C(14*A*)	−0.0750 (3)	0.5548 (10)	0.5449 (7)	0.069 (2)
C(15*A*)	−0.0491 (3)	0.4543 (10)	0.5790 (7)	0.074 (2)
O(16*A*)	−0.0232 (3)	0.4345 (13)	0.6825 (8)	0.103 (2)
O(16*B*)	−0.0312 (3)	0.3642 (12)	0.3118 (9)	0.092 (2)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(2)	1.411 (5)	O(1*B*)—C(6*A*)	1.363 (8)
C(7)—N(8)	1.418 (8)	C(7*A*)—N(8*A*)	1.428 (7)
N(8)—C(9)	1.317 (6)	N(8*A*)—C(9*A*)	1.301 (7)
C(9)—C(10)	1.395 (9)	C(9*A*)—C(10*A*)	1.419 (7)
O(16)—C(15)	1.399 (6)	O(16*A*)—C(15*A*)	1.397 (8)
O(1*A*)—C(2*A*)	1.388 (8)	O(16*B*)—C(11*A*)	1.385 (8)
C(7)—C(2)—O(1)	119.2 (5)	C(7*A*)—C(6*A*)—O(1*B*)	110.6 (8)
N(8)—C(7)—C(2)	115.8 (5)	N(8*A*)—C(7*A*)—C(2*A*)	120.3 (6)
N(8)—C(7)—C(6)	123.9 (6)	N(8*A*)—C(7*A*)—C(6*A*)	122.8 (7)
C(9)—N(8)—C(7)	128.4 (5)	C(9*A*)—N(8*A*)—C(7*A*)	121.8 (8)
C(10)—C(9)—N(8)	125.7 (6)	C(9*A*)—C(10*A*)—C(11*A*)	123.6 (7)
C(11)—C(10)—C(9)	119.7 (6)	C(9*A*)—C(10*A*)—C(15*A*)	118.8 (7)
C(15)—C(10)—C(9)	122.2 (6)	C(10*A*)—C(9*A*)—N(8*A*)	120.5 (8)
O(16)—C(15)—C(10)	121.8 (6)	O(16*A*)—C(15*A*)—C(10*A*)	105.8 (8)
C(7*A*)—C(2*A*)—O(1*A*)	106.6 (8)	O(16*B*)—C(11*A*)—C(10*A*)	111.3 (8)

Refinement was based on full-matrix least-squares methods with H atoms in calculated positions (C—H 1.08 Å), except for the H atoms at O(1) (0.821 Å) and O(16) (0.863 Å), which were taken from a difference Fourier map and whose coordinates were kept fixed during the refinement, together with those of the C(9) and C(9*A*) H atoms. The coordinates and isotropic temperature factors were kept fixed during refinement for all H atoms. The bond lengths C(15)—O(16), O(1)—C(2), N(8)—C(9), O(18)—C(6*A*), N(8*A*)—C(9*A*), O(1*A*)—C(2*A*), O(16*B*)—C(15*A*), C(11*A*)—O(16*A*), C(7*A*)—N(8*A*) and C(9*A*)—C(10*A*) were constrained during the refinement. The relatively high residuals can be attributed to the problem of modelling disorder in the second molecule coupled with radiation damage and weak diffraction (pairs of disordered atoms are labelled *A* and *B*)

Data collection: *Enraf–Nonius Structure Determination Package* (Frenz, 1985). Cell refinement: *Enraf–Nonius Structure Determination Package*. Data reduction: *Enraf–Nonius Structure Determination Package*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1119). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Aminoethylammonium Tartrate

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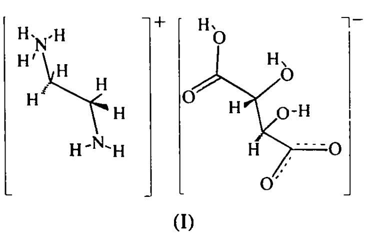
Abstract

The structure of aminoethylammonium tartrate, $C_2H_9N_2^+.C_4H_5O_6^-$, is ionic. Ethylenediamine forms a very stable salt with tartaric acid, similar to $C_2H_{10}N_2^{2+}.2HPO_4^{2-}.6H_2O$ [Averbuch-Pouchot, Durif & Guitel (1987). *Acta Cryst.* **C43**, 1896–1898] and $C_2H_{10}N_2^{2+}.2C_4H_5O_6^-.2H_2O$ [Perez, (1977). *Acta Cryst.* **B33**, 1083–1087]. The protonated ethylenediamine monocations are linked to the tartrate anions by strong N—H···O hydrogen bonds [N···O 2.921 (4), H···O 2.083 (4) Å, N—H···O 156.8 (3)°]. The bond lengths and angles are comparable with corresponding values observed in related molecules.

Comment

The cation and anion in the title salt, (I), do not display any unusual structural features, the C—C—C—C chain of the tartrate anion being antiperiplanar with a torsion angle value of 170.4 (3)°. The two halves of the tartrate anion, consisting of atoms C1, C2, O1, O2 and O3, and C3, C4, O4, O5 and O6, form individual planes, the dihedral angle between them being 32.16 (12)°. This dihedral angle is significantly smaller than that observed in other tartrates, *e.g.* 41° in manganese(II) L-tartrate (Soylu, 1985), 54.6° in D-tartaric acid (Okaya, Stemple & Kay, 1966), 57.3° in potassium hydrogen



(I)

L(+)-tartrate (Akkurt, Hökelek & Soylu, 1987), 62° in ammonium tartrate (Yadava & Padmanabhan, 1973), 63° in tartaric acid (Stern & Beevers, 1950), 69° in sodium D-tartrate dihydrate (Ambady & Kartha, 1968) and 90° in strontium tartrate trihydrate (Ambady, 1968).

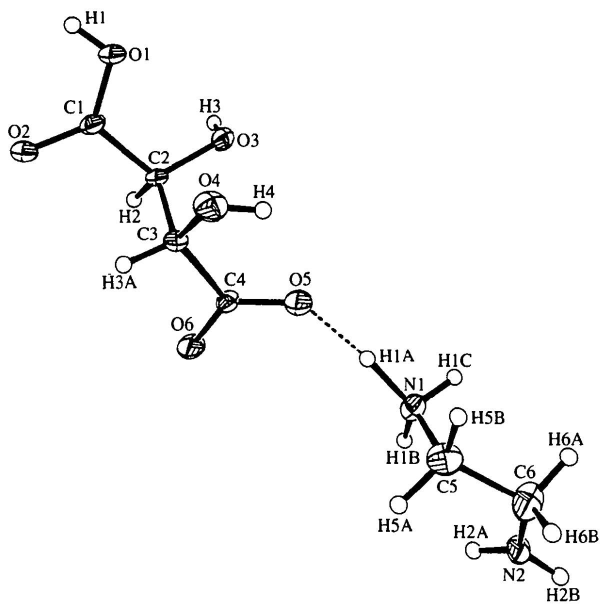


Fig. 1. A perspective view of the molecular structure of the title compound with the atom-numbering scheme. The displacement ellipsoids are plotted at the 50% probability level.

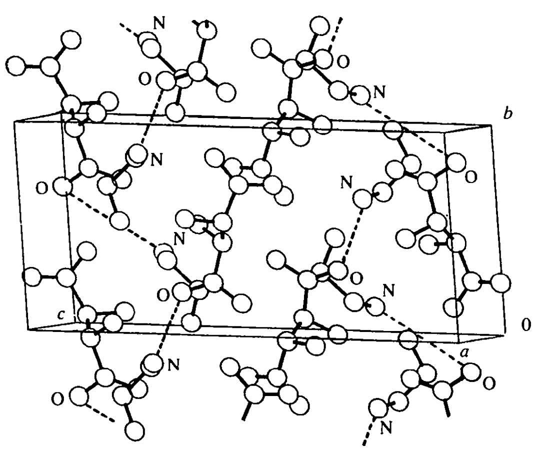


Fig. 2. A view of the crystal packing along the *a* axis. H atoms are omitted for clarity and hydrogen bonds are represented by dashed lines.

Experimental

The title compound was prepared by mixing equimolar amounts of ethylenediamine and tartaric acid. After evaporation of the solvent, light-orange crystals were formed. The density D_m was measured by flotation in CCl_4/C_7H_8.

Crystal data

$C_2H_9N_2^+.C_4H_5O_6^-$
$M_r = 210.19$
Orthorhombic
$P2_12_12_1$
$a = 7.4393 (8)$ Å
$b = 7.6507 (4)$ Å
$c = 15.6806 (8)$ Å
$V = 892.47 (12)$ Å^3
$Z = 4$
$D_x = 1.564$ Mg m^{-3}
$D_m = 1.540$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10–19°$
$\mu = 0.140$ mm^{-1}
$T = 293 (2)$ K
Parallelepiped
0.60 × 0.40 × 0.28 mm
Light orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.535$, $T_{max} = 0.999$
1079 measured reflections
1077 independent reflections

1009 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0411$
$\theta_{max} = 26.28°$
$h = -9 \rightarrow 0$
$k = -9 \rightarrow 0$
$l = -19 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: 1.8%

Refinement

Refinement on F^2
$R(F) = 0.0618$
$wR(F^2) = 0.1706$
$S = 1.131$
1076 reflections
168 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1361P)^2 + 0.1616P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.042$

$\Delta\rho_{max} = 0.457$ e Å^{-3}
$\Delta\rho_{min} = -0.526$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = −5.3 (25)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.3766 (5)	0.7807 (4)	0.4987 (2)	0.0246 (7)
C2	0.4354 (4)	0.9580 (4)	0.4626 (2)	0.0220 (7)
C3	0.2656 (4)	1.0640 (4)	0.4402 (2)	0.0219 (7)
C4	0.3164 (4)	1.2512 (4)	0.4186 (2)	0.0223 (7)
C5	0.1882 (5)	1.6761 (5)	0.2414 (3)	0.0356 (8)
C6	0.1794 (7)	1.8120 (5)	0.1722 (3)	0.0415 (10)
O1	0.3961 (5)	0.6521 (3)	0.4503 (2)	0.0395 (8)
O2	0.3132 (4)	0.7787 (3)	0.57235 (15)	0.0307 (6)
O3	0.5396 (4)	0.9435 (4)	0.3870 (2)	0.0282 (6)
O4	0.1666 (4)	0.9841 (4)	0.3742 (2)	0.0337 (7)
O5	0.3260 (4)	1.3022 (4)	0.3444 (2)	0.0371 (7)
O6	0.3469 (5)	1.3455 (3)	0.4850 (2)	0.0366 (7)
N1	0.3768 (5)	1.6483 (4)	0.2696 (2)	0.0322 (7)
N2	0.2470 (5)	1.9754 (4)	0.2022 (2)	0.0288 (7)

Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.534 (4)	O3—C2	1.420 (4)
C3—C2	1.541 (5)	O4—C3	1.409 (4)
C3—C4	1.520 (4)	O5—C4	1.228 (4)
C5—C6	1.504 (6)	O6—C4	1.287 (4)
O1—C1	1.251 (4)	N1—C5	1.487 (5)
O2—C1	1.248 (4)	N2—C6	1.427 (5)

O2—C1—O1	126.6 (3)	O4—C3—C4	112.0 (3)
O2—C1—C2	117.4 (3)	C4—C3—C2	110.0 (3)
O1—C1—C2	116.0 (3)	O5—C4—O6	125.3 (3)
O3—C2—C1	113.2 (3)	O5—C4—C3	121.7 (3)
O3—C2—C3	107.4 (3)	O6—C4—C3	113.0 (3)
C1—C2—C3	108.4 (3)	N1—C5—C6	110.7 (3)
O4—C3—C2	111.6 (3)	N2—C6—C5	110.6 (3)
O4—C3—C4—O5	−23.5 (4)	O1—C1—C2—C3	105.8 (4)
C2—C3—C4—O5	101.1 (4)	O4—C3—C2—O3	58.0 (3)
O4—C3—C4—O6	156.5 (3)	C4—C3—C2—O3	−67.0 (3)
C2—C3—C4—O6	−78.8 (3)	O4—C3—C2—C1	−64.7 (3)
O2—C1—C2—O3	167.4 (3)	C4—C3—C2—C1	170.4 (2)
O1—C1—C2—O3	−13.3 (5)	N1—C5—C6—N2	−60.6 (5)
O2—C1—C2—C3	−73.5 (4)		

Table 3. *Least-squares-planes data*

Plane 1: C1, C2, C3, C4; equation: $-0.375(30)x + 2.477(11)y + 14.815(6)z = 9.121(18)$. Plane 2: C1, C2, O1, O2, O3; equation: $6.589(4)x - 1.360(15)y + 6.723(22)z = 4.785(20)$. Plane 3: C3, C4, O4, O5, O6; equation: $-6.851(3)x - 2.909(9)y - 1.343(32)z = 2.066(15)$.

Dihedral angles (°)

Plane 1/Plane 2	72.37 (24)	Plane 2/Plane 3	32.16 (12)
Plane 1/Plane 3	75.49 (25)		

Deviations of atoms (Å) from their least-squares planes

	Plane 1	Plane 2	Plane 3
C1	0.060 (2)	−0.012 (3)	
C2	−0.058 (2)	−0.109 (2)	
C3	−0.063 (2)		−0.199 (2)
C4	0.061 (2)		−0.032 (3)
O1		−0.035 (1)	
O2		0.068 (2)	
O3		0.089 (2)	
O4			0.158 (2)
O5			−0.049 (1)
O6			0.123 (2)

H atoms, except H1, H2*A* and H2*B*, were obtained by difference Fourier syntheses and were refined isotropically. Atoms H1, H2*A* and H2*B* were located geometrically.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1993). Cell refinement: *CAD*-4-*PC*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1121). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Acetoxymethylpyrrole-2-carbaldehyde

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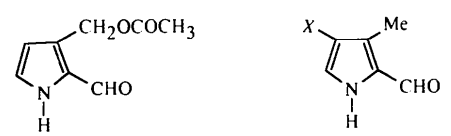
Abstract

In the title compound (the ester 2-formyl-3-pyrrolylmethyl acetate, $C_8H_9NO_3$), hydrogen bonding between the N—H function in one molecule and the ester carbonyl O atom in a molecule related to the first by the 2_1 screw axis leads to the formation of zigzag chains.

Comment

3-Acetoxymethylpyrrole-2-carbaldehyde, (1), was prepared as an intermediate in a new synthetic route to 3,8-didehydroheliotridin-5-one (McNab & Thornley, 1993). Because of interest in the crystal structures of 3-substituted pyrrole-2-carbaldehyde derivatives [(2)–(4)] (Smith, Bobe, Minnetian, Hope & Yanuck, 1985), we undertook a determination of its structure at 150 K.



(1)

(2) X = H
(3) X = Et
(4) X = Cl

(I)