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(*S*)-2-Hydroxy-3-(1*H*-imidazol-5-yl)-propanoic acid hydrate

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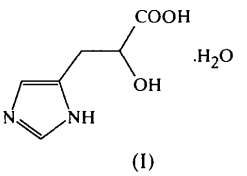
Abstract

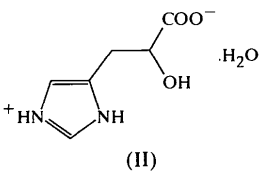
(*S*)-2-Hydroxy-3-(1*H*-imidazol-5-yl)propanoic acid hydrate, $C_6H_8N_2O_3\cdot H_2O$, has a fully extended lactic acid side chain with a *trans* conformation, which is oriented nearly perpendicular to the imidazole plane. The imidazole ring is protonated, and the carboxylate group deprotonated, to give a zwitterionic structure. The molecules are held together by intermolecular hydrogen bonds between the carboxylate, imino and hydroxyl groups, and the water molecules.

Comment

The title compound, (I) is a well known final product of L-histidine catabolism. Patients with liver cirrhosis or histidinemia have high urinary values of (I) (Dubovsky & Dubovska, 1965; Murray *et al.*, 1993). It also has an inhibitory action on cholinesterase and monoamine oxidase (Kurocochi *et al.*, 1956). Accordingly, an accurate crystal structure determination is important for the elucidation of its catabolic pathway and physiological action.



(I)



(II)

The molecular structure of (I) is shown in Fig. 1, with the atomic labelling scheme. The molecule is in a zwitterionic form, *i.e.* (II) rather than (I). The carboxylate group is negatively charged, as evidenced by the similar C—O distances, as well as by the lack of an H atom, and the imidazole ring is positively charged, as evidenced by two imino H atoms. The conformation may be compared with that of histidine hydrochloride (Donohue *et al.*, 1956), because the only difference between the title compound and histidine is the substitution of OH in (I) for the α-amino group in histidine. The chief conformational change involves rotations around the C4—C6 and C6—C7 bonds. The ionized lactic acid side chain of the title compound is fully extended, with a *trans* conformation [C4—C6—C7—C8 −175.2 (2)°], and it is oriented nearly perpendicular to the protonated imidazole plane [C5—C4—C6—C7 92.1 (4)°]. The imidazole ring and the carboxylate group are sited in an *anti* form. The imidazole ring of histidine hydrochloride is also protonated; its side chain is oriented nearly perpendicular, as in (I), but its carboxylate and imidazole groups are in a *cis* form. The other main differences between these compounds are in the bond angles around the asymmetric C atoms. For instance, the O3—C7—C8 angle [112.1 (2)°] of the title compound is larger than that of the corresponding N—C—C angle of 108.4° in histidine. This difference may be caused by the attractive force between the positively charged amino and the negatively charged carboxylate groups of histidine. As a result of the differences of the substituted groups (hydroxyl or amino) at the asymmetric C atom, the C6—C7—C8 angle [108.5 (2)°] of the title compound is smaller than the corresponding value of 115.0° in histidine. No stacking interactions between imidazole rings are observed. The crystal structure is stabilized by hydrogen bonds between the carboxylate, imino and hydroxyl groups and the water molecules, as shown in Table 2.

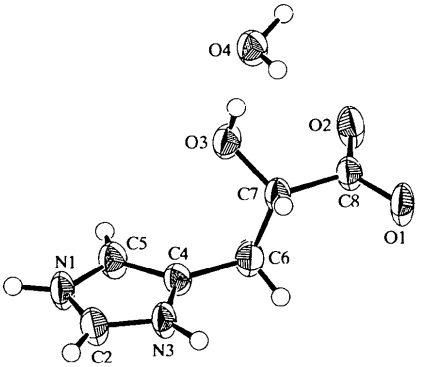


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound, with the atom-numbering scheme for non-H atoms: displacement ellipsoids correspond to 50% probability. H atoms are shown as circles of an arbitrary radius.

Experimental

A commercial sample of the title compound (Sigma Chemicals) was recrystallized from aqueous solution by slow evaporation of the solvent at room temperature.

Crystal data

$C_6H_8N_2O_3 \cdot H_2O$	Mo $K\alpha$ radiation
M_r = 174.156	λ = 0.71069 Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	θ = 18.25–22.15°
a = 7.756 (6) Å	μ = 0.113 mm^{-1}
b = 17.107 (2) Å	T = 296 K
c = 6.090 (2) Å	Plate
V = 808.1 (6) $Å^3$	0.4 × 0.3 × 0.1 mm
Z = 4	Colourless
D_x = 1.431 Mg m^{-3}	
D_m not measured	

Data collection

Rigaku AFC-5*R* diffractometer	1100 reflections with $I > 0$
$\omega/2\theta$ scans	θ_{max} = 27.5°
Absorption correction: ψ-scan (North *et al.*, 1968)	$h = 0 \rightarrow 9$
	$k = 0 \rightarrow 22$
T_{min} = 0.97, T_{max} = 1.00	$l = 0 \rightarrow 7$
1120 measured reflections	3 standard reflections
1120 independent reflections	every 150 reflections
	intensity decay: 0.73%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R(F)$ = 0.060	$\Delta\rho_{max}$ = 0.38 e $Å^{-3}$
$wR(F^2)$ = 0.107	$\Delta\rho_{min}$ = −0.20 e $Å^{-3}$
S = 1.44	Extinction correction: none
1100 reflections	Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
117 parameters	
H atoms: see below	
$w = 4F_o^2/\sigma^2(F_o^2)$	

Table 1. *Selected bond lengths (Å)*

O1—C8	1.244 (3)	N3—C4	1.366 (4)
O2—C8	1.246 (3)	C4—C5	1.357 (4)
O3—C7	1.410 (3)	C4—C6	1.488 (4)
N1—C2	1.314 (4)	C6—C7	1.533 (4)
N1—C5	1.364 (4)	C7—C8	1.541 (4)
N3—C2	1.330 (3)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.947	1.818	2.677 (3)	149.4 (2)
N3—H3···O2^{ii}	0.949	1.780	2.716 (3)	169.0 (2)
O3—H8···O4	0.925	1.812	2.734 (4)	174.6 (1)
O4—H9···O1^{iii}	0.98 (3)	1.79 (3)	2.751 (3)	166 (3)
O4—H10···O2^{iv}	0.79 (4)	1.97 (4)	2.760 (3)	176 (4)

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

H atoms were fixed, except for those on H_2O, which were freely refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1254). Services for accessing these data are described at the back of the journal.

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A 1:1 complex of 4-nitropyridine *N*-oxide and 3-hydroxybenzoic acid

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Abstract

The title molecular complex, $C_5H_4N_2O_3 \cdot C_7H_6O_3$, is hydrogen bonded, the molecules being linked by an O—H···O hydrogen bond between the phenol hydroxyl group and the *N*-oxide O atom [O···O 2.642 (2) Å].