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Methyl 3-(3-benzoylthioureido)-propanoate

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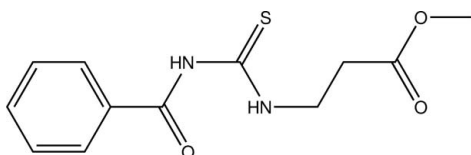
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.057; wR factor = 0.141; data-to-parameter ratio = 19.8.

In the title compound, $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_3\text{S}$, the propyl acetate group and the benzoyl group adopt a *cis-trans* conformation, respectively, with respect to the thiono S atom across the C–N bonds. The phenyl ring is twisted relative to the thiourea mean plane, forming a dihedral angle of 24.16 (9°). An intramolecular N–H \cdots O hydrogen bond occurs. The crystal packing is stabilized by intermolecular N–H \cdots O and C–H \cdots O hydrogen bonds, forming a chain along the a axis.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see: Yamin & Hassan (2004); Hassan *et al.* (2008*a,b,c*, 2009), Hung *et al.* (2010). For a related synthesis, see: Hassan *et al.* (2008*a*).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_3\text{S}$ $M_r = 266.31$ Triclinic, $P\bar{1}$ $a = 7.5901$ (18) Å $b = 8.2688$ (19) Å $c = 10.547$ (3) Å $\alpha = 86.168$ (5°) $\beta = 86.892$ (4°) $\gamma = 81.545$ (4°) $V = 652.6$ (3) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.25$ mm⁻¹ $T = 298$ K $0.35 \times 0.31 \times 0.23$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 2000)

 $T_{\min} = 0.918$, $T_{\max} = 0.945$

8953 measured reflections

3229 independent reflections

2654 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.024$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.057$ $wR(F^2) = 0.141$ $S = 1.12$

3229 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O1}$	0.86	1.94	2.625 (2)	136
$\text{N1}-\text{H1A}\cdots\text{O2}^i$	0.86	2.17	3.022 (2)	169
$\text{C1}-\text{H1B}\cdots\text{O2}^i$	0.93	2.50	3.187 (3)	130
$\text{C9}-\text{H9A}\cdots\text{O1}^{ii}$	0.97	2.59	3.464 (3)	150

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y+1, -z+1$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEPIII (Burnett & Johnson, 1996), ORTEP-3 for Windows (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: SHELXTL (Sheldrick, 2008), PARST (Nardelli, 1995) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2657).

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supporting information

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Methyl 3-(3-benzoylthioureido)propanoate

Ibrahim N. Hassan, Chong Yan Yi and Mohammad B. Kassim

S1. Comment

The title compound, **I**, is a methyl ester derivative of beta-alanine thiourea analogous to our previous reported, methyl-2-(3-benzoylthioureido)acetate, **II** (Hassan *et al.* 2009).

The molecule maintains the same *cis-trans* conformation with respect to the positions of the methyl propanoate and benzoyl groups, relative to the S atom across the C—N bonds (Fig 1), respectively. The phenyl group, [C1/C2/C3/C4/C5/C6], and the methyl propanoate fragment, [O2/O3/C10/C11/C12], are essentially planar and the dihedral angle between them is 82.28 (11)°. The bond lengths (Allen *et al.*, 1987) and angles in the molecules are in normal ranges and comparable to those of **II**. The C=S bond length [1.670 (2) Å] is identical within experimental error to that of **II** [1.662 (5) Å]. The thiourea fragment, [S1/O1/N1/N2/C6/C7/C8/C9], is essentially planar with a maximum deviation of 0.037 (2) Å, at the atom N2. The phenyl ring [C1—C6] is inclined to the thiourea mean plane making a dihedral angle of 24.16 (9)° which is slightly larger than that of **II** [20.12 (19)°]. Atom O3 of the methyl propanoate group, (O2/O3/C10/C11/C12), has the maximum deviation 0.033 (2) Å from the mean plane.

There is one intramolecular hydrogen bonds, N2—H2A···O1 (Table 1) forming a pseudo-six-membered ring (N2/H2A/O1/C7/N1/C8). The intermolecular N1—H1A···O2, C1—H1B···O2 and C9—H9A···O1 hydrogen bonds, (Table 1), link the molecules into a chain parallel to the *a* axis (Fig 2).

S2. Experimental

The title compound was synthesized according to a previously reported compound (Hassan *et al.*, 2008a). A yellowish crystal, suitable for X-ray crystallography, was obtained by a slow evaporation from CH₂Cl₂ solution at room temperature (yield 79%).

S3. Refinement

H atoms of both C and N atoms were positioned geometrically and allowed to ride on their parent atoms, with $U_{\text{iso}} = 1.2U_{\text{eq}}$ (C) for aromatic 0.93 Å, $U_{\text{iso}} = 1.2U_{\text{eq}}$ (C) for CH₂ 0.97 Å, $U_{\text{iso}} = 1.5U_{\text{eq}}$ (C) for CH₃ 0.96 Å, $U_{\text{iso}} = 1.2U_{\text{eq}}$ (N) for N—H 0.86 Å.

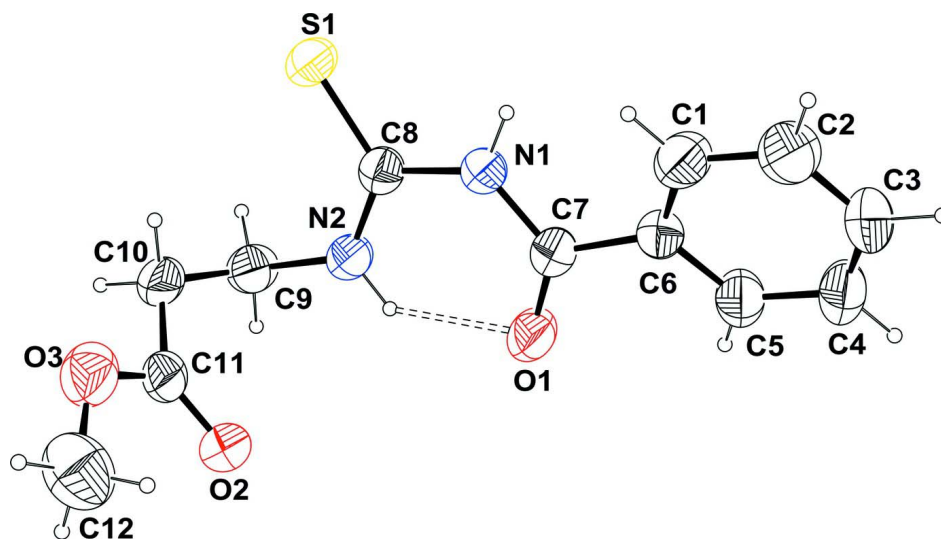


Figure 1

The molecular structure of (I), with the atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bond is shown as dashed line and H atoms are represented as small spheres of arbitrary radii.

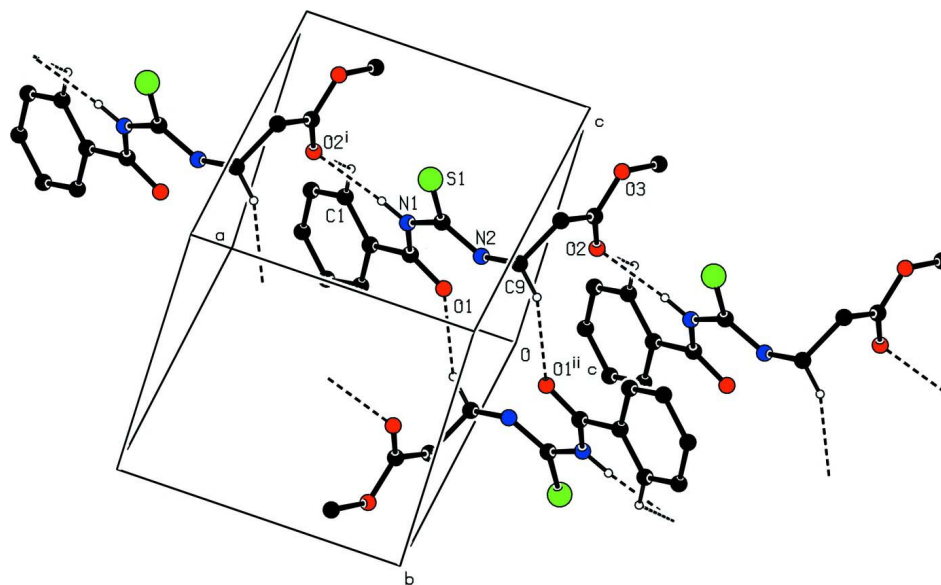


Figure 2

Partial packing view of (I) showing the chain formed by N-H...O and C-H...O hydrogen bonds which are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y+1, -z+1$]

Methyl 3-(3-benzoylthioureido)propanoate

Crystal data

$C_{12}H_{14}N_2O_3S$

$M_r = 266.31$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.5901(18)\ \text{\AA}$

$b = 8.2688(19)\ \text{\AA}$

$c = 10.547(3)\ \text{\AA}$

$\alpha = 86.168(5)^\circ$

$\beta = 86.892 (4)^\circ$
 $\gamma = 81.545 (4)^\circ$
 $V = 652.6 (3) \text{ \AA}^3$
 $Z = 2$
 $F(000) = 280$
 $D_x = 1.355 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2861 reflections
 $\theta = 1.9\text{--}28.3^\circ$
 $\mu = 0.25 \text{ mm}^{-1}$
 $T = 298 \text{ K}$
 Block, colourless
 $0.35 \times 0.31 \times 0.23 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2000)
 $T_{\min} = 0.918, T_{\max} = 0.945$

8953 measured reflections
 3229 independent reflections
 2654 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$
 $\theta_{\max} = 28.3^\circ, \theta_{\min} = 1.9^\circ$
 $h = -10 \rightarrow 10$
 $k = -11 \rightarrow 11$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.141$
 $S = 1.12$
 3229 reflections
 163 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.1657P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.27 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.38506 (7)	0.35748 (7)	0.87738 (5)	0.05290 (19)
O1	0.2339 (2)	0.3507 (2)	0.46757 (14)	0.0658 (5)
O2	-0.1984 (2)	0.2418 (2)	0.71396 (14)	0.0594 (4)
O3	-0.1813 (2)	0.1042 (2)	0.90333 (16)	0.0656 (5)
N1	0.4213 (2)	0.3096 (2)	0.63147 (15)	0.0442 (4)
H1A	0.5305	0.2778	0.6496	0.053*
N2	0.1375 (2)	0.4127 (2)	0.70422 (15)	0.0472 (4)
H2A	0.1086	0.4052	0.6273	0.057*
C1	0.6740 (3)	0.1203 (3)	0.4582 (2)	0.0572 (6)
H1B	0.6873	0.0919	0.5444	0.069*

C2	0.7984 (3)	0.0513 (3)	0.3688 (3)	0.0685 (7)
H2B	0.8948	-0.0242	0.3949	0.082*
C3	0.7794 (3)	0.0944 (3)	0.2416 (2)	0.0644 (6)
H3A	0.8635	0.0484	0.1820	0.077*
C4	0.6379 (3)	0.2044 (3)	0.2022 (2)	0.0630 (6)
H4A	0.6256	0.2331	0.1160	0.076*
C5	0.5136 (3)	0.2726 (3)	0.2903 (2)	0.0544 (5)
H5A	0.4173	0.3473	0.2630	0.065*
C6	0.5295 (3)	0.2318 (2)	0.41872 (18)	0.0429 (4)
C7	0.3820 (3)	0.3027 (3)	0.50660 (18)	0.0457 (5)
C8	0.3050 (3)	0.3619 (2)	0.73260 (17)	0.0412 (4)
C9	-0.0010 (3)	0.4804 (3)	0.7945 (2)	0.0524 (5)
H9A	-0.0993	0.5396	0.7477	0.063*
H9B	0.0463	0.5584	0.8432	0.063*
C10	-0.0714 (3)	0.3532 (3)	0.88575 (19)	0.0548 (6)
H10A	0.0264	0.2960	0.9341	0.066*
H10B	-0.1579	0.4087	0.9453	0.066*
C11	-0.1567 (2)	0.2306 (3)	0.82278 (19)	0.0498 (5)
C12	-0.2732 (4)	-0.0183 (4)	0.8564 (3)	0.0828 (8)
H12A	-0.2834	-0.1032	0.9219	0.124*
H12B	-0.2074	-0.0642	0.7838	0.124*
H12C	-0.3900	0.0309	0.8323	0.124*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0462 (3)	0.0734 (4)	0.0374 (3)	0.0019 (2)	-0.0064 (2)	-0.0099 (2)
O1	0.0455 (8)	0.1087 (14)	0.0376 (8)	0.0111 (8)	-0.0050 (6)	-0.0092 (8)
O2	0.0487 (9)	0.0918 (12)	0.0405 (8)	-0.0169 (8)	-0.0049 (6)	-0.0074 (8)
O3	0.0587 (10)	0.0788 (12)	0.0545 (9)	0.0018 (8)	-0.0010 (8)	0.0032 (8)
N1	0.0347 (8)	0.0593 (10)	0.0377 (8)	-0.0015 (7)	-0.0020 (6)	-0.0076 (7)
N2	0.0380 (8)	0.0664 (11)	0.0360 (8)	-0.0001 (8)	-0.0033 (6)	-0.0086 (7)
C1	0.0571 (13)	0.0664 (14)	0.0464 (11)	0.0019 (11)	-0.0066 (10)	-0.0101 (10)
C2	0.0546 (14)	0.0803 (17)	0.0663 (15)	0.0120 (12)	-0.0043 (11)	-0.0194 (13)
C3	0.0532 (13)	0.0839 (17)	0.0544 (13)	-0.0035 (12)	0.0137 (10)	-0.0200 (12)
C4	0.0623 (14)	0.0824 (17)	0.0419 (11)	-0.0071 (12)	0.0108 (10)	-0.0037 (11)
C5	0.0491 (12)	0.0660 (14)	0.0450 (11)	-0.0022 (10)	0.0038 (9)	0.0005 (10)
C6	0.0407 (10)	0.0494 (11)	0.0400 (10)	-0.0096 (8)	0.0009 (8)	-0.0072 (8)
C7	0.0434 (10)	0.0571 (12)	0.0362 (9)	-0.0061 (9)	-0.0008 (8)	-0.0039 (8)
C8	0.0406 (10)	0.0456 (10)	0.0369 (9)	-0.0042 (8)	-0.0007 (7)	-0.0043 (8)
C9	0.0415 (10)	0.0669 (14)	0.0467 (11)	0.0063 (9)	-0.0034 (9)	-0.0172 (10)
C10	0.0410 (10)	0.0857 (16)	0.0365 (10)	-0.0008 (10)	0.0006 (8)	-0.0160 (10)
C11	0.0300 (9)	0.0757 (14)	0.0398 (10)	0.0046 (9)	0.0044 (8)	-0.0075 (10)
C12	0.0768 (19)	0.0713 (18)	0.098 (2)	-0.0117 (15)	0.0022 (16)	0.0070 (16)

Geometric parameters (Å, °)

S1—C8	1.6699 (19)	C3—C4	1.365 (4)
O1—C7	1.220 (2)	C3—H3A	0.9300
O2—C11	1.201 (2)	C4—C5	1.375 (3)
O3—C11	1.331 (3)	C4—H4A	0.9300
O3—C12	1.438 (3)	C5—C6	1.381 (3)
N1—C7	1.373 (2)	C5—H5A	0.9300
N1—C8	1.397 (2)	C6—C7	1.491 (3)
N1—H1A	0.8600	C9—C10	1.514 (3)
N2—C8	1.323 (2)	C9—H9A	0.9700
N2—C9	1.454 (2)	C9—H9B	0.9700
N2—H2A	0.8600	C10—C11	1.489 (3)
C1—C2	1.387 (3)	C10—H10A	0.9700
C1—C6	1.388 (3)	C10—H10B	0.9700
C1—H1B	0.9300	C12—H12A	0.9600
C2—C3	1.375 (4)	C12—H12B	0.9600
C2—H2B	0.9300	C12—H12C	0.9600
C11—O3—C12	116.5 (2)	O1—C7—C6	120.56 (18)
C7—N1—C8	127.78 (16)	N1—C7—C6	116.94 (17)
C7—N1—H1A	116.1	N2—C8—N1	116.04 (16)
C8—N1—H1A	116.1	N2—C8—S1	125.16 (15)
C8—N2—C9	124.28 (16)	N1—C8—S1	118.80 (14)
C8—N2—H2A	117.9	N2—C9—C10	113.98 (18)
C9—N2—H2A	117.9	N2—C9—H9A	108.8
C2—C1—C6	119.8 (2)	C10—C9—H9A	108.8
C2—C1—H1B	120.1	N2—C9—H9B	108.8
C6—C1—H1B	120.1	C10—C9—H9B	108.8
C3—C2—C1	120.0 (2)	H9A—C9—H9B	107.7
C3—C2—H2B	120.0	C11—C10—C9	114.09 (17)
C1—C2—H2B	120.0	C11—C10—H10A	108.7
C4—C3—C2	120.5 (2)	C9—C10—H10A	108.7
C4—C3—H3A	119.8	C11—C10—H10B	108.7
C2—C3—H3A	119.8	C9—C10—H10B	108.7
C3—C4—C5	119.8 (2)	H10A—C10—H10B	107.6
C3—C4—H4A	120.1	O2—C11—O3	123.6 (2)
C5—C4—H4A	120.1	O2—C11—C10	125.4 (2)
C4—C5—C6	121.0 (2)	O3—C11—C10	110.98 (18)
C4—C5—H5A	119.5	O3—C12—H12A	109.5
C6—C5—H5A	119.5	O3—C12—H12B	109.5
C5—C6—C1	118.93 (19)	H12A—C12—H12B	109.5
C5—C6—C7	117.30 (18)	O3—C12—H12C	109.5
C1—C6—C7	123.63 (18)	H12A—C12—H12C	109.5
O1—C7—N1	122.51 (18)	H12B—C12—H12C	109.5

Hydrogen-bond geometry (Å, °)

<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···O1	0.86	1.94	2.625 (2)	136
N1—H1A···O2 ⁱ	0.86	2.17	3.022 (2)	169
C1—H1B···O2 ⁱ	0.93	2.50	3.187 (3)	130
C9—H9A···O1 ⁱⁱ	0.97	2.59	3.464 (3)	150

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y+1, -z+1$.