β-Alaninium oxalate hemihydrate

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In the title compound, $C_3H_8NO_2^+ \cdot C_2HO_4^- \cdot 0.5H_2O$, the β-alanine molecule exists in the cationic form and the oxalic acid in the mono-ionized state. The water molecule occurs as a hemihydrate, lying on a twofold axis within the unit cell. The centrosymmetrically related semi-oxalate anions are surrounded by six β-alanine molecules and the complex may be described as an inclusion compound.

Comment

X-ray studies on crystalline complexes of amino acids with carboxylic acids have provided a wealth of information regarding intermolecular interactions and biomolecular aggregation patterns (Vijayan, 1988; Prasad & Vijayan, 1993). The crystal structures of complexes of oxalic acid with DL-tryptophan (Bakke & Mostad, 1980), L- and DL-histidine (Prabu et al., 1996), L- and DL-arginine (Chandra et al., 1998), sarcosine (Krishnakumar et al., 1998), glycine (Subha Nandhini et al., 2001a), L-alanine (Subha Nandhini et al., 2001b), and DL-alanine (Subha Nandhini et al., 2001c) have already been reported. The present study reports the crystal structure of a complex of β-alanine with oxalic acid.

(I)
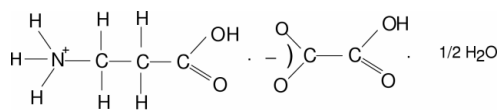
The β-alanine molecule exists in the cationic form with a neutral carboxyl group and a positively charged amino group. The oxalic acid molecule exists in the mono-ionized state as a semi-oxalate anion and is essentially planar. The C1=O2 bond distance of the carboxylic acid group in the β-alaninium cation has a value of 1.271 (2) Å, which is larger than the usually expected C=O bond distance. The larger value may be attributed to the presence of an O—H···O hydrogen bond among the carboxyl groups of the symmetrically related amino acid molecules, leading to the formation of a dimer. Similar instances of the carboxylic acid group exhibiting significant lengthening of the acceptor (C=O) and the shortening of the donor (C—OH) bond owing to valence-bond resonance effects have been found in the Cambridge Crystallographic Database (Allen & Kennard, 1993).

Fig. 2 shows the packing of molecules viewed down the b axis, in which a pair of centrosymmetrically related oxalate ions is surrounded by six neighbouring β-alanine molecules. The water molecule exists as a hemihydrate, lying on a twofold axis within the unit cell. It participates in the hydrogen bond
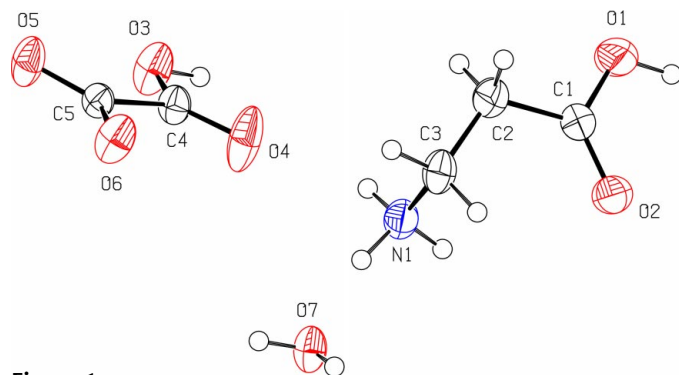
Figure 1
The molecular structure of (I) with the atom-numbering scheme. Displacement ellipsoids are at the 50% probability level.

network both as an acceptor and a donor, mediating interactions, between the oxalate anion and the β-alaninium cation. The semi-oxalate ions aggregate into hydrogen-bonded strings generated by translations along the shortest cell axis b. The crystal structure is also characterized by the presence of a C—H···O hydrogen bond between the amino acid and the semi-oxalic acid molecules. The complex can be described as an inclusion compound with the amino acid as the host and the semi-oxalic acid as the guest.

Experimental

Colourless single crystals of (I) were grown as transparent needles by slow evaporation of a saturated aqueous solution containing the oxalic acid and β-alanine in a 1:1 stoichiometric ratio.

Crystal data

$C_3H_8NO_2^+ \cdot C_2HO_4^- \cdot 0.5H_2O$	D_m measured by flotation in a liquid
$M_r = 188.14$	mixture of xylene and bromoform
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 22.373$ (3) Å	Cell parameters from 25
$b = 5.6925$ (7) Å	reflections
$c = 14.917$ (3) Å	$\theta = 6$–$22°$
$\beta = 115.31$ (1)°	$\mu = 0.14$ mm^{-1}
$V = 1717.5$ (4) Å^3	$T = 293$ (2) K
$Z = 8$	Needle, colourless
$D_x = 1.455$ Mg m^{-3}	$0.44 \times 0.30 \times 0.20$ mm
$D_m = 1.46$ Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4	$R_{int} = 0.023$
diffractometer	$\theta_{max} = 25.0°$
ω–2θ scans	$h = 0 \rightarrow 26$
Absorption correction: ψ scan	$k = 0 \rightarrow 6$
(North et al., 1968)	$l = -16 \rightarrow 15$
$T_{min} = 0.978$, $T_{max} = 0.996$	2 standard reflections
1463 measured reflections	frequency: 60 min
1424 independent reflections	intensity decay: <2%
1189 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$+ 1.2943P]$
$wR(F^2) = 0.090$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
1424 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
156 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
All H-atom parameters refined	Extinction correction: SHELXL97
	Extinction coefficient: 0.047 (2)
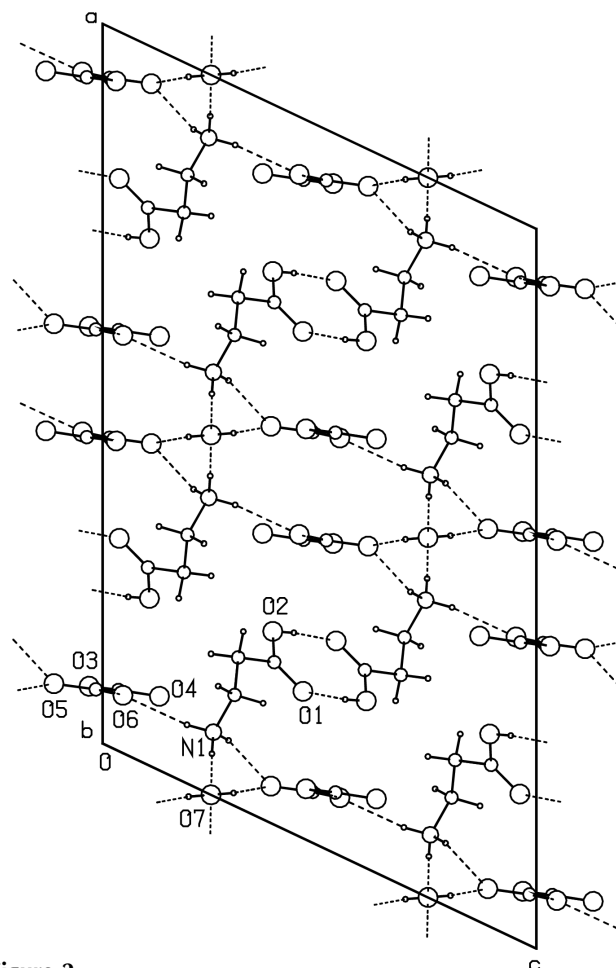
Figure 2
Packing diagram of (I) viewed down the b axis.

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.303 (2)	O6—C5	1.284 (2)
O2—C1	1.271 (2)	N1—C3	1.480 (2)
O3—C4	1.339 (2)	C1—C2	1.540 (3)
O4—C4	1.275 (2)	C2—C3	1.506 (3)
O5—C5	1.313 (2)	C4—C5	1.565 (2)
O2—C1—C2—C3	8.3 (3)	C1—C2—C3—N1	77.0 (2)
O1—C1—C2—C3	−173.00 (17)	O4—C4—C5—O6	3.3 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O1···O2^i	0.89 (3)	1.87 (3)	2.750 (2)	170 (3)
O3—H1O3···O6^{ii}	0.96 (3)	1.65 (3)	2.6098 (17)	172 (2)
N1—H1N1···O7^{iii}	0.94 (3)	1.87 (3)	2.787 (2)	165 (2)
N1—H2N1···O6	0.92 (3)	2.21 (2)	3.039 (2)	150.0 (19)
N1—H3N1···O5^{iv}	0.93 (2)	2.06 (2)	2.955 (2)	163.3 (18)
O7—H1OW···O5^{iv}	0.90 (2)	1.91 (2)	2.7818 (17)	163 (2)
C3—H1C3···O4^{iii}	0.99 (2)	2.44 (2)	3.386 (2)	158.7 (15)

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

All the H atoms were clearly revealed in a difference Fourier map and were included in the refinement with isotropic displacement parameters. The C—H, N—H and O—H bond lengths are 0.96 (3)–1.03 (2), 0.92 (2)–0.94 (3) and 0.89 (3)–0.96 (3) Å, respectively.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); 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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