C18	0.1506 (3)	0.0918 (4)	0.33955 (8)	0.0724 (7)
C19	0.2270 (3)	0.0534 (3)	0.28979 (7)	0.0595 (5)
C20	0.3036 (3)	−0.1011 (3)	0.27981 (9)	0.0686 (6)
C21	0.3825 (3)	−0.1337 (3)	0.23287 (10)	0.0677 (6)
C22	0.3885 (2)	−0.0132 (2)	0.19209 (8)	0.0539 (5)
C23	0.1331 (3)	0.2341 (3)	0.12036 (9)	0.0615 (5)

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

N1—C2	1.388 (3)	C12—C13	1.515 (2)
N1—C22	1.394 (3)	C12—C23	1.538 (3)
C2—C11	1.380 (3)	C13—C22	1.374 (3)
C2—C3	1.422 (3)	C13—C14	1.433 (2)
C3—C4	1.350 (3)	C14—C15	1.418 (3)
C4—C5	1.414 (3)	C14—C19	1.426 (3)
C5—C6	1.414 (3)	C15—C16	1.364 (3)
C5—C10	1.430 (3)	C16—C17	1.397 (4)
C6—C7	1.356 (4)	C17—C18	1.355 (4)
C7—C8	1.398 (3)	C18—C19	1.413 (3)
C8—C9	1.357 (3)	C19—C20	1.412 (3)
C9—C10	1.426 (3)	C20—C21	1.350 (3)
C10—C11	1.418 (3)	C21—C22	1.412 (3)
C11—C12	1.517 (2)		
C2—N1—C22	120.6 (2)	C13—C12—C23	110.2 (2)
C11—C2—N1	120.0 (2)	C11—C12—C23	110.6 (1)
C2—C11—C12	119.4 (2)	C22—C13—C12	119.9 (2)
C13—C12—C11	110.9 (2)	C13—C22—N1	119.5 (2)

The H atoms were located from difference Fourier maps and refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976), *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93. Geometric calculations: *PARST* (Nardelli, 1983*b*).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1170). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hydrazinium L-Tartrate

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Abstract

The crystal structure of $N_2H_5^+.C_4H_5O_6^-$ consists of infinite chains of tartrate anions linked head-to-tail by O—H···O hydrogen bonds. Two such chains are cross-connected by O—H···O hydrogen bonds to form dimeric chains. The hydrazinium cations sit at the centre of four such tartrate dimers and bridge them by two-centre and three-centre N—H···O hydrogen bonds. As a whole, the structure is stabilized by numerous hydrogen bonds.

Comment

The salts of tartaric acid are becoming the latest interest among scientists of various disciplines owing to their applications in diversified fields such as chiral synthesis, crystal engineering, nonlinear optics, solid-state physics and wine-making technology (Aakeroy & Hitchcock, 1994; Zyss, Pecaut, Levy & Masse, 1993; Marshall & Luke, 1992; Aakeroy, Hitchcock & Seddon, 1992). Rare earth tartrates are used as precursors in the synthesis of high T_c superconductors (Garcia-Jaca *et al.*, 1994). Chiral tartaric acid is a good resolving agent; basic enantiomers with N atoms can easily be turned into salts and resolved *via* recrystallization. Single crystals of the title compound, (I), were obtained when we attempted to resolve the hydrazone from a spirodiacetone derivative.

(I)
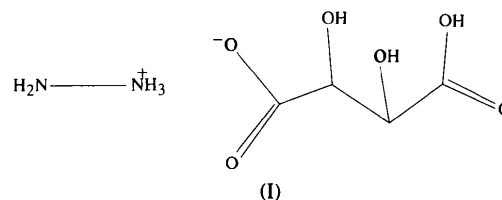
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The bond lengths and angles in both the hydrazinium and tartrate ions are normal (Table 2); the anionic backbone is planar with a C1—C2—C3—C4 torsion angle of 174.5 (2)°. Since all the H atoms were located from the difference Fourier synthesis and refined, the hydrogen-bonding scheme is worth discussing for this structure. The details of the hydrogen bonds are given in Table 3.

The terminal hydroxy group O5—H connects to O2 of the adjacent molecule *via* a strong hydrogen bond [O···O 2.471 (2) Å], thus forming a linear chain of tartrate anions in head-to-tail fashion. These chains are parallel to the crystallographic *a* axis. We note that the H atom involved here (H5O) has an extended O—H distance of 1.15 (2) Å and leans towards O2 of the next molecule. The other two hydroxy groups, O3—H and O4—H, in the tartrate ion form hydrogen bonds with O6 and O1, respectively, of the adjacent chain, leading to the formation of a pair of anionic tartrate chains. These two chains are screw-related along the *a* axis.

The N—N bond of the hydrazinium cation lies approximately in the *bc* plane along the *c* axis. Adjacent cations are positioned head-to-tail, which enables them to form a chain *via* N—H···N (N1—H···N2) hydrogen bonds along the *a* axis, running parallel to the anionic tartrate chains. These cations are surrounded by four of the tartrate dimers in the crystal lattice, which enables them to form multiple two-centre and three-centre N—H···O hydrogen bonds (Taylor, Kennard & Versichel, 1984) with the neighbouring tartrate anions.

An analysis of the crystal structures of organic salts of L-tartaric acid (33 structures) retrieved from the Cambridge Structural Database (Allen *et al.*, 1979) indicates that the tartrate anions always form head-to-tail linear chains parallel to any one of the crystallographic axes. This axis length is *ca* 7.5 (4) Å and all the structures crystallize in non-centrosymmetric space groups, which facilitates this group of compounds for nonlinear optical studies (Aakeroy, Hitchcock & Seddon, 1992; Watanabe, Noritake, Hirose, Okada & Kurauchi, 1994).
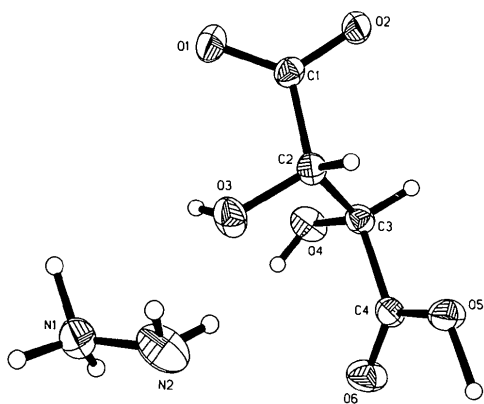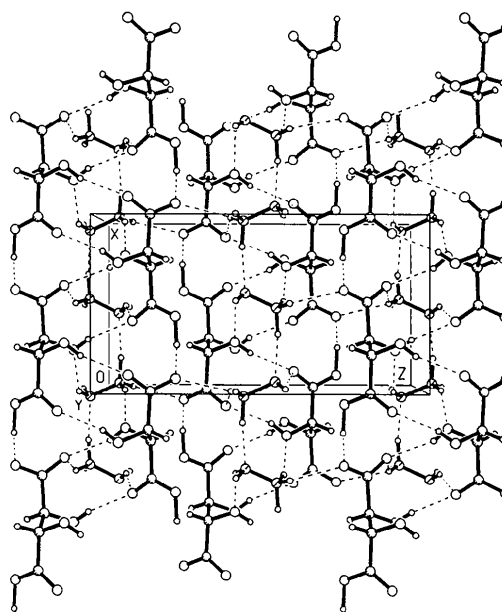

Fig. 2. Packing of the molecules viewed down the *b* axis. The hydrogen bonds are shown by dashed lines.

Experimental

Transparent crystals were obtained from an aqueous methanol solution by slow evaporation.

Crystal data

$N_2H_5^+.C_4H_5O_6^-$	Mo $K\alpha$ radiation
$M_r = 182.14$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 25
$P2_12_12_1$	reflections
$a = 7.037$ (1) Å	$\theta = 8-25°$
$b = 7.645$ (1) Å	$\mu = 0.158$ mm^{-1}
$c = 13.445$ (1) Å	$T = 293$ (2) K
$V = 723.31$ (15) Å^3	Transparent needle
$Z = 4$	$0.60 \times 0.36 \times 0.20$ mm
$D_x = 1.673$ Mg m^{-3}	Colourless

Data collection

Siemens *P4* four-circle	$R_{int} = 0.0147$
diffractometer	$\theta_{max} = 27.49°$
$\theta-2\theta$ scans	$h = -1 \rightarrow 9$
Absorption correction:	$k = -1 \rightarrow 9$
none	$l = -1 \rightarrow 17$
1379 measured reflections	3 standard reflections
1236 independent reflections	monitored every 100
1061 observed reflections	reflections
$[I > 2\sigma(I)]$	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.0314$	$\Delta\rho_{max} = 0.276$ e Å^{-3}
$wR(F^2) = 0.0811$	$\Delta\rho_{min} = -0.258$ e Å^{-3}
$S = 0.989$	Extinction correction: none

Fig. 1. The structure of hydrazinium L-tartrate showing 50% probability displacement ellipsoids and the atom-numbering scheme.

1236 reflections
149 parameters
All H-atom parameters
  refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors
from *International Tables*
for Crystallography (1992,
Vol. C, Tables 4.2.6.8 and
6.1.1.4)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1026). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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

	x	y	z	U_{eq}
C1	−0.5088 (2)	0.1460 (2)	0.67035 (11)	0.0211 (3)
C2	−0.2908 (2)	0.1438 (2)	0.66390 (12)	0.0208 (3)
C3	−0.2116 (2)	0.3294 (2)	0.66479 (11)	0.0207 (3)
C4	0.0057 (2)	0.3258 (2)	0.66823 (11)	0.0213 (3)
O1	−0.6036 (2)	0.0667 (2)	0.60748 (8)	0.0312 (3)
O2	−0.57617 (15)	0.2294 (2)	0.74456 (8)	0.0282 (3)
O3	−0.2276 (2)	0.0498 (2)	0.57938 (9)	0.0295 (3)
O4	−0.2781 (2)	0.42691 (15)	0.58258 (9)	0.0292 (3)
O5	0.07300 (15)	0.2416 (2)	0.74383 (8)	0.0286 (3)
O6	0.1013 (2)	0.4000 (2)	0.60540 (9)	0.0334 (3)
N1	−0.0252 (2)	−0.2130 (2)	0.45011 (12)	0.0374 (4)
N2	0.0594 (3)	−0.2336 (2)	0.54541 (12)	0.0384 (4)

Table 2. *Selected bond lengths (Å) and angles (°)*

C1—O1	1.236 (2)	C3—O4	1.413 (2)
C1—O2	1.275 (2)	C3—C4	1.530 (2)
C1—C2	1.537 (2)	C4—O6	1.220 (2)
C2—O3	1.416 (2)	C4—O5	1.293 (2)
C2—C3	1.524 (2)	N1—N2	1.422 (2)
O1—C1—O2	125.4 (1)	O4—C3—C2	111.4 (1)
O1—C1—C2	119.7 (2)	O4—C3—C4	111.4 (1)
O2—C1—C2	114.9 (1)	C2—C3—C4	110.4 (1)
O3—C2—C3	111.3 (1)	O6—C4—O5	125.0 (1)
O3—C2—C1	111.4 (1)	O6—C4—C3	121.5 (2)
C3—C2—C1	110.8 (1)	O5—C4—C3	113.5 (1)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5O···O2^i	1.15 (2)	1.33 (2)	2.471 (2)	175 (2)
O3—H3O···O6^{ii}	0.86 (2)	1.99 (2)	2.787 (2)	155 (2)
O4—H4O···O1^{iii}	0.82 (2)	2.08 (2)	2.835 (2)	154 (2)
N1—H1N1···O4^{iii}	0.92 (3)	2.02 (2)	2.828 (2)	146 (3)
N1—H2N1···O2^{iv}	0.92 (3)	2.39 (3)	2.857 (2)	112 (2)
N1—H2N1···O1^v	0.92 (3)	2.11 (3)	2.867 (2)	138 (2)
N1—H3N1···N2^{vi}	1.05 (3)	1.91 (3)	2.952 (2)	171 (3)
N2—H1N2···O3	0.85 (3)	2.29 (3)	2.997 (2)	141 (3)
N2—H2N2···O6^{vii}	0.85 (3)	2.24 (4)	2.930 (2)	140 (3)

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

Data collection: *XSCANS* (Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93. Geometric calculations: *PARST* (Nardelli, 1983).

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Coumarin 311

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Abstract

Coumarin 311, 7-(dimethylamino)-4-methyl-2*H*-1-benzo-pyran-2-one, $C_{12}H_{13}NO_2$, crystallizes in a triclinic crystal system with two molecules in the asymmetric unit. The molecules are planar and make an angle of 10.41 (2)° between each other. The molecules are stacked in layers parallel to (103).

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