(*R*)-1-Hydroxybutan-2-ammonium (2*R*,3*R*)-tartrate monohydrate

Yue-Cheng Zhang,[a] Guo-Yi Bai,[b] Tao Zeng,[a] Jiang-Sheng Li[a] and Xi-Long Yan[a]*

[a]School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China, and [b]College of Chemistry and Environmental Science, Hebei University, Baoding 071002, People's Republic of China

Correspondence e-mail: yan@tju.edu.cn

In the formation of the title compound, $C_4H_{12}NO^+ \cdot C_4H_5O_6^- \cdot H_2O$, the (*R*)-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 (*R*)-(−)-2-amino-1-butanol. (*S*)-2-Amino-1-butanol is a key intermediate in the synthesis of ethambutol hydrochloride, which is an important antitubercular agent (Wilkinson *et al.*, 1961; Kazan *et al.*, 1973). Typically, most processes for the synthesis of 2-amino-1-butanol produce a racemate, and enantiomerically pure (*S*)-2-amino-1-butanol and (*R*)-2-amino-1-butanol were obtained through the resolution of racemic (*R*,*S*)-2-amino-1-butanol with (2*R*,3*R*)-tartaric acid (Singh, 1976; Bai *et al.*, 2004). (*S*)-2-Amino-1-butanol was used for the synthesis of ethambutol, and the (*R*)-2-amino-1-butanol was converted back to the racemate for recycling (Zhang *et al.*, 2005; Bai *et al.*, 2005). (*R*)-1-Hydroxybutan-2-ammonium (2*R*,3*R*)-tartrate monohydrate, (I), is a key intermediate in this process and its structure is reported here.

(I)
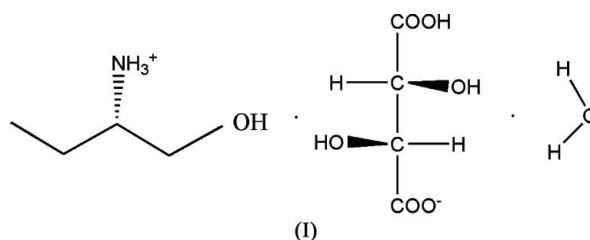
A view of (I) with the atom-numbering scheme is illustrated in Fig. 1. In this crystal structure, the (*R*)-2-amino-1-butanol molecule is in a cationic form, and has a positively charged amino group. The tartaric acid molecule is a semitartrate ion, with a neutral carboxylic acid group at one end and 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 formed (Fig. 2 and Table 1), to which water molecules make an important contribution.

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 water (4:1 *v*/*v*).

Crystal data

$C_4H_{12}NO^+ \cdot C_4H_5O_6^- \cdot H_2O$
$M_r = 257.24$
Orthorhombic, $P2_12_12_1$
$a = 7.1962\,(16)$ Å
$b = 10.532\,(2)$ Å
$c = 16.963\,(4)$ Å
$V = 1285.7\,(5)$ Å^3
$Z = 4$
$D_x = 1.329$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 2779
  reflections
$\theta = 2.3–25.7°$
$\mu = 0.12$ mm^{-1}
$T = 294\,(2)$ K
Block, colourless
$0.24 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector
  diffractometer
φ and ω scans
Absorption correction: multi-scan
  (SADABS; Sheldrick, 1996)
  $T_{min} = 0.972$, $T_{max} = 0.977$
7213 measured reflections

1530 independent reflections
1245 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 26.4°$
$h = -9 \rightarrow 8$
$k = -8 \rightarrow 13$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.117$
$S = 1.05$
1530 reflections
179 parameters
H atoms treated by a mixture of
  independent and constrained
  refinement

$w = 1/[\sigma^2(F_o^2) + (0.0689P)^2$
  $+ 0.2901P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2···O6^i	0.82	1.66	2.466 (3)	168
O3—H3···O7	0.84 (3)	1.85 (3)	2.671 (3)	165 (3)
O4—H4···O5	0.82 (4)	2.05 (4)	2.577 (3)	122 (3)
O4—H4···O8	0.82 (4)	2.48 (4)	2.994 (5)	121 (3)
O7—H7···O8	0.79 (5)	1.84 (5)	2.624 (4)	175 (5)
N1—H1A···O5^{ii}	0.92 (4)	1.87 (4)	2.787 (3)	174 (4)
N1—H1B···O4^{iii}	0.92 (4)	1.90 (4)	2.801 (3)	167 (3)
N1—H1C···O1	0.96 (4)	1.92 (4)	2.849 (3)	162 (4)
O8—H8D···O3^{iv}	0.83 (5)	1.87 (5)	2.692 (3)	171 (4)
O8—H8E···O1^v	0.84 (5)	1.92 (5)	2.738 (4)	167 (5)

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

All N—H and O—H H atoms were initially located in a difference Fourier map and were then refined, with O—H restrained to 0.79 (5)–0.84 (5) Å and N—H restrained to 0.92 (4)–0.96 (4) Å and with $U_{iso}(H) = 1.5U_{eq}(N,O)$, except that O2—H2 was constrained to 0.82 Å in a riding model. Other H atoms were positioned geometrically [C—H = 0.96–0.98 Å] and treated as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$. In the absence of significant anomalous dispersion effects, Friedel pairs were merged prior to refinement. The absolute configuration was assigned from that of the (2R,3R)-tartaric acid which 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:
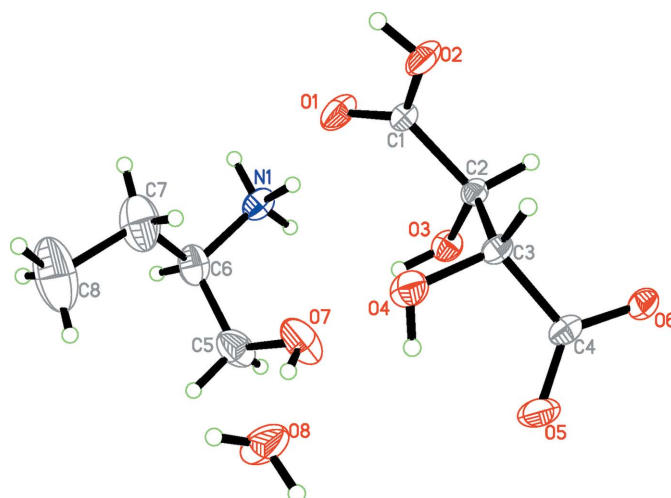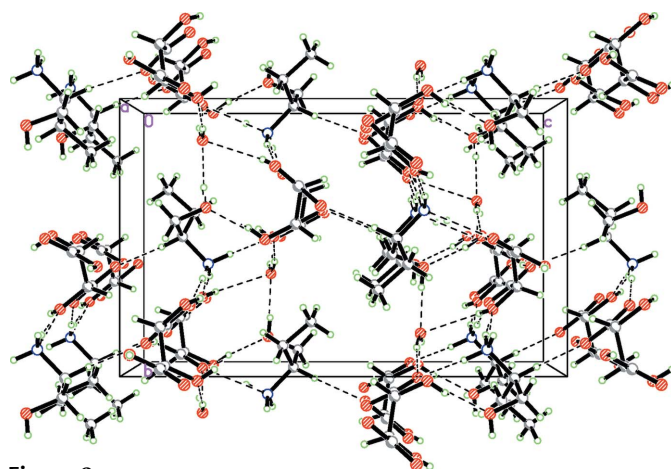

Figure 2
A packing diagram of (I). Dashed lines indicate hydrogen bonds.

SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

References

Bai, G. Y., Chen, L. G., Xing, P., Li, Y. & Yan, X. L. (2004). *Fine Chem.* **21**, 943–945.
Bai, G. Y., Chen, L. G., Zhang, Y. C., He, F., Li, Y., Yan, X. L. & Duan, X. M. (2005). *React. Kinet. Catal. Lett.* **86**, 203–209.
Bruker (1997). *SMART, SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kazan, J., Township, B. & County, S. (1973). US Patent No. 3 769 347.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Singh, B. (1976). US Patent No. 3 944 617.
Wilkinson, R. G., Shepherd, R. G., Thomas, J. P. & Baughn, C. (1961). *J. Am. Chem. Soc.* **83**, 2212–2213.
Zhang, Y. C., Chen, L. G., Bai, G. Y., Li, Y. & Yan, X. L. (2005). *J. Catal.* **236**, 176–180.