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2-[3-(4-Methoxybenzoyl)thioureido]-3-methylbutyric acid

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.062
wR factor = 0.162
Data-to-parameter ratio = 14.5

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

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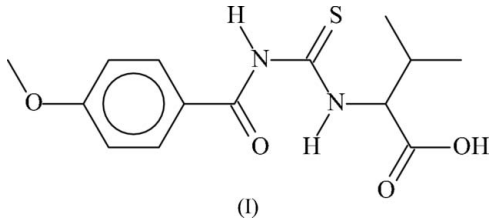
Molecules of the title compound, $C_{14}H_{18}N_2O_4S$, are linked into a linear chain, which propagates along the *a* axis of the triclinic unit cell through intermolecular hydrogen bonding between the carboxyl group and the thioxo S atom.

Comment

In an earlier study, L-valine was reacted with benzoyl isothiocyanate to form the optically active compound (2*S*)-2-(3-benzoylthioureido)-3-methylbutyric acid. The carboxylic acid portion of that molecule interacts with the doubly bonded S atom of an adjacent molecule to furnish a hydrogen-bonded helical chain that propagates along the *b* axis of the orthorhombic unit cell (Ngah *et al.*, 2005). The introduction of a methoxy substituent in the 4-position of the aromatic ring in the title optically-inactive compound, (I) (Fig. 1), leads a disruption of the helical motif. Compound (I) also adopts a linear chain motif, but the hydrogen bond (Table 1, Fig. 2) is much shorter.



(I)

Experimental

A solution of DL-valine (5 g, 4.3 mmol) in acetone (10 ml) was added dropwise to a solution of 4-methoxybenzoyl isothiocyanate (7.6 g, 4.3 mmol) in acetone (25 ml). The mixture was refluxed for several hours and then poured on to ice. The resulting yellow solid was washed with water and acetone and dried in air (yield 85%). Recrystallization from chloroform gave colourless crystals of (I) (m.p. 413–414 K).

Crystal data

$C_{14}H_{18}N_2O_4S$
M_r = 310.36
Triclinic, $P\overline{1}$
a = 8.051 (1) Å
b = 8.084 (1) Å
c = 13.205 (2) Å
α = 83.741 (2)°
β = 80.164 (3)°
γ = 66.162 (2)°
V = 773.8 (2) Å^3
Z = 2
D_x = 1.332 Mg m^{-3}
Mo *Kα* radiation
μ = 0.23 mm^{-1}
T = 295 (2) K
Block, colourless
0.31 × 0.16 × 0.12 mm

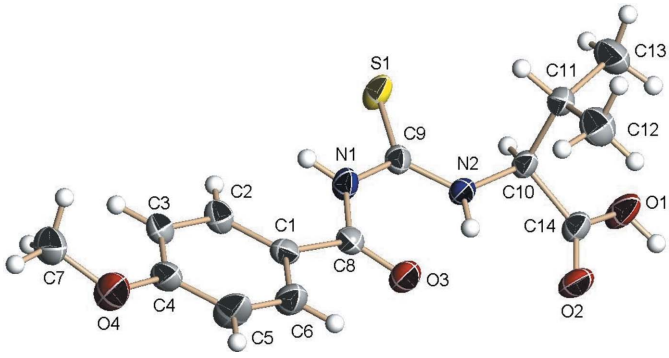


Figure 1
A plot of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii. The minor components of the disordered C atoms of the benzene ring are not shown.

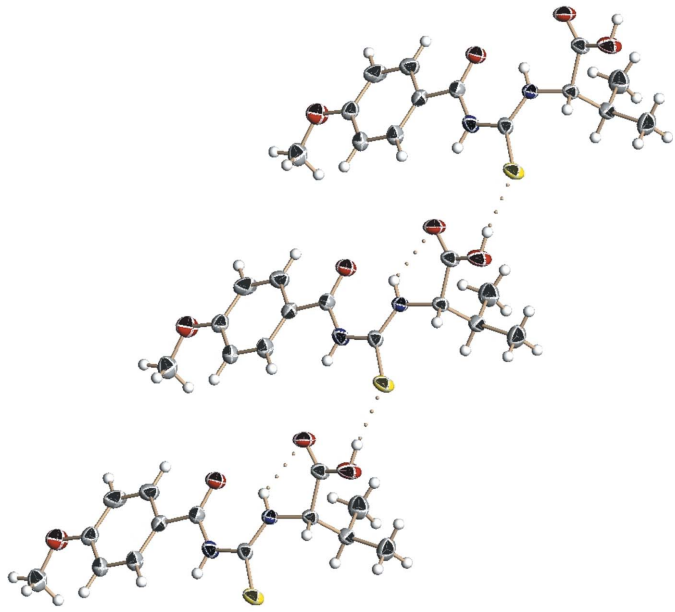


Figure 2
A plot depicting the linear chain motif of (I), propagating along the *a* axis of the unit cell. Dotted lines indicate hydrogen bonds.

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: none
8876 measured reflections
3509 independent reflections
2078 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.162$
$S = 1.01$
3509 reflections
242 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···S1^{i}	0.85 (1)	2.25 (1)	3.080 (2)	165 (3)
N2—H2n···O3	0.85 (1)	2.03 (2)	2.651 (3)	129 (2)

Symmetry code: (i) $x, y-1, z$.

The 1,4-phenylene ring is disordered over two sites and the occupancies refined to 0.52 (2):0.48 (2). Atoms C1 and C4 are not disordered, so that the ring appears to adopt two orientations along the C1—C4 axis. The C—C distances were restrained to 1.39 (1) Å and the 1,4-related distances were restrained to 2.78 (1) Å. Each component ring was restrained to be nearly planar.

C-bound H atoms were positioned geometrically, with C—H distances in the range 0.93–0.98 Å, and were included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C). The methyl groups were rotated to fit the electron density. The acid and amino H atoms were located in a difference Fourier map and were refined with a distance restraint of O—H = N—H = 0.85 (1) Å; their displacement parameters were refined freely.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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