(2S)-1-(Benzoylthiocarbamoyl)pyrrolidine-2-carboxylic acid monohydrate

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Key indicators

Single-crystal X-ray study

T = 298 K

Mean σ(C–C) = 0.008 Å

Disorder in main residue

R factor = 0.037

wR factor = 0.088

Data-to-parameter ratio = 13.2

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

In the title compound, $C_{13}H_{14}N_2O_3S \cdot H_2O$, the benzoyl group lies out of the plane of the N_2CS thiourea unit, having a $C_{carbonyl}$—N—C—S torsion angle of 119.4 (4)°. The crystal structure is stabilized by N—H···O, O—H···O, C—H···O and C—H···S intermolecular hydrogen bonds, forming a two-dimensional network.

Comment

In the benzoylthiourea derivatives of amino acids such as (2S)-2-(3-benzoylthioureido)-3-methylbutyric acid (Ngah *et al.*, 2005), and 2-(3-benzoylthioureido)propionic acid (Ngah *et al.*, 2006), the N atom of the amino acid attached to the thione carbon has an H atom bonded to it. However, in the title molecule, the pyrrolidine N atom has no attached H atom. The C7—N1—C8—S1 torsion angle of 119.4 (4)° shows that the benzoyl group lies significantly out of the thiourea plane. As in most benzoylthiourea compounds, the benzoyl group is approximately *trans* to the thiono S atom across the C7—N1 bond. The bonds length and angles are in normal ranges (Allen *et al.*, 1987).

(I)
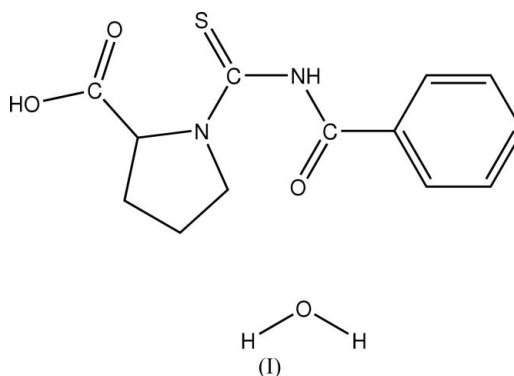
The crystal structure is stabilized by intermolecular N—H···O, O—H···O, C—H···O and C—H···S hydrogen bonds, forming a two-dimensional network (Fig. 2, Table 1).

Experimental

An equimolar solution of benzoyl isothiocyanate (0.03 mol, 5.04 g) and proline (0.03 mol, 3.45 g) in acetone (200 ml) was refluxed for 1 h in a two-necked round-bottomed flask. The mixture was filtered into a beaker containing some ice. The resulting white precipitate was washed with distilled water followed by cold acetone, then dried in a vaccum desiccator (yield 75%, 6.26 g). Recrystallization from methanol yielded colourless crystals suitable for X-ray analysis (m.p. 430.1–430.6 K). The water of crystallization comes from wet solvent or atmospheric moisture.

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Crystal data

$C_{13}H_{14}N_2O_3S \cdot H_2O$
$M_r = 296.34$
Orthorhombic, $P2_12_12_1$
$a = 9.05 (4)$ Å
$b = 12.00 (6)$ Å
$c = 13.33 (6)$ Å
$V = 1448 (12)$ Å^3

$Z = 4$
$D_x = 1.360$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.24$ mm^{-1}
$T = 298 (2)$ K
Block, colourless
$0.50 \times 0.30 \times 0.10$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.890$, $T_{max} = 0.977$

6893 measured reflections
2527 independent reflections
2047 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.088$
$S = 0.99$
2527 reflections
191 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Absolute structure: Flack (1983), 634 Friedel pairs
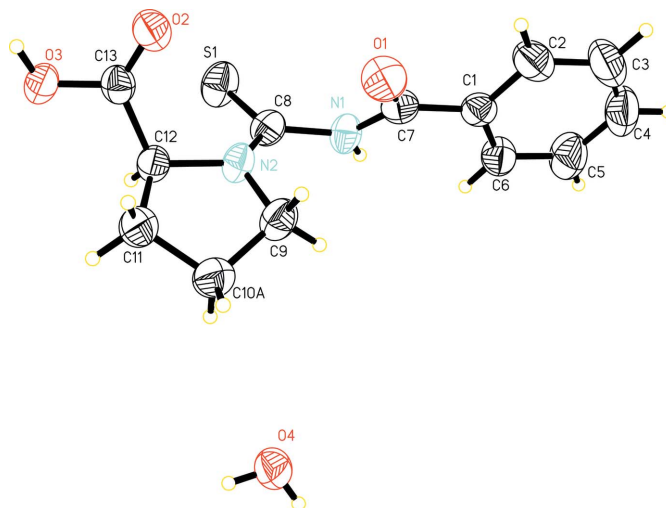Flack parameter: $-0.01 (10)$

Figure 1
The molecular structure of the title compound, (I), shown with 50% probability displacement ellipsoids. The minor disorder component has been omitted.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1$A\cdots$O4^i	0.86	2.31	3.113 (16)	156
O3—H3$\cdots$O4^{ii}	0.82	1.97	2.754 (14)	160
O4—H4$A\cdots$O2^{iii}	0.82 (2)	2.22 (2)	2.945 (15)	148 (3)
O4—H4$B\cdots$O1^{iii}	0.82 (2)	1.91 (2)	2.728 (14)	177 (3)
C11—H11$A\cdots$S1^{iv}	0.97	2.87	3.754 (19)	152

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

H atoms of the water molecule were located in a difference map and refined freely. Other H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å, O—H = 0.82 Å and N—H = 0.86 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$ for CH, CH$_2$ and NH, and $U_{iso}(H) = 1.5_{eq}(O)$ for OH. Atom C10 and its attached H atoms are disordered over two sites, A and B, with site occupancy factors of 0.916 (7) and 0.084 (7), respectively.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

Figure 2
Packing diagram of (I), viewed down the *a* axis. The dashed lines denote hydrogen bonds. The minor disorder component has been omitted.

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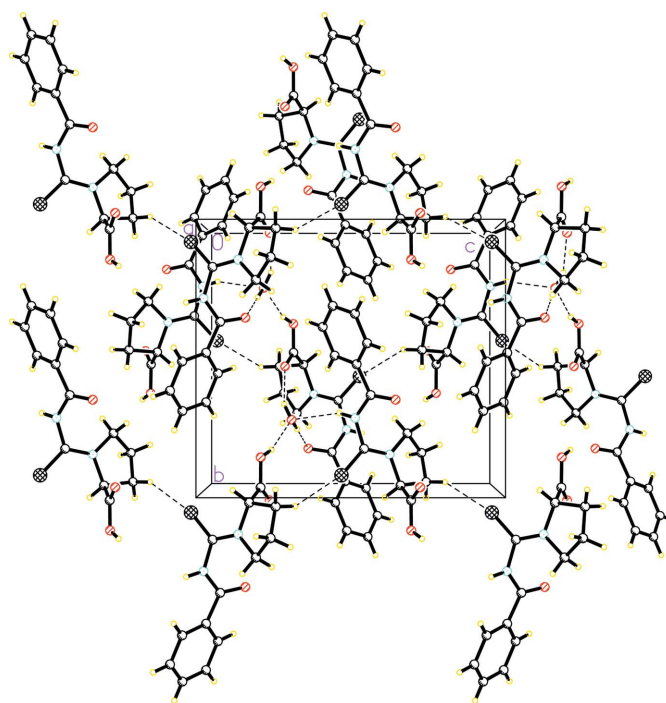
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