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# DL-Histidine DL-tartrate

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**Key indicators**

Single-crystal X-ray study
*T* = 295 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.043
*wR* factor = 0.118
Data-to-parameter ratio = 15.3

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



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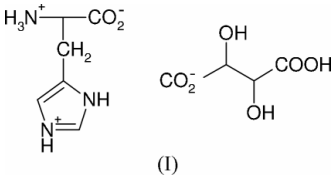
The crystal structure of DL-histidine DL-tartrate, $C_6H_{10}N_3O_2^+\cdot C_4H_5O_6^-$, has been determined as part of an ongoing study of the fundamental effects of chirality on salt formation and hydrates. Discrete single-enantiomer chains of histidine are linked in two dimensions by hydrogen bonds to a racemic pair of tartrate molecules.

## 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 counter-ion. DL-Histidine and DL-tartrate samples were purchased from Fluka and used in the crystallization. The asymmetric unit of the title compound, (I), contains one molecule of histidine as a monocation (protonated at the amine and imidazole N atoms and deprotonated at the carboxylic acid) and the tartrate as a monoanion (Fig. 1).




(I)

The histidines form chains of single enantiomers (Fig. 2) linked along the *b* axis by hydrogen bonds from the NH group of the imidazole ring to a carboxyl O atom of the next histidine, similar to those described by Suresh & Vijayan (1987). The tartrate anions form dimers containing one D- and one L-tartrate ion in each pair (Fig. 2). The dimers are formed by means of a carboxylic acid O atom bonding to a neighbouring tartrate utilizing a side OH group [2.817 (2) Å]. Each histidine molecule in a chain is linked to the next chain below (viewed down the *a* axis in Fig. 2) by a single hydrogen bond from a carboxyl O atom to an NH group of the ammonium group [2.749 (2) Å]. The tartrates link the chains of histidine in two dimensions to create a three-dimensional hydrogen-bond network.

## Experimental

A 5 ml saturated aqueous solution of DL-histidine was mixed with a 5 ml saturated aqueous solution of DL-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 three weeks at room temperature.

*Crystal data*

$C_6H_{10}N_3O_2^+\cdot C_4H_5O_6^-$
$M_r$ = 305.25
Monoclinic, $P2_1/c$
$a$ = 4.9695 (5) Å
$b$ = 13.4392 (12) Å
$c$ = 19.2749 (18) Å
$\beta$ = 90.253 (2)°
$V$ = 1287.3 (2) Å$^3$
$Z$ = 4
$D_x$ = 1.575 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 2967 reflections
$\theta$ = 1.9–28.0°
$\mu$ = 0.14 mm$^{-1}$
$T$ = 295 (2) K
Needle, colourless
0.50 × 0.10 × 0.10 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Thin-slice $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997; Blessing, 1995)
$T_{min}$ = 0.843, $T_{max}$ = 0.990
7512 measured reflections
2967 independent reflections
2207 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023
$\theta_{max}$ = 28.0°
$h = -6 \rightarrow 6$
$k = -17 \rightarrow 17$
$l = -25 \rightarrow 25$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.118
$S$ = 1.02
2967 reflections
194 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0666P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.44 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$A$···O8 | 0.86 | 1.93 | 2.7689 (19) | 166 |
| N1—H1···O2$^{i}$ | 0.86 | 1.84 | 2.6871 (19) | 169 |
| N3—H3$A$···O2$^{ii}$ | 0.89 | 1.87 | 2.7532 (18) | 173 |
| N3—H3$B$···O7$^{iii}$ | 0.89 | 2.09 | 2.7937 (18) | 135 |
| N3—H3$B$···O6$^{iii}$ | 0.89 | 2.35 | 3.1374 (18) | 147 |
| N3—H3$C$···O7$^{ii}$ | 0.89 | 1.85 | 2.7178 (18) | 164 |
| O3—H3$D$···O1$^{iv}$ | 0.82 | 1.77 | 2.5856 (17) | 173 |
| O5—H5$A$···O4$^{v}$ | 0.82 | 2.11 | 2.8174 (19) | 145 |
| O5—H5$A$···O4 | 0.82 | 2.30 | 2.7015 (19) | 111 |
| O6—H6···O8$^{vi}$ | 0.82 | 1.92 | 2.7152 (18) | 162 |

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

The unit-cell dimensions and angles were compared to those reported for the parent histidine enantiomers by Edington & Harding (1974) and Madden *et al.* (1972). All H atoms were placed geometrically [C—H = 0.93–0.98, N—H = 0.86–0.89 and O—H = 0.82 Å; $U_{iso}$(H) = 1.2 or 1.5 times $U_{eq}$(parent atom)] and refined using a riding model.

Data collection: *SMART* (Siemens, 1994); cell refinement: *SAINT* (Siemens, 1994); 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: *ORTEP*-3 (Farrugia, 1997) and *Materials Studio* (Accelrys, 2001); software used to prepare material for publication: *SHELXL*97.

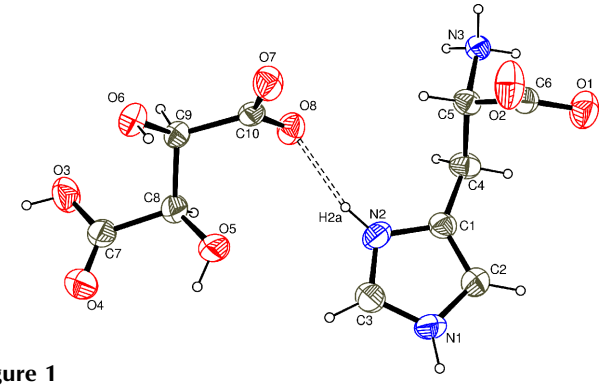



**Figure 1**
*ORTEP*-3 (Farrugia, 1997) plot of the asymmetric unit of (I) ($Z$ = 4), with displacement ellipsoids drawn at the 50% probability level.

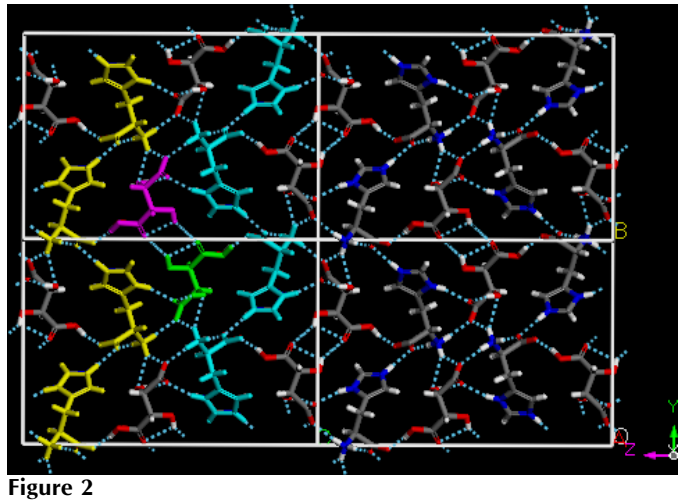


**Figure 2**
Hydrogen-bonding motifs for D-tartrate (green), L-histidine (yellow), D-histidine (blue) and L-tartrate (pink).

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