

D-Histidinium (2*S*,3*S*)-tartrate

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## Key indicators

Single-crystal X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.029  
 $wR$  factor = 0.057  
Data-to-parameter ratio = 6.9For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The crystal structure of the title compound,  $\text{C}_6\text{H}_{10}\text{N}_3\text{O}_2^+ \cdot \text{C}_4\text{H}_5\text{O}_6^-$ , has been determined as part of an ongoing study into the fundamental effects of chirality on salt formation and hydration. This structure has distinct layers of histidine, which are linked by stacks of tartrates, forming a complex three-dimensional hydrogen-bond network.

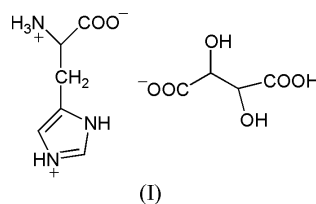
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## Comment

The present study was undertaken to identify the effects of chirality on the formation of salts, especially the way chirality may affect hydration, as a result of interactions between a chiral drug and a chiral counter-ion. D-Histidine and (2*S*,3*S*)-tartrate samples were purchased from Fluka and used in the crystallization, implying that the only possible final configuration is that presented here. The asymmetric unit of the title salt, (I), contains one histidine as a monocation (protonated at the amine and imidazole ring N atoms and deprotonated at the carboxylic acid), and one tartrate as a monoanion (Fig. 1).



Within the unit cell are chains of histidines linked by N—H...O [1.85 (2) Å] contacts from the imidazole ring to the carboxyl of the zwitterion (Fig. 2), similar to those described by Suresh & Vijayan (1987). These chains are linked across the *bc* plane by tartrate molecules (stacking down the *a* axis in Fig. 2), through the carboxylate and carboxyl groups. This layer, across the *bc* plane, is linked to successive layers by a tartrate stack. The tartrates link to each other *via* the OH groups, creating a long chain or stack perpendicular to that of the line of histidines. The tartrates act as hydrogen-bond crossroads within this structure, linking histidine chain to histidine chain along the *bc* plane and tartrate to tartrate to histidine down the *a* axis. This gives rise to a complex, but stable, three-dimensional packing arrangement.

## Experimental

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

## Crystal data

$C_6H_{10}N_3O_2^+ \cdot C_4H_5O_6^-$   
 $M_r = 305.25$   
 Orthorhombic,  $P2_12_12_1$   
 $a = 5.0207$  (9) Å  
 $b = 12.109$  (2) Å  
 $c = 20.797$  (4) Å  
 $V = 1264.4$  (4) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.604$  Mg m<sup>-3</sup>

## Data collection

Bruker SMART APEX CCD  
 diffractometer  
 Thin-slice  $\omega$  scans  
 Absorption correction: multi-scan  
 (Blessing, 1995)  
 $T_{\min} = 0.941$ ,  $T_{\max} = 0.998$   
 6322 measured reflections

Mo  $K\alpha$  radiation  
 Cell parameters from 2110  
 reflections  
 $\theta = 2.6$ – $22.4^\circ$   
 $\mu = 0.14$  mm<sup>-1</sup>  
 $T = 295$  (2) K  
 Needle, colourless  
 $0.40 \times 0.10 \times 0.05$  mm

1334 independent reflections  
 1117 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.044$   
 $\theta_{\text{max}} = 25.1^\circ$   
 $h = -3 \rightarrow 5$   
 $k = -13 \rightarrow 14$   
 $l = -23 \rightarrow 24$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.057$   
 $S = 0.91$   
 1334 reflections  
 194 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.019P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.17$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.13$  e Å<sup>-3</sup>

Table 1

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1 \cdots O2^i$	0.86	1.89	2.747 (3)	173
$N2-H2A \cdots O7^{ii}$	0.86	1.83	2.683 (3)	171
$N3-H3B \cdots O8^{ii}$	0.89	1.87	2.743 (3)	167
$N3-H3C \cdots O6^{iii}$	0.89	2.06	2.901 (3)	157
$N3-H3C \cdots O8^{iii}$	0.89	2.39	3.004 (3)	127
$O5-H5A \cdots O4^{iv}$	0.82	2.00	2.713 (2)	145
$O5-H5A \cdots O5^{iv}$	0.82	2.44	3.136 (2)	143
$O6-H6 \cdots O7^v$	0.82	1.95	2.754 (2)	167
$O3-H3D \cdots O2$	0.82	1.75	2.565 (2)	178

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

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

Data collection: SMART (Siemens, 1994); cell refinement: SAINT (Siemens, 1994); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP (Bruker, 1998) and Materials Studio (Accelrys, 2001); software used to prepare material for publication: SHELXL97.

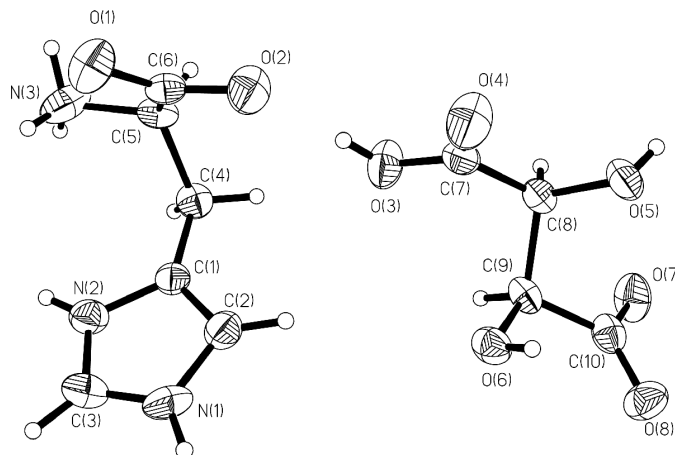


Figure 1

View of the title compound, (I), with displacement ellipsoids drawn at the 50% probability level.

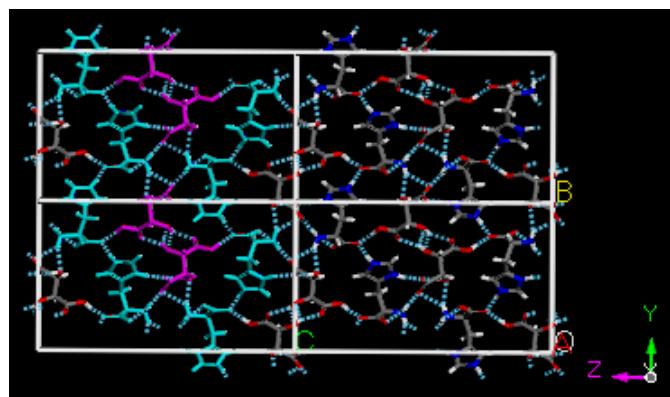


Figure 2

Hydrogen-bonding motifs for D-histidine (blue) and (2S,3S)-tartrate (pink).

## References

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