Fig. 1 shows the molecular structure of (I) with the atom-numbering scheme. The β-alanine molecule in (I) exists in the cationic form, with a positively charged amino group and an uncharged carboxylic acid group. The trichloroacetic acid molecule exists as an anion. The asymmetric unit of (I) consists of one β-alanininium residue and a trichloroacetate anion. The backbone conformation angles ψ^1 and ψ^2 are 22.3 (3) and −159.62 (1)°, respectively, for the alaninium residue. These are significantly different from the values

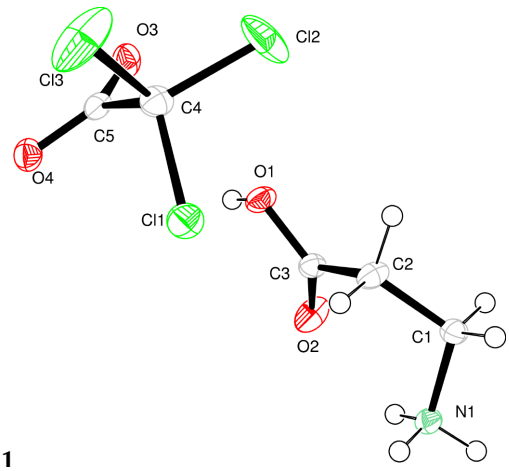


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids.

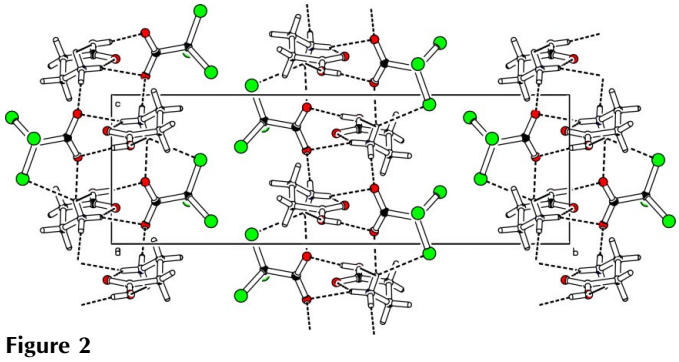


Figure 2
Packing of the molecules of (I), viewed down the *a* axis.

reported for β-alanine (25.3 and −177.8°), β-alaninium oxalate hemihydrate [8.3 (2) and −173.0 (2)°] and β-alaninium perchlorate [8.0 (4) and −171.5 (3)°], but are in good agreement with the values reported for β-alaninium maleate [24.6 (4) and −155.8 (2)°]. The straight-chain conformation angle χ^1 is in the *gauche* II form [−58.9 (2)°], as was also observed in β-alaninium perchlorate [−65.0 (3)°]. The straight-chain conformation angles for β-alanine, β-alaninium maleate and β-alaninium oxalate hemihydrate are −154.8, −177.4 (2) and 77.0 (2)°, respectively, indicating different conformations.

Fig. 2 shows the packing of molecules of (I), viewed down the *a* axis. In the crystal, the alanine and trichloroacetic acid molecules are alternately linked by O—H···O and N—H···O hydrogen bonds to form infinite one-dimensional chains along [110]. The glide-related chains are interlinked by N—H···O hydrogen bonds to form an infinite two-dimensional network parallel to (001), similar to that in DL-valinium trichloroacetate. The trichloroacetate ions do not have direct hydrogen-bonded interactions among themselves. The β-alaninium ions link trichloroacetate ions through bifurcated N—H···O hydrogen bonds. The O—H···O, N—H···O and N—H···Cl interactions that exist between the trichloroacetate anion and the alaninium residue play an important role in stabilizing the structure. A short contact between Cl1 and Cl2($x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$) of 3.428 (1) Å is also observed in the structure. Strikingly, the title compound, (I), β-alaninium maleate and β-alaninium perchlorate all crystallize in the same space group, but the crystal packings are distinctly different.

Experimental

Colourless, plate-shaped single crystals of (I) were grown from a saturated aqueous solution containing β-alanine and trichloroacetic acid in a 1:1 stoichiometric ratio.

Crystal data

$C_3H_8NO_2^+\cdot C_2Cl_3O_2^-$
M_r = 252.47
Monoclinic, $P2_1/n$
a = 6.8049 (14) Å
b = 21.100 (4) Å
c = 6.8968 (14) Å
β = 95.75 (3)°
V = 985.3 (3) Å^3
Z = 4
D_x = 1.702 Mg m^{-3}
D_m = 1.69 Mg m^{-3}
D_m measured by flotation in a mixture of xylene and bromoform
Mo $K\alpha$ radiation
Cell parameters from 1024 reflections
θ = 3–28°
μ = 0.91 mm^{-1}
T = 105 (2) K
Plate, colourless
0.4 × 0.3 × 0.3 mm

Data collection

Bruker SMART diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.694, T_{max} = 0.761
12338 measured reflections
2442 independent reflections
2350 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 28.3°
$h = -9 \rightarrow 9$
$k = -28 \rightarrow 28$
$l = -9 \rightarrow 9$
1024 standard reflections
every 100 reflections
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.104
S = 1.05
2442 reflections
119 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 2.3578P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.75 e Å^{-3}
$\Delta\rho_{min}$ = −1.39 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0067 (14)

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.765 (2)	O2—C3	1.214 (3)
Cl2—C4	1.776 (2)	O3—C5	1.251 (3)
Cl3—C4	1.776 (2)	O4—C5	1.240 (3)
O1—C3	1.321 (3)		
O2—C3—O1	123.4 (2)	O4—C5—O3	129.1 (2)
O2—C3—C2	123.63 (19)	O4—C5—C4	114.73 (19)
O1—C3—C2	112.95 (18)	O3—C5—C4	116.15 (19)
N1—C1—C2—C3	−58.9 (2)	C1—C2—C3—O1	−159.62 (18)
C1—C2—C3—O2	22.3 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.82	1.83	2.649 (2)	173
N1—H1A···O4^{ii}	0.89	1.96	2.840 (2)	172
N1—H1B···O2	0.89	2.14	2.785 (3)	129
N1—H1B···O4^{iii}	0.89	2.32	3.016 (2)	135
N1—H1C···Cl2^{iv}	0.89	2.78	3.458 (2)	134
N1—H1C···O3^{iv}	0.89	2.04	2.846 (3)	150

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

All the H atoms were positioned geometrically and were allowed to ride on their respective parent atoms with *SHELXL*97 (Sheldrick, 1997) defaults for bond lengths and displacement parameters. The residual density peaks in the final difference Fourier map (1.75 and -1.39 e Å^{-3}) indicate ripples around the Cl atoms and have no structural significance.

Data collection: *SMART-NT* (Bruker, 1999); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (Bruker, 1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.

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