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L-Histidinium iodide

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Keywords: crystal structure; L-histidine; zinc metabolism; antimicrobial activity; intermolecular hydrogen bonds.

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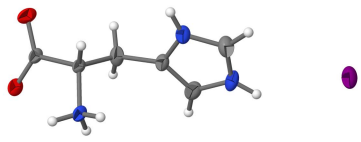
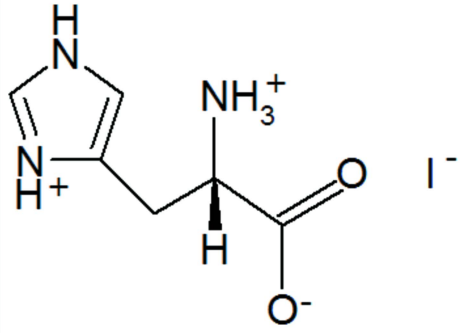
Structural data: full structural data are available from iucrdata.iucr.org

In the title salt, $C_6H_{10}N_3O_2^+\cdot I^-$, the cation is protonated at the imidazole ring and the amine group and deprotonated at the carboxylate group. The crystal packing features N—H···O and N—H···I hydrogen bonds.

3D view



Chemical scheme



Structure description

L-Histidine derivatives plays a major role in zinc metabolism, as a zinc binding moiety in serum (Casella & Gullotti, 1983), and these derivatives exhibit antimicrobial activity (Garza-Ortiz *et al.*, 2013). The title compound comprises a protonated L-histidine cation and an iodide anion (Fig. 1). The geometric parameters of the title cation agree well with those reported for similar structures (Gokul Raj *et al.*, 2006; Johnson & Feeder, 2004). The crystal packing (Fig. 2) is controlled by N—H···O and N—H···I hydrogen bonds (Table 1). The title compound is isostructural with the bromide and chloride salts.

Synthesis and crystallization

L-histidine (15.0 g, 0.0966 mol) and hydriodic acid (13.2150 ml mol^{-1}) were dissolved in 50 ml of double-distilled water and stirred at 293 K for 4 h. The solution was filtered and allowed to dry at room temperature by slow evaporation. After 30 d, pale-yello block-shaped crystals were obtained in a yield of 95% (m.p. 370 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

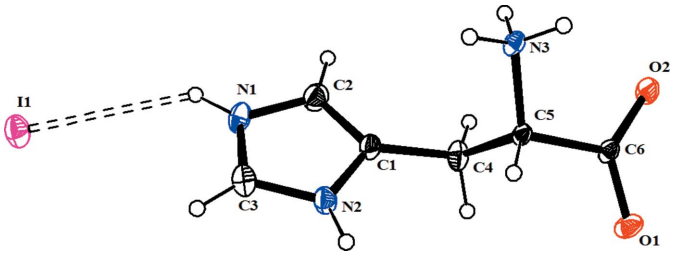


Figure 1
The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids for the non-H atoms.

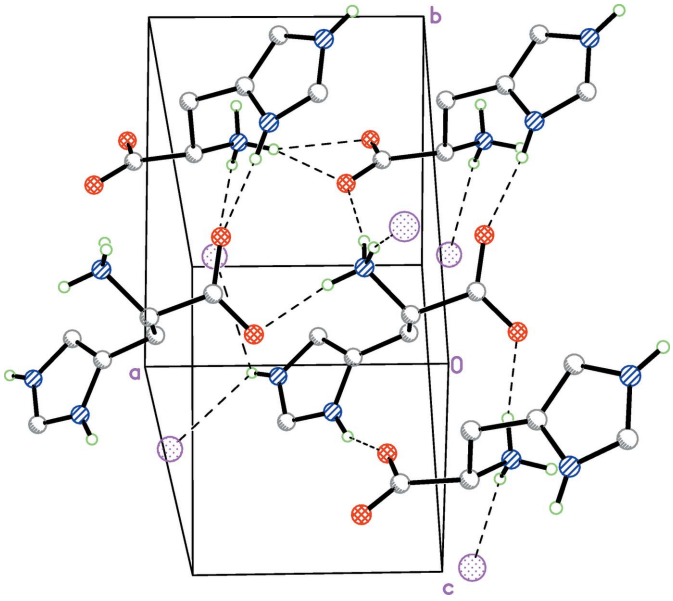


Figure 2
Packing diagram of the title compound. Hydrogen bonds are shown as dashed lines. H atoms bonded to C atoms have been omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···I1	0.86	2.91	3.546 (3)	133
N2—H2A···O2^{i}	0.86	1.91	2.693 (5)	151
N3—H3A···O1^{ii}	0.89	1.94	2.819 (3)	168
N3—H3B···I1^{iii}	0.89	2.70	3.546 (2)	160
N3—H3C···O1^{iv}	0.89	1.98	2.855 (3)	167
N3—H3C···O2^{iv}	0.89	2.56	3.214 (3)	131

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_6H_{10}N_3O_2^+\cdot I^-$
M_r	283.07
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	296
a, b, c (Å)	5.7363 (2), 8.2696 (3), 10.0169 (4)
β (°)	94.314 (1)
V (Å^3)	473.82 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.35
Crystal size (mm)	0.10 × 0.10 × 0.05
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.731, 0.851
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9180, 3569, 3231
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.770
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.022, 0.082, 1.20
No. of reflections	3569
No. of parameters	110
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.87
Absolute structure	Flack (1983), with 1725 Friedel pairs
Absolute structure parameter	0.05 (3)

Computer programs: *APEX2* (Bruker, 2008), *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Acknowledgements

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full crystallographic data

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Crystal data

$C_6H_{10}N_3O_2^+\cdot I^-$
M_r = 283.07
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 5.7363 (2) Å
b = 8.2696 (3) Å
c = 10.0169 (4) Å
β = 94.314 (1)°
V = 473.82 (3) Å^3
Z = 2
$F(000)$ = 272
D_x = 1.984 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3626 reflections
θ = 3.2–33.1°
μ = 3.35 mm^{-1}
T = 296 K
Block, pale yellow
0.10 × 0.10 × 0.05 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.731, T_{max} = 0.851
9180 measured reflections
3569 independent reflections
3231 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 33.2°, θ_{min} = 3.2°
h = −8→8
k = −12→12
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.082
S = 1.20
3569 reflections
110 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.0342P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}
Absolute structure: Flack (1983), with 1725 Friedel pairs
Absolute structure parameter: 0.05 (3)

Special details

Refinement. H atoms were positioned geometrically and refined using riding model with C-H = 0.97Å and U_{iso}(H) = 1.2Ueq(C) for C-H_2, C-H = 0.93Å and U_{iso}(H) = 1.2Ueq(C) for aromatic C-H, N-H = 0.89Å and U_{iso}(H) = 1.5Ueq(C) for N-H3, and N-H = 0.86Å and U_{iso}(H) = 1.2Ueq(C) for N-H.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	1.11212 (3)	0.45507 (5)	1.108570 (15)	0.03755 (7)
O1	−0.3126 (3)	0.4108 (3)	0.4822 (3)	0.0346 (5)
O2	−0.2046 (4)	0.6389 (3)	0.3852 (3)	0.0316 (4)
N1	0.6300 (6)	0.5429 (4)	0.8774 (3)	0.0322 (6)
H1A	0.7417	0.5871	0.9266	0.039*
N2	0.4093 (5)	0.3651 (3)	0.7777 (3)	0.0279 (5)
H2A	0.3535	0.2730	0.7512	0.033*
C4	0.0921 (5)	0.5337 (4)	0.6622 (3)	0.0297 (6)
H4A	0.0461	0.6462	0.6679	0.036*
H4B	−0.0251	0.4698	0.7032	0.036*
N3	0.2571 (3)	0.5839 (3)	0.4421 (2)	0.0221 (4)
H3A	0.2532	0.6863	0.4691	0.033*
H3B	0.2195	0.5790	0.3544	0.033*
H3C	0.4003	0.5442	0.4600	0.033*
C3	0.5991 (7)	0.3871 (5)	0.8599 (4)	0.0346 (7)
H3	0.6940	0.3059	0.8984	0.042*
C1	0.3171 (5)	0.5127 (4)	0.7421 (3)	0.0262 (5)
C5	0.0875 (4)	0.4873 (2)	0.5136 (2)	0.0193 (5)
H5	0.1272	0.3725	0.5068	0.023*
C6	−0.1639 (4)	0.5135 (3)	0.4518 (3)	0.0216 (4)
C2	0.4567 (6)	0.6230 (4)	0.8052 (4)	0.0338 (6)
H2	0.4381	0.7346	0.8003	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.04010 (10)	0.03940 (10)	0.03123 (9)	0.00380 (11)	−0.01003 (6)	−0.00188 (11)
O1	0.0164 (8)	0.0278 (10)	0.0587 (14)	−0.0016 (6)	−0.0025 (8)	0.0096 (9)
O2	0.0230 (9)	0.0252 (9)	0.0444 (12)	0.0030 (7)	−0.0104 (8)	0.0063 (9)
N1	0.0295 (12)	0.0385 (15)	0.0272 (13)	−0.0035 (11)	−0.0077 (10)	−0.0098 (11)
N2	0.0307 (12)	0.0248 (11)	0.0270 (11)	0.0017 (9)	−0.0059 (10)	−0.0021 (9)
C4	0.0219 (11)	0.0428 (16)	0.0239 (12)	0.0047 (11)	−0.0012 (9)	−0.0031 (11)
N3	0.0139 (8)	0.0241 (9)	0.0280 (10)	0.0007 (7)	−0.0007 (7)	−0.0019 (8)
C3	0.0324 (16)	0.0397 (17)	0.0307 (16)	0.0052 (14)	−0.0055 (12)	−0.0001 (13)
C1	0.0255 (11)	0.0304 (12)	0.0220 (11)	−0.0026 (10)	−0.0033 (9)	−0.0016 (9)
C5	0.0133 (8)	0.0181 (13)	0.0260 (10)	0.0014 (6)	−0.0025 (7)	0.0013 (7)
C6	0.0152 (9)	0.0188 (9)	0.0302 (12)	0.0019 (8)	−0.0029 (8)	−0.0012 (9)
C2	0.0323 (14)	0.0323 (15)	0.0360 (15)	−0.0033 (12)	−0.0030 (11)	−0.0036 (12)

Geometric parameters (Å, º)

O1—C6	1.258 (3)	C4—H4B	0.9700
O2—C6	1.245 (3)	N3—C5	1.484 (3)
N1—C3	1.310 (5)	N3—H3A	0.8900
N1—C2	1.357 (5)	N3—H3B	0.8900

N1—H1A	0.8600	N3—H3C	0.8900
N2—C3	1.327 (5)	C3—H3	0.9300
N2—C1	1.367 (4)	C1—C2	1.340 (5)
N2—H2A	0.8600	C5—C6	1.542 (3)
C4—C1	1.477 (4)	C5—H5	0.9800
C4—C5	1.535 (4)	C6—O2	1.245 (3)
C4—H4A	0.9700	C2—H2	0.9300
C3—N1—C2	108.8 (3)	N1—C3—H3	125.8
C3—N1—H1A	125.6	N2—C3—H3	125.8
C2—N1—H1A	125.6	C2—C1—N2	106.2 (3)
C3—N2—C1	108.8 (3)	C2—C1—C4	129.9 (3)
C3—N2—H2A	125.6	N2—C1—C4	123.5 (3)
C1—N2—H2A	125.6	N3—C5—C4	111.7 (2)
C1—C4—C5	116.5 (2)	N3—C5—C6	110.96 (19)
C1—C4—H4A	108.2	C4—C5—C6	107.6 (2)
C5—C4—H4A	108.2	N3—C5—H5	108.9
C1—C4—H4B	108.2	C4—C5—H5	108.9
C5—C4—H4B	108.2	C6—C5—H5	108.9
H4A—C4—H4B	107.3	O2—C6—O1	126.1 (2)
C5—N3—H3A	109.5	O2—C6—O1	126.1 (2)
C5—N3—H3B	109.5	O2—C6—C5	117.7 (2)
H3A—N3—H3B	109.5	O2—C6—C5	117.7 (2)
C5—N3—H3C	109.5	O1—C6—C5	116.0 (2)
H3A—N3—H3C	109.5	C1—C2—N1	107.8 (3)
H3B—N3—H3C	109.5	C1—C2—H2	126.1
N1—C3—N2	108.3 (3)	N1—C2—H2	126.1
C2—N1—C3—N2	0.0 (5)	N3—C5—C6—O2	−21.5 (3)
C1—N2—C3—N1	0.0 (5)	C4—C5—C6—O2	100.9 (3)
C3—N2—C1—C2	0.0 (4)	N3—C5—C6—O2	−21.5 (3)
C3—N2—C1—C4	−173.7 (3)	C4—C5—C6—O2	100.9 (3)
C5—C4—C1—C2	116.5 (4)	N3—C5—C6—O1	163.6 (2)
C5—C4—C1—N2	−71.3 (4)	C4—C5—C6—O1	−74.0 (3)
C1—C4—C5—N3	−59.8 (3)	N2—C1—C2—N1	0.0 (4)
C1—C4—C5—C6	178.2 (3)	C4—C1—C2—N1	173.2 (3)
O2—O2—C6—O1	0.0 (5)	C3—N1—C2—C1	0.0 (5)
O2—O2—C6—C5	0.0 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···I1	0.86	2.91	3.546 (3)	133
N2—H2*A*···O2^{i}	0.86	1.91	2.693 (5)	151
N3—H3*A*···O1^{ii}	0.89	1.94	2.819 (3)	168
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N3—H3*C*···O1^{iv}	0.89	1.98	2.855 (3)	167
N3—H3*C*···O2^{iv}	0.89	2.56	3.214 (3)	131

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1; (ii) −*x*, *y*+1/2, −*z*+1; (iii) *x*−1, *y*, *z*−1; (iv) *x*+1, *y*, *z*.