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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.043
 wR factor = 0.106
Data-to-parameter ratio = 10.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

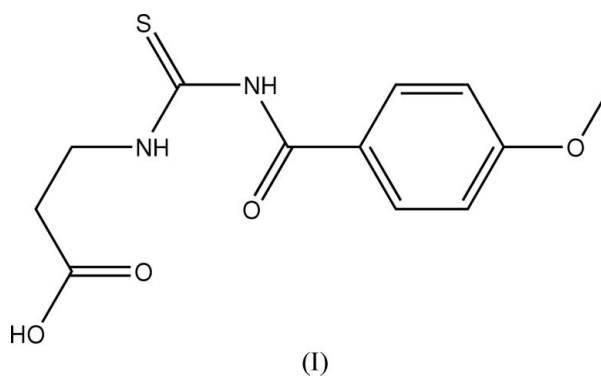
3-[3-(4-Methoxybenzoyl)thioureido]propionic acid

In the title molecule, $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_4\text{S}$, the methoxybenzoyl group is *trans* to the thiono S atom across the C—N bond. The propionic acid group adopts a *gauche* conformation about the C—C bond, with a $\text{C}_{\text{COOH}}-\text{C}-\text{N}$ torsion angle of $64.9(4)^\circ$. In the crystal structure, intermolecular O—H···S, N—H···O and C—H···O hydrogen bonds link the molecules into chains parallel to the *c* axis.

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Comment

The title compound, (I), is a carbonylthiourea derivative of β -alanine analogous to 3-(3-benzoylthiouredo)propionic acid, (II) (Yusof & Yamin, 2003), except that a methoxy group is attached at the *para* position of the benzoyl group (Fig. 1).



The molecule maintains its *cis-trans* configuration with respect to the positions of the propionic acid and 4-methoxybenzoyl groups relative to the S1 atom across the C9—N2 and C9—N1 bonds, respectively. In addition, the molecule adopts a *gauche* conformation about the C10—C11 bond, with a C12—C11—C10—N2 torsion angle of $64.9(4)^\circ$; in contrast, compound (II) adopts an *anti* conformation with a torsion angle of $-179.12(16)^\circ$. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and comparable to those in (II). However, the O3—C12 bond length [$1.325(4)\text{ \AA}$] is slightly longer than that in (II) [$1.296(2)\text{ \AA}$]. The 4-methoxyphenyl [C1—C6/O1/C7 (*A*)] and central carbonylthiourea [S1/O2/N1/N2/C8/C9 (*B*)] fragments are essentially planar. In the propionic acid fragment [O3/O4/C10/C11/C12 (*C*)], the maximum deviation from the mean plane is $0.186(4)\text{ \AA}$ for atom C10. The dihedral angles *A/B* and *B/C* are $10.35(13)$ and $62.08(17)^\circ$, respectively. The 4-methoxyphenyl unit is inclined to the propionic acid mean plane with a dihedral angle of $54.29(19)^\circ$, compared with $22.46(13)^\circ$ in (II). The intramolecular hydrogen bond N2—H2A···O2

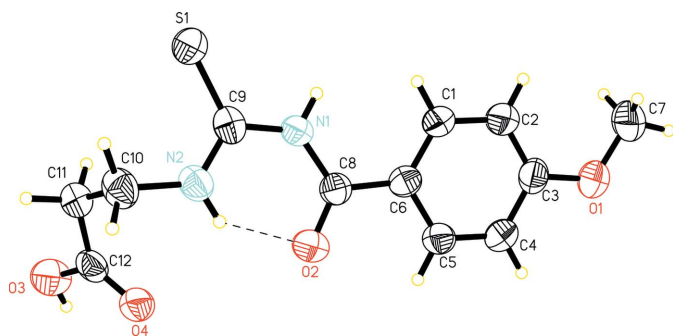


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids. The dashed line represents a hydrogen bond.

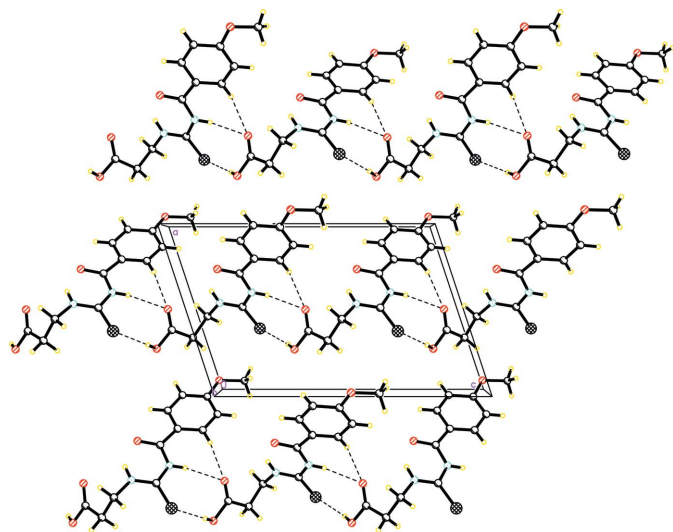


Figure 2
Packing diagram of the title complex, viewed down the *b* axis. The dashed lines denote intermolecular N—H...O, O—H...S and C—H...O hydrogen bonds.

(Table 2) forms a pseudo-six-membered ring (N2/H2A/O2/C8/N1/C9).

In the crystal structure, molecules are linked by intermolecular O—H...S, N—H...O and C—H...O hydrogen bonds (Table 2), forming a one-dimensional chain parallel to the *c* axis, in contrast to a two-dimensional hydrogen-bonded network in (II). The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

Experimental

A solution of 3-aminopropionic acid (0.05 mol, 4.45 g) in 20 ml acetone was added dropwise to a two-necked round-bottomed flask containing an equimolar solution of 4-methoxybenzoylisothiocyanate (0.05 mol, 9.65 g) in 20 ml acetone. The mixture was refluxed for about 5 h and then filtered into a beaker containing ice cubes. The resulting yellow precipitate was washed with cold acetone and distilled water. The product was dried under vacuum and kept in a desiccator (yield 11.99 g, 85%; m.p. 430.3–431.1 K). Recrystallization from ethanol yielded yellow crystals suitable for X-ray analysis.

Crystal data

$C_{12}H_{14}N_2O_4S$
 $M_r = 282.31$
Monoclinic, P_2
 $a = 9.795(3) \text{ \AA}$
 $b = 4.7790(13) \text{ \AA}$
 $c = 14.951(4) \text{ \AA}$
 $\beta = 108.275(5)^\circ$
 $V = 664.6(3) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.411 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
 $\mu = 0.26 \text{ mm}^{-1}$
 $T = 298(2) \text{ K}$
Plate, yellow
 $0.34 \times 0.17 \times 0.07 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.912$, $T_{\max} = 0.979$

3159 measured reflections
1792 independent reflections
1660 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$
 $\theta_{\max} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.106$
 $S = 1.15$
1792 reflections
172 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.0617P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.15 \text{ e \AA}^{-3}$
Absolute structure: Flack (1983),
628 Friedel pairs
Flack parameter: 0.16 (11)

Table 1

Selected geometric parameters (\AA , $^\circ$).

S1—C9	1.687 (4)	N1—C9	1.375 (4)
O2—C8	1.231 (4)	N1—C8	1.382 (5)
O3—C12	1.325 (4)	N2—C9	1.318 (4)
O4—C12	1.201 (5)	N2—C10	1.463 (5)
C9—N1—C8	128.2 (3)	N2—C9—S1	122.0 (3)
N2—C9—N1	117.7 (3)	N1—C9—S1	120.3 (3)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2A...O2	0.86	1.94	2.622 (4)	135
O3—H3...S1 ⁱ	0.82	2.32	3.126 (3)	169
N1—H1A...O4 ⁱⁱ	0.86	2.26	3.113 (4)	170
C1—H1B...O4 ⁱⁱ	0.93	2.28	3.155 (6)	156

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.97 \AA , N—H = 0.86 \AA and O—H = 0.82 \AA , with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ for CH and NH groups and $1.5U_{\text{eq}}(\text{C,O})$ for CH₃ and OH groups.

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).

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