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(*R*)-Histidinium (2*R*,3*R*)-tartrate

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.076
Data-to-parameter ratio = 7.3

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

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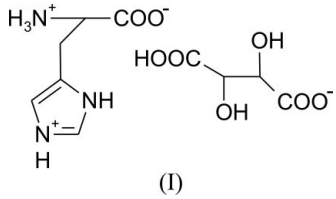
The crystal structure of (*R*)-histidinium (2*R*,3*R*)-tartrate, $C_6H_{10}N_3O_2^+\cdot C_4H_5O_6^-$, has been determined as part of an ongoing study into the fundamental effects of chirality on salt formation and hydration. Repeating layers of (*R*)-histidinium and (2*R*,3*R*)-tartrate interlink to form a three-dimensional network through simple translational symmetry of the unit cell.

Comment

This study was undertaken to identify the effects of chirality on the formation of salts, specifically the way chirality may affect hydration, as a result of interactions between a chiral drug and a chiral counterion. The absolute configuration can be considered determined as the chiral properties of the starting materials are well characterized. (*R*)-Histidine and (2*R*,3*R*)-tartaric acid samples were purchased from Fluka and used in the crystallization. The asymmetric unit and unit cell contain one histidine as a monocation (protonated at the amine and imidazole ring N atoms and deprotonated at the carboxylic acid group), and one tartrate as a monoanion (see scheme below and Fig. 1).



(I)

The (*R*)-histidinium layer is formed by chains of linked histidinium cations hydrogen bonded from the imidazole ring (N2) to carboxyl oxygen (O1). The two-dimensional layer is created by a hydrogen bond from N3 (ammonium group) to the adjacent (*R*)-histidinium carboxyl group (O2). The (2*R*,3*R*)-tartrate anions also form chains, with each link created by hydrogen bonding between the carboxyl OH (O3) group to an oxygen (O8) of the carboxylate group (Fig. 2). The (2*R*,3*R*)-tartrate two-dimensional layer is maintained by a single hydrogen bond from a hydroxyl group (O6) to a neighbouring carboxyl oxygen (O7). The three-dimensional crystal structure is formed of repeating layers of (*R*)-histidinium and (2*R*,3*R*)-tartrate ions, as shown in Fig. 2. The (*R*)-histidinium layer is linked to one (2*R*,3*R*)-tartrate layer through two hydrogen bonds from the ammonium group (N3) to O7 (carboxylate). The next layer of tartrates is hydrogen bonded to the imidazole ring (N1—H1···O5) and the carboxylate atom O1. Atom O1 is bifurcated, maintaining both the histidine chains and the tartrate/histidine interaction with O5.

Experimental

A saturated aqueous solution (5 ml) of (*R*)-histidine was mixed with a saturated aqueous solution (5 ml) of (2*R*,3*R*)-tartartic acid and the vial was covered with a pierced film. This was placed in a larger glass vial containing methanol (25 ml), sealed and allowed to stand for three weeks at room temperature. Crystals of a suitable size for use for single-crystal X-ray diffraction analysis were removed and mounted on glass fibres.

Crystal data

$C_6H_{10}N_3O_2^+\cdot C_4H_5O_6^-$
M_r = 305.25
Triclinic, *P*1
a = 5.3712 (18) Å
b = 7.637 (3) Å
c = 8.460 (3) Å
α = 72.025 (5)°
β = 73.872 (5)°
γ = 81.144 (5)°
V = 316.2 (2) Å^3
Z = 1
D_x = 1.602 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 1902 reflections
θ = 2.6–28.1°
μ = 0.14 mm^{-1}
T = 291 (2) K
Tablet, colourless
0.5 × 0.3 × 0.1 mm

Data collection

Bruker SMART APEX CCD diffractometer
Thin-slice ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.802, T_{max} = 0.990
2658 measured reflections
1407 independent reflections
1382 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 28.4°
h = −6 → 7
k = −10 → 10
l = −11 → 11

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.076
S = 1.07
1407 reflections
194 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 0.0492P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O8^{i}	0.86	1.90	2.760 (2)	173
N1—H1···O5^{i}	0.86	2.55	2.993 (3)	113
N2—H2*A*···O1^{ii}	0.86	2.09	2.810 (3)	142
N2—H2*A*···O6^{ii}	0.86	2.47	2.961 (3)	117
N3—H3*A*···O7^{iii}	0.89	1.91	2.799 (2)	176
N3—H3*C*···O2^{iv}	0.89	1.96	2.822 (3)	162
O5—H500···O1^{v}	0.82	1.95	2.741 (3)	163
O6—H600···O7^{iv}	0.82	2.08	2.744 (2)	138
O3—H300···O8^{ii}	0.82	1.72	2.533 (2)	172

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

All H atoms were positioned geometrically (N—H = 0.86–0.89, O—H = 0.82 and C—H = 0.93–0.98 Å) and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(N), $1.5U_{eq}$(O) and $1.2U_{eq}$(C). In the absence of significant anomalous dispersion effects Friedel pairs were merged prior to refinement.

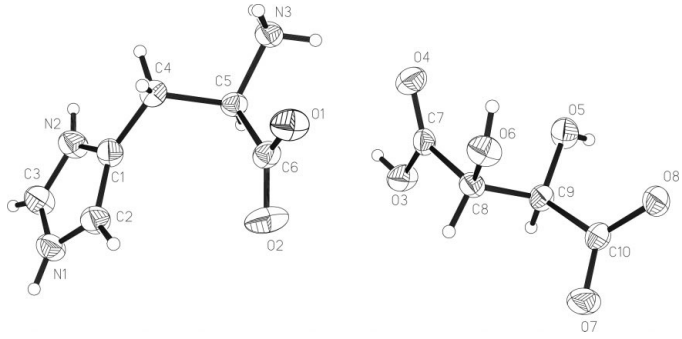


Figure 1
View of the title compound, with atomic displacement ellipsoids drawn at the 50% probability level.

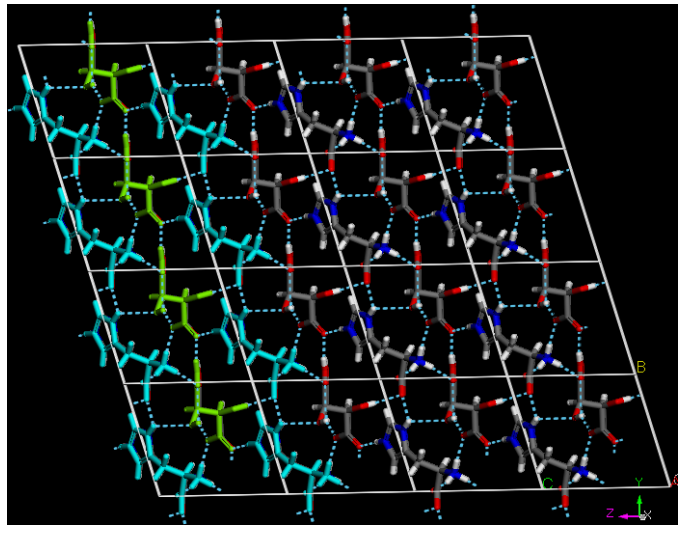


Figure 2
Hydrogen-bonding (dashed lines) motifs in the (*R*)-histidinium (blue) and (2*R*,3*R*)-tartrate (green) ions of (I).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1996) and *Materials Studio* (Accelrys, 2001); software used to prepare material for publication: *SHELXL*97.

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