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(2*S*)-2-(3-Benzoylthioureido)-3-methylbutyric acid

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Key indicators

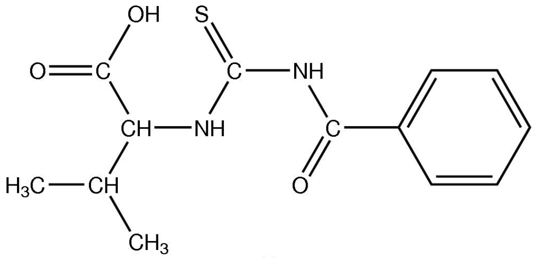
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.090
Data-to-parameter ratio = 9.9

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

The 3-methylbutyric acid and benzoyl fragments in the title compound, $C_{13}H_{16}N_2O_3S$, are *cis* and *trans*, respectively, with respect to the thiono group across the thiourea C—N bonds. In the crystal structure the molecules are stabilized by intermolecular N—H···O, O—H···S and C—H···O hydrogen bonds to form zigzag chains parallel to the *b* axis.

Comment

The title compound, (I), is analogous to 3-(3-benzoylthioureido)propionic acid (Yusof & Yamin, 2003), except that one of the substituents at the terminal N atom of the thiourea group is 3-methylbutyric acid derived from the reaction with L-valine. The molecule maintains its *cis–trans* configuration of the 3-methylbutyric acid and benzoyl fragments with respect to the C=S bond across their C8—N2 and C8—N1 bonds, respectively (Fig. 1). The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and are in agreement with those of 3-(3-benzoylthioureido)propionic acid (Yusof & Yamin, 2003). The phenyl fragment including the carbonyl group, (C1–C6)/C7/O1 [maximum deviation 0.065 (2) Å for atom O1], and the central thiourea, S1/N1/N2/C8 [maximum deviation 0.034 (2) Å for atom N2], are essentially coplanar, the dihedral angle being 3.94 (11)°. The molecule is stabilized by weak intramolecular and intermolecular hydrogen bonds, N—H···O, O—H···S and C—H···O (Table 2), to form zigzag chains parallel to the *b* axis (Fig. 2).



(I)

Experimental

A solution of L-valine (0.25 g, 2.13 mmol) in acetone was added dropwise to a two-necked round-bottomed flask containing an equimolar amount of benzoyl isothiocyanate in acetone. The mixture was refluxed for about 2 h and filtered into a beaker containing some ice. The white precipitate obtained was washed with acetone–distilled water and then dried under vacuum. Crystals suitable for X-ray analysis were obtained by recrystallization from toluene (m.p. 425–426 K). Analysis found: C 54.8, H 5.8, N 9.8, O 16.9, S 10.9%; calculated: C 55.8, H 5.7, N 10.0, O 17.1, S 11.4%.

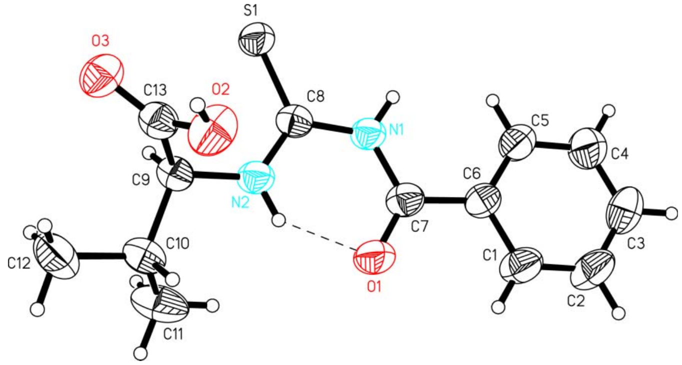


Figure 1
Molecular structure of (I), with 50% probability displacement ellipsoids. The dashed line indicates the intramolecular hydrogen bond.

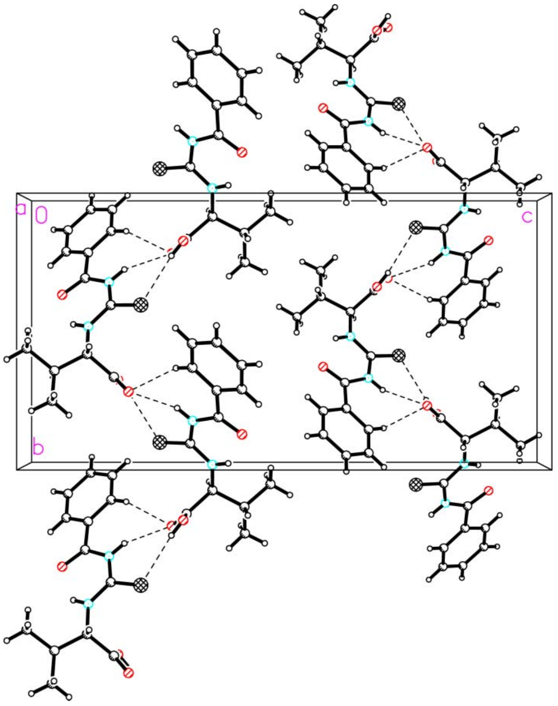


Figure 2
Packing diagram of (I), viewed down the *a* axis. The dashed lines denote N—H···O, O—H···S and C—H···O hydrogen bonds.

Crystal data

$C_{13}H_{16}N_2O_3S$
M_r = 280.34
Orthorhombic, $P2_12_12_1$
a = 5.2223 (13) Å
b = 11.781 (3) Å
c = 22.738 (5) Å
V = 1399.0 (6) Å^3
Z = 4
D_x = 1.331 Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 947 reflections
θ = 1.7–26.5°
μ = 0.24 mm^{-1}
T = 298 (2) K
Block, colourless
0.48 × 0.44 × 0.26 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.894, T_{max} = 0.941
7838 measured reflections

1711 independent reflections
1624 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 26.5°
h = −6 → 6
k = −14 → 14
l = −28 → 21

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.090
S = 1.07
1711 reflections
172 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.2185P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ <0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Selected interatomic distances (Å).

S1—C8	1.675 (2)	N1—C8	1.382 (3)
O1—C7	1.223 (3)	N2—C8	1.321 (3)
O2—C13	1.319 (3)	N2—C9	1.451 (3)
O3—C13	1.199 (3)	C9—C13	1.521 (3)
N1—C7	1.381 (3)	C9—C10	1.546 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	1.89	2.583 (3)	136
N1—H1*A*···O3^{i}	0.86	2.18	2.997 (3)	159
O2—H2···S1^{ii}	0.82	2.44	3.251 (2)	170
C5—H5*A*···O3^{i}	0.93	2.35	3.179 (3)	148

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

All H atoms were found in a difference map and were allowed to refine as riding, with C—H = 0.93–0.98 Å, N—H = 0.86 Å and O—H = 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C) for CH, NH and OH, and U_{iso}(H) = 1.5$_{eq}$(C) for CH$_3$. Owing to the absence of any significant anomalous dispersion in the molecules, Friedel pairs were merged before the final refinement. The absolute configuration was known from the chiral centre of a starting material, L-valine, which remained unchanged during the synthesis of the title compound. The Flack (1983) parameter for the unmerged data was 0.03 (8) for 1711 Friedel pairs.

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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