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(*S*)-2-Ammoniobutan-1-ol (2*R*,3*R*)-tartrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.083
Data-to-parameter ratio = 9.1

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

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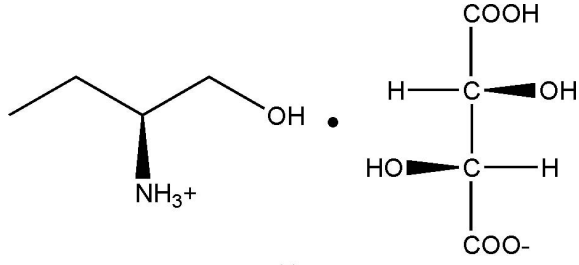
The formation of the title compound, (*S*)-2-ammonio-1-butanol (2*R*,3*R*)-tartrate, $C_4H_{12}NO^+\cdot C_4H_5O_6^-$, the (*S*)-2-amino-1-butanol molecule is converted to a cationic form containing a positively charged amino group, and the tartaric acid molecule to a mono- or half-ionized tartrate anion. The structure is stabilized by a three-dimensional network of hydrogen bonds.

Comment

The title compound, (I), is a (+)-tartrate salt of the (+)-enantiomer of 2-amino-1-butanol. (*S*)-2-Amino-1-butanol is an intermediate in the synthesis of ethambutol hydrochloride, a widely used antituberculosis agent (Fadnavis *et al.*, 1999). It has been reported (Singh, 1976; Bai *et al.*, 2004) that the resolution of racemic 2-amino-1-butanol with (2*R*,3*R*)-tartaric acid is the best way to obtain the enantiomerically pure (*S*)-2-amino-1-butanol which is needed for the ethambutol synthesis. (*S*)-2-Ammonio-1-butanol (2*R*,3*R*)-tartrate, (I), is thus a key intermediate in this process and its structure is reported here.



(I)

A view of (I) and its numbering scheme are illustrated in Fig. 1. In this crystal form, the (*S*)-2-amino-1-butanol molecule is in a cationic form, and has a positively charged amino group. The tartaric acid molecule is a semi-tartrate ion, with a neutral carboxylic acid group at one end and a negatively charged carboxylate group at the other. The bond distances and angles in the cation and the anion are normal within experimental error.

In the crystal structure, an extensive hydrogen-bond network is built up (Fig. 2 and Table 1) in which every OH and NH H atom is used at least once as a hydrogen-bond donor.

Experimental

The title compound was prepared according to the procedure of Bai *et al.* (2004). Colourless single crystals of (I) were grown by the slow evaporation of a solution in methanol and dioxane.

Crystal data

$C_4H_{12}NO^+\cdot C_4H_5O_6^-$
M_r = 239.23
Orthorhombic, $P2_12_12_1$
a = 7.2959 (7) Å
b = 9.4843 (9) Å
c = 16.0204 (16) Å
V = 1108.56 (19) Å^3
Z = 4
D_x = 1.433 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3501 reflections
θ = 2.5–27.9°
μ = 0.13 mm^{-1}
T = 293 (2) K
Block, colourless
0.32 × 0.28 × 0.26 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.956, T_{max} = 0.968
7492 measured reflections
1551 independent reflections
1441 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 27.9°
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -21 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.083
S = 1.06
1551 reflections
171 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.0747P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7···O4^{i}	0.866 (10)	2.44 (2)	3.0647 (19)	130 (2)
O7—H7···O6^{i}	0.866 (10)	1.892 (14)	2.7010 (18)	155 (2)
N1—H1*C*···O3^{ii}	0.890 (10)	2.364 (19)	2.8901 (19)	117.9 (16)
N1—H1*C*···O1^{ii}	0.890 (10)	1.997 (11)	2.860 (2)	162.8 (18)
N1—H1*B*···O7^{ii}	0.886 (10)	1.930 (10)	2.809 (2)	171 (2)
N1—H1*A*···O5^{iii}	0.893 (10)	2.041 (12)	2.8970 (18)	160 (2)
O4—H4···O2^{iv}	0.824 (10)	2.30 (2)	2.9879 (19)	142 (3)
O3—H3···O6^{v}	0.853 (10)	1.831 (11)	2.6777 (18)	172 (2)
O2—H2···O5^{iv}	0.861 (10)	1.649 (11)	2.5081 (17)	175 (3)

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

All N—H and O—H H atoms were initially located in a difference Fourier map and were then refined with N—H restrained to 0.88 (1) Å and O—H restrained to 0.86 (1) Å. Other H atoms were positioned geometrically (C—H = 0.96–0.98 Å) and refined using riding constraints, with U_{iso}(H) = 1.2U_{eq}(C).

Due to the absence of significant anomalous dispersion effects, Friedel-pair reflections were merged prior to refinement. The correct absolute configuration was used; it is known from the hand of the (2*R*,3*R*)-tartaric acid that was used in the preparation of the salt.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

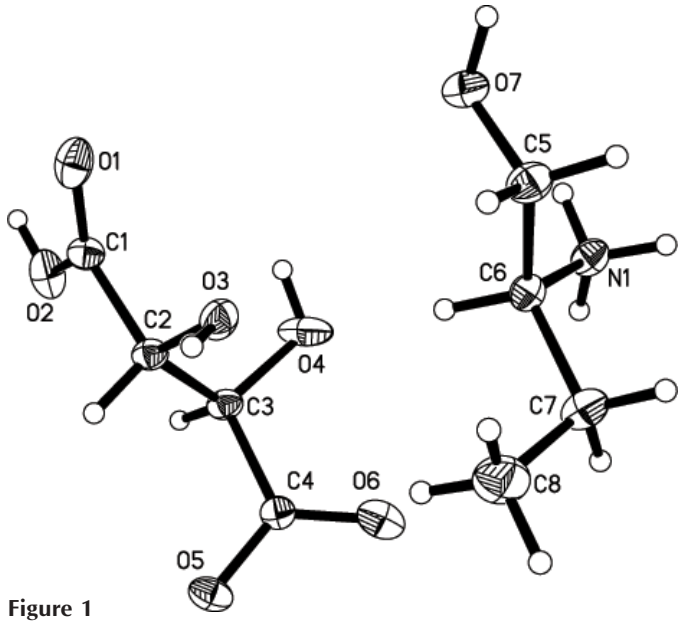


Figure 1
The molecular structure of (I), with the atom-numbering scheme and 30% probability atomic displacement ellipsoids.

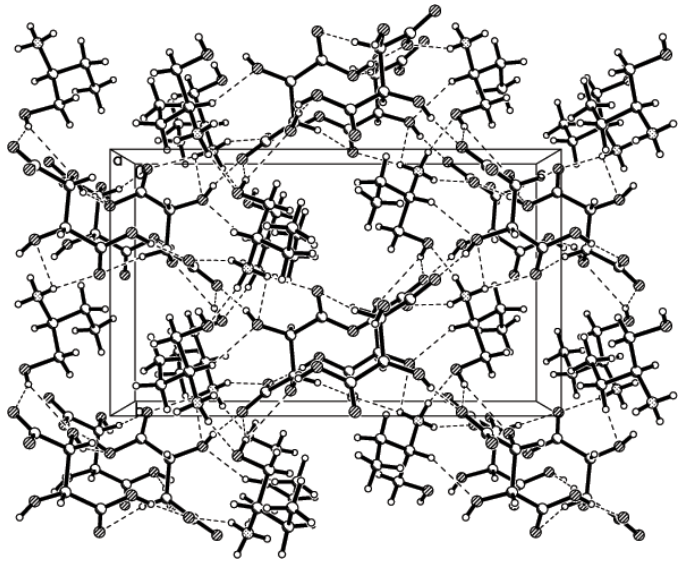


Figure 2
The packing of (I), viewed down the *a* axis, showing the hydrogen-bond interactions as dashed lines.

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