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2-(3-Benzoylthioureido)-3-phenylpropanoic acid

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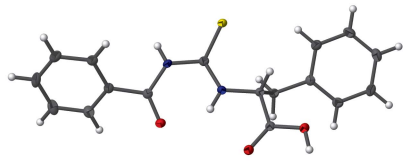
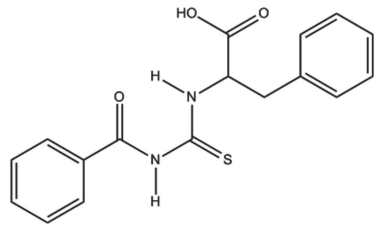
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{16}N_2O_3S$, the phenylpropanoic acid and the benzoyl moieties adopt a *cis–trans* conformation, respectively, with respect to the thiono S atom across the C—N bonds. An intramolecular N—H···O hydrogen bond generates an $S(6)$ ring. The crystal structure features carboxylic acid inversion dimers and pairwise N—H···S hydrogen bonds, which together generate $[20\bar{1}]$ chains. Weak C—H···O hydrogen bonds are also observed.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) adopts a *cis–trans* conformation with respect to the positions of the phenylpropanoic acid and benzoyl groups, relative to the S atom across the C8—N2 and C8—N1 bonds, respectively. The C8—S1, C7—O1, N1—C7, N1—C8 and N2—C8 bond lengths are similar to the corresponding bond lengths in related structures (Hassan *et al.*, 2008, 2009). The plane through the central thiourea unit (S1/N1/N2/C8/C9) forms dihedral angles of 14.90 (6) and 50.41 (6)°, with respect to the phenyl rings of the phenylpropanoic acid (C11–C16) and benzoyl (C1–C6) groups, respectively. The latter angle is larger than that previously reported for methyl 2-(3-benzoylthioureido)acetate (Hassan *et al.*, 2009). The phenyl rings of the phenylpropanoic acid and benzoyl groups subtend a dihedral angle of 36.06 (8)°. An intramolecular hydrogen bond, N2—H2*A*···O1, generates an $S(6)$ ring.

The crystal structure (Fig. 2) features carboxylic-acid inversion dimers linked by pairs of O3—H3a···O2 hydrogen bonds (Table 1). The dimers are linked by pairwise N1—H1*A*···S1 hydrogen bonds, generating $[20\bar{1}]$ chains. Weak C16—H11···O1 hydrogen bonds are also observed.

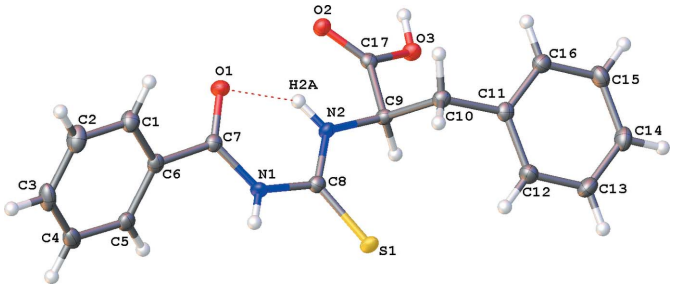


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bond is shown as a dashed line.

Synthesis and crystallization

The title compound was synthesized according to a previously reported method (Ngah *et al.*, 2005) with modification. Instead of 2-aminopropionic acid, 2-amino-3-phenylpropanoic acid was used for this reaction. Colourless plates were obtained by recrystallization from ethanol solution at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank Universiti Kebangsaan Malaysia for research grants (OUP-2012–073 and UKM-PTS-016–2010), the Ministry of Higher Education for the UKM-ST-06-FRGS0111–2009 grant and MyMaster funding for CYY.

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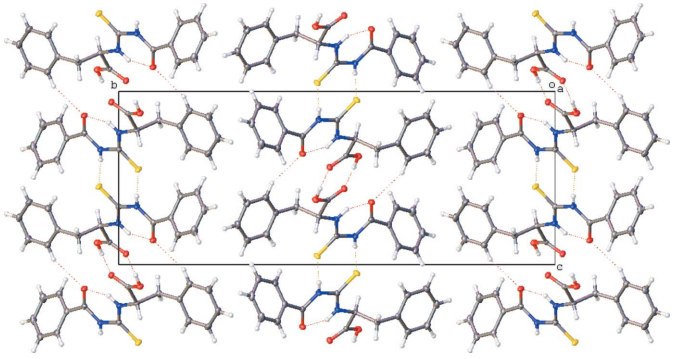

Figure 2
Partial packing view of 2-(3-benzoylthioureido)-3-phenylpropanoic acid, showing the zigzag chain formed by N—H···S, C—H···O and O—H···O hydrogen bonds which are shown as dashed lines [Symmetry codes: (i) $-x, -y+1, -z+2$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y+1, -z+1$.]

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.86 (2)	1.98 (2)	2.6480 (16)	134.1 (16)
N1—H1A···S1^{i}	0.790 (19)	2.571 (19)	3.3496 (13)	168.9 (18)
O3—H3A···O2^{ii}	0.87 (2)	1.81 (2)	2.6696 (14)	175 (2)
C16—H16···O1^{iii}	0.93	2.54	3.4026 (18)	155

Symmetry codes: (i) $-x, -y+1, -z+2$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y+1, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{16}N_2O_3S$
M_r	328.38
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	5.8750 (2), 25.9891 (12), 10.3089 (4)
β (°)	90.761 (4)
V (Å^3)	1573.89 (11)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.97
Crystal size (mm)	0.25 × 0.09 × 0.05
Data collection	
Diffractometer	Area
Absorption correction	Multi-scan (*DENZO/SCALE-PACK*; Otwinowski & Minor, 1997)
T_{min}, T_{max}	0.638, 0.908
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10980, 3045, 2824
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.096, 1.04
No. of reflections	3045
No. of parameters	220
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.23

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2006), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *OLEX2* (Dolomanov *et al.*, 2009), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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full crystallographic data

IUCrData (2016). **1**, x161091 [https://doi.org/10.1107/S2414314616010919]

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Crystal data

$C_{17}H_{16}N_2O_3S$
M_r = 328.38
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 5.8750 (2) Å
b = 25.9891 (12) Å
c = 10.3089 (4) Å
β = 90.761 (4)°
V = 1573.89 (11) Å^3
Z = 4
$F(000)$ = 688
D_x = 1.386 Mg m^{-3}
Melting point: 423 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 5690 reflections
θ = 3–71°
μ = 1.97 mm^{-1}
T = 100 K
Plate, colourless
0.25 × 0.09 × 0.05 mm

Data collection

Area diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)
T_{min} = 0.638, T_{max} = 0.908
10980 measured reflections
3045 independent reflections
2824 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 71.4°, θ_{min} = 3.4°
h = −7→7
k = −29→31
l = −11→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.04
3045 reflections
220 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.745P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.27806 (6)	0.545305 (13)	0.95051 (3)	0.01781 (13)
O1	0.22425 (17)	0.42118 (4)	0.64014 (9)	0.0181 (2)
O2	0.75216 (16)	0.48331 (4)	0.56133 (10)	0.0160 (2)
O3	0.97354 (17)	0.54898 (4)	0.62633 (10)	0.0161 (2)
N1	0.1462 (2)	0.45848 (5)	0.83583 (12)	0.0153 (3)
N2	0.4436 (2)	0.50147 (5)	0.73866 (12)	0.0145 (3)
C1	−0.1982 (3)	0.36410 (6)	0.67098 (15)	0.0203 (3)
H1	−0.1854	0.3781	0.5883	0.024*
C2	−0.3611 (3)	0.32678 (6)	0.69518 (17)	0.0254 (4)
H2	−0.4607	0.3165	0.6293	0.031*
C3	−0.3758 (3)	0.30477 (6)	0.81781 (18)	0.0250 (3)
H3	−0.4849	0.2797	0.8337	0.030*
C4	−0.2279 (3)	0.32014 (6)	0.91634 (16)	0.0220 (3)
H4	−0.2360	0.3049	0.9977	0.026*
C5	−0.0675 (3)	0.35843 (5)	0.89373 (15)	0.0179 (3)
H5	0.0299	0.3691	0.9603	0.022*
C6	−0.0534 (2)	0.38063 (5)	0.77145 (14)	0.0157 (3)
C7	0.1188 (2)	0.42121 (5)	0.74175 (14)	0.0145 (3)
C8	0.2955 (2)	0.50034 (5)	0.83430 (13)	0.0147 (3)
C9	0.6127 (2)	0.54169 (5)	0.72320 (13)	0.0139 (3)
H9	0.6863	0.5489	0.8071	0.017*
C10	0.5081 (2)	0.59226 (5)	0.66649 (14)	0.0155 (3)
H10A	0.4980	0.5893	0.5728	0.019*
H10B	0.3547	0.5962	0.6987	0.019*
C11	0.6439 (2)	0.63986 (5)	0.70080 (14)	0.0156 (3)
C12	0.6241 (3)	0.66130 (6)	0.82414 (15)	0.0205 (3)
H12	0.5286	0.6460	0.8842	0.025*
C13	0.7457 (3)	0.70522 (6)	0.85818 (15)	0.0231 (3)
H13	0.7292	0.7195	0.9402	0.028*
C14	0.8916 (3)	0.72781 (6)	0.77023 (16)	0.0233 (3)
H14	0.9739	0.7571	0.7932	0.028*
C15	0.9138 (3)	0.70643 (6)	0.64755 (16)	0.0221 (3)
H15	1.0122	0.7213	0.5884	0.027*
C16	0.7893 (3)	0.66287 (6)	0.61291 (14)	0.0191 (3)
H16	0.8036	0.6490	0.5302	0.023*

C17	0.7876 (2)	0.52116 (5)	0.62938 (13)	0.0136 (3)
H1A	0.060 (3)	0.4587 (7)	0.894 (2)	0.018 (5)*
H2A	0.440 (3)	0.4775 (8)	0.6817 (19)	0.022 (5)*
H3A	1.055 (4)	0.5387 (9)	0.562 (2)	0.036 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0200 (2)	0.0174 (2)	0.0163 (2)	−0.00387 (12)	0.00715 (14)	−0.00508 (12)
O1	0.0211 (5)	0.0186 (5)	0.0147 (5)	−0.0030 (4)	0.0044 (4)	−0.0023 (4)
O2	0.0157 (5)	0.0152 (5)	0.0173 (5)	−0.0006 (4)	0.0040 (4)	−0.0022 (4)
O3	0.0128 (5)	0.0187 (5)	0.0170 (5)	−0.0017 (4)	0.0035 (4)	−0.0022 (4)
N1	0.0167 (6)	0.0157 (6)	0.0137 (6)	−0.0026 (4)	0.0062 (5)	−0.0014 (4)
N2	0.0158 (6)	0.0137 (6)	0.0140 (6)	−0.0015 (5)	0.0028 (4)	−0.0031 (5)
C1	0.0209 (7)	0.0172 (7)	0.0227 (8)	0.0006 (6)	−0.0022 (6)	0.0008 (6)
C2	0.0194 (7)	0.0199 (8)	0.0368 (9)	−0.0012 (6)	−0.0058 (7)	−0.0010 (7)
C3	0.0189 (8)	0.0156 (7)	0.0406 (9)	−0.0027 (6)	0.0081 (7)	0.0012 (7)
C4	0.0261 (8)	0.0152 (7)	0.0251 (8)	0.0023 (6)	0.0109 (6)	0.0012 (6)
C5	0.0199 (7)	0.0142 (7)	0.0198 (7)	0.0021 (5)	0.0047 (6)	−0.0024 (5)
C6	0.0144 (7)	0.0129 (7)	0.0198 (7)	0.0023 (5)	0.0035 (5)	−0.0015 (5)
C7	0.0143 (7)	0.0138 (7)	0.0155 (7)	0.0020 (5)	−0.0002 (5)	0.0007 (5)
C8	0.0151 (7)	0.0143 (7)	0.0149 (7)	0.0016 (5)	0.0010 (5)	0.0001 (5)
C9	0.0134 (7)	0.0154 (7)	0.0129 (6)	−0.0013 (5)	0.0015 (5)	−0.0014 (5)
C10	0.0137 (6)	0.0164 (7)	0.0164 (7)	0.0007 (5)	0.0016 (5)	−0.0013 (5)
C11	0.0139 (6)	0.0144 (7)	0.0185 (7)	0.0021 (5)	−0.0006 (5)	0.0005 (5)
C12	0.0234 (8)	0.0186 (7)	0.0196 (7)	−0.0017 (6)	0.0045 (6)	−0.0004 (6)
C13	0.0302 (8)	0.0199 (8)	0.0193 (8)	−0.0008 (6)	−0.0003 (6)	−0.0052 (6)
C14	0.0254 (8)	0.0164 (8)	0.0279 (8)	−0.0040 (6)	−0.0037 (6)	−0.0010 (6)
C15	0.0223 (8)	0.0193 (8)	0.0248 (8)	−0.0037 (6)	0.0042 (6)	0.0037 (6)
C16	0.0223 (7)	0.0172 (7)	0.0177 (7)	0.0011 (6)	0.0010 (6)	−0.0006 (6)
C17	0.0135 (7)	0.0145 (7)	0.0127 (6)	0.0012 (5)	−0.0006 (5)	0.0020 (5)

Geometric parameters (Å, º)

S1—C8	1.6778 (14)	C5—C6	1.390 (2)
O1—C7	1.2242 (18)	C5—H5	0.9300
O2—C17	1.2245 (18)	C6—C7	1.4956 (19)
O3—C17	1.3108 (17)	C9—C17	1.5172 (19)
O3—H3A	0.87 (2)	C9—C10	1.5612 (19)
N1—C7	1.3787 (19)	C9—H9	0.9800
N1—C8	1.3977 (18)	C10—C11	1.5114 (19)
N1—H1A	0.79 (2)	C10—H10A	0.9700
N2—C8	1.3240 (19)	C10—H10B	0.9700
N2—C9	1.4522 (18)	C11—C16	1.389 (2)
N2—H2A	0.85 (2)	C11—C12	1.395 (2)
C1—C2	1.388 (2)	C12—C13	1.389 (2)
C1—C6	1.399 (2)	C12—H12	0.9300
C1—H1	0.9300	C13—C14	1.386 (2)

C2—C3	1.391 (2)	C13—H13	0.9300
C2—H2	0.9300	C14—C15	1.389 (2)
C3—C4	1.386 (2)	C14—H14	0.9300
C3—H3	0.9300	C15—C16	1.392 (2)
C4—C5	1.392 (2)	C15—H15	0.9300
C4—H4	0.9300	C16—H16	0.9300

C17—O3—H3A	108.5 (15)	N2—C9—C10	112.38 (11)
C7—N1—C8	127.33 (13)	C17—C9—C10	108.90 (11)
C7—N1—H1A	117.9 (13)	N2—C9—H9	109.6
C8—N1—H1A	114.4 (13)	C17—C9—H9	109.6
C8—N2—C9	123.71 (12)	C10—C9—H9	109.6
C8—N2—H2A	118.9 (13)	C11—C10—C9	113.41 (11)
C9—N2—H2A	117.4 (13)	C11—C10—H10A	108.9
C2—C1—C6	119.71 (15)	C9—C10—H10A	108.9
C2—C1—H1	120.1	C11—C10—H10B	108.9
C6—C1—H1	120.1	C9—C10—H10B	108.9
C1—C2—C3	120.12 (15)	H10A—C10—H10B	107.7
C1—C2—H2	119.9	C16—C11—C12	118.83 (14)
C3—C2—H2	119.9	C16—C11—C10	121.81 (13)
C4—C3—C2	120.10 (14)	C12—C11—C10	119.36 (13)
C4—C3—H3	120.0	C13—C12—C11	120.65 (14)
C2—C3—H3	120.0	C13—C12—H12	119.7
C3—C4—C5	120.15 (15)	C11—C12—H12	119.7
C3—C4—H4	119.9	C14—C13—C12	120.21 (15)
C5—C4—H4	119.9	C14—C13—H13	119.9
C6—C5—C4	119.81 (14)	C12—C13—H13	119.9
C6—C5—H5	120.1	C13—C14—C15	119.51 (14)
C4—C5—H5	120.1	C13—C14—H14	120.2
C5—C6—C1	120.05 (14)	C15—C14—H14	120.2
C5—C6—C7	121.81 (13)	C14—C15—C16	120.23 (15)
C1—C6—C7	118.11 (13)	C14—C15—H15	119.9
O1—C7—N1	123.14 (13)	C16—C15—H15	119.9
O1—C7—C6	121.74 (13)	C11—C16—C15	120.57 (14)
N1—C7—C6	115.12 (12)	C11—C16—H16	119.7
N2—C8—N1	116.39 (12)	C15—C16—H16	119.7
N2—C8—S1	124.28 (11)	O2—C17—O3	124.38 (13)
N1—C8—S1	119.32 (11)	O2—C17—C9	122.44 (12)
N2—C9—C17	106.66 (11)	O3—C17—C9	113.15 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86 (2)	1.98 (2)	2.6480 (16)	134.1 (16)
N1—H1*A*···S1^{i}	0.790 (19)	2.571 (19)	3.3496 (13)	168.9 (18)

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C16—H16···O1^{iii}	0.93	2.54	3.4026 (18)	155

Symmetry codes: (i) −*x*, −*y*+1, −*z*+2; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1.