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(2*S*)-2-(3-Benzoylthioureido)-3-(1*H*-imidazol-4-yl)propionic acid

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The title compound, (2*S*)-2-(3-benzoylthioureido)-3-(1*H*-imidazolium-4-yl)propionate, $C_{14}H_{14}N_4O_3S$, is zwitterionic with protonated positively charged imidazoyl and deprotonated negatively charged carboxylate groups. The intermolecular hydrogen bonds, C—H···O and N—H···O, involve all the O atoms with the H atoms from the amine and imidazoylium, forming a two-dimensional network parallel to the (100) plane.

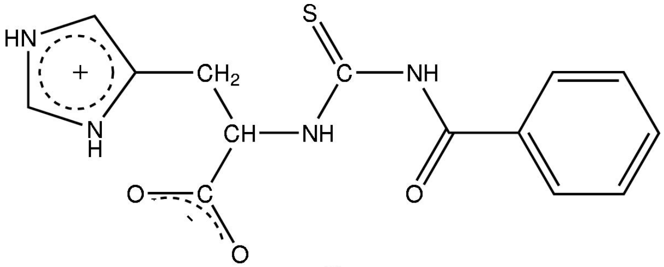
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.039
wR factor = 0.097
Data-to-parameter ratio = 8.4

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

Comment

L-Histidine is known to exist as a zwitterion with protonated amino and negatively charged carboxylate groups (Edington & Harding, 1974 Madden *et al.*, 1972). In some histidine salts, such as L-histidinium dinitrate, the histidine moiety acts as dication with protonated α-amino and imidazoyl groups and the nitrate counter-anions (Benali-Cherif *et al.*, 2002). The title compound, (I), is a neutral thiourea derivative of L-histidine, which exists as a zwitterion with protonated positively charged imidazoyl and deprotonated negatively charged carboxylate groups. The bond lengths of the imidazolium group (Table 1) are shorter than those in L-(+)-histidine (Madden *et al.*, 1972) and DL-histidine (Edington & Harding, 1974), indicating strong delocalization of electrons.



(I)

The benzoyl and 3-(1*H*-imidazol-4-yl)propionic acid groups are at *trans* and *cis* positions with respect to the thiono C═S group across the C8—N1 and C8—N2 bonds, respectively. The structural geometry of the benzoylthiourea moiety is normal (Allen *et al.*, 1987) and in agreement with 3-(3-benzoylthiouredo)propionic acid (Yusof & Yamin, 2003). The phenyl ring (C1–C6), the central thiourea group (S1/N1/N2/C7/C8/C9) [maximum deviation 0.031 (2)° for atom N1] and the imidazoylium group (C11/C12/N3/C13/N4/C14) [maximum deviation 0.025 (3)° for atom C11] are each planar. The central thiourea S1/N1/N2/C7/C8/C9 fragment makes dihedral angles with the phenyl and the imidazolium (C11/C12/N3/C13/N4/C14) fragments of 37.24 (13) and 33.94 (13)°, respectively.

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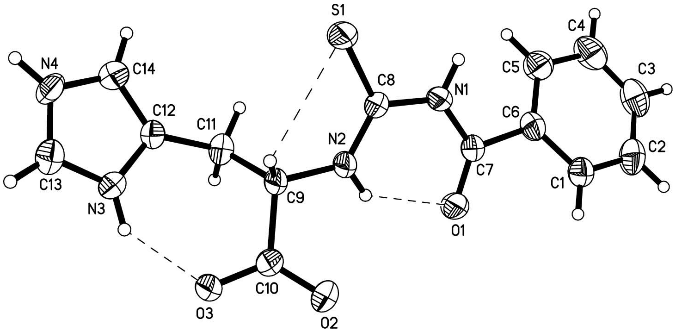

Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

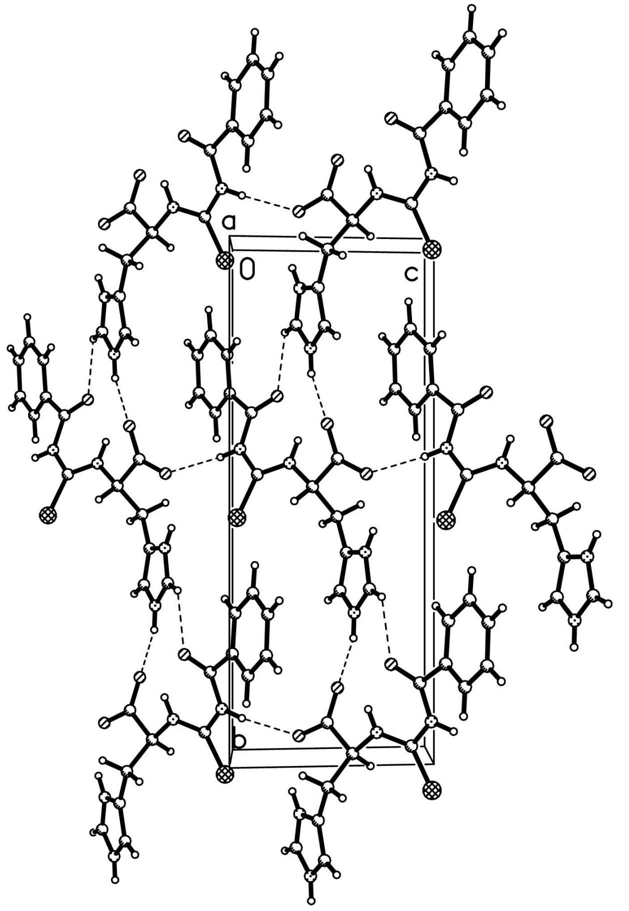

Figure 2
Packing diagram of the title complex, viewed down the *a* axis. Dashed lines denote C—H···O and N—H···O hydrogen bonds.

There are three intramolecular interactions, *viz.*C9—H9*A*···S1, N2—H2*A*···O1 and N3—H3*C*···O3, forming a pseudo-five-membered ring (C9—H9*A*—S1—C8—N2), a pseudo-six-membered ring (N2—H2*A*—O1—C7—N1—C8) and a pseudo-seven-membered ring (N3—H3*C*—O3—C10—C9—C11—C12), respectively. In the crystal structure, the molecules are linked by intermolecular hydrogen bonds, N1—H1*A*···O3^{i}, N4—H4*B*···O2^{ii} and C13—H13*A*···O1^{ii} (symmetry codes as in Table 2) to form a two-dimensional network parallel to the (100) plane (Fig. 2).

Experimental

A solution of L-histidine (0.01 mol, 1.55 g) in acetone (50 ml) was added dropwise to a two-necked round-bottomed flask containing equimolar amount of benzoyl isothiocyanate in acetone. The mixture was refluxed about 2 h and filtered into a beaker containing some ice. The resulting white precipitate was washed with acetone–distilled water and dried under vacuum. Recrystallization from ethanol yielded single crystals suitable for X-ray analysis (m.p. 445–446 K). Analysis found (calculated): C 51.8 (52.77), H 4.35 (4.43), N 16.86 (17.59), O 14.81 (15.08), S 9.12% (10.05%).

Crystal data

$C_{14}H_{14}N_4O_3S$
$M_r = 318.35$
Monoclinic, $P2_1$
$a = 5.6995$ (13) Å
$b = 17.916$ (4) Å
$c = 7.0525$ (17) Å
$\beta = 99.424$ (5)°
$V = 710.4$ (3) Å^3
$Z = 2$
$D_x = 1.488$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2717 reflections
$\theta = 2.2$–27.5°
$\mu = 0.25$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.29 × 0.27 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.931$, $T_{max} = 0.952$
4765 measured reflections
1678 independent reflections
1648 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.5°$
$h = -7 \rightarrow 7$
$k = -14 \rightarrow 23$
$l = -9 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.097$
$S = 1.14$
1678 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.1656P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.677 (3)	N2—C9	1.456 (3)
O1—C7	1.221 (4)	C14—C12	1.348 (4)
O2—C10	1.229 (4)	C14—N4	1.357 (4)
O3—C10	1.244 (4)	C13—N4	1.304 (4)
N1—C7	1.369 (4)	C13—N3	1.318 (4)
N1—C8	1.382 (4)	C12—N3	1.377 (4)
N2—C8	1.316 (4)		
C8—N2—C9—C10	−155.5 (3)	C9—C11—C12—C14	124.1 (4)
C10—C9—C11—C12	84.7 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9*A*···S1	0.98	2.75	3.076 (3)	100
N2—H2*A*···O1	0.86	1.95	2.630 (3)	135
N3—H3*C*···O3	0.86	1.94	2.650 (4)	139
N1—H1*A*···O3^{i}	0.86	2.05	2.870 (4)	158
N4—H4*B*···O2^{ii}	0.86	1.84	2.650 (4)	156
C13—H13*A*···O1^{ii}	0.93	2.40	3.070 (4)	129

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

After their location in a difference map, all H atoms were placed geometrically at ideal positions and allowed to ride on the parent C atoms, with C—H = 0.93–0.98 Å, N—H = 0.86 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N). Due to the absence of any significant anomalous scatterers in the crystal, Friedel pairs were merged before the the final refinement.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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