CRYSTALLOGRAPHIC COMMUNICATIONS

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ISSN 2056-9890

Crystal structure of *N*-(propan-2-ylcarbamothioyl)benzamide

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Received 9 December 2014; accepted 11 December 2014

Edited by E. R. T. Tiekink, University of Malaya, Malaysia

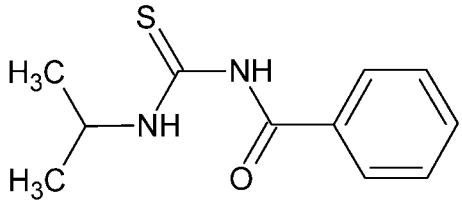
In the crystal structure of the title compound, $C_{11}H_{14}N_2OS$, the six atoms of the central C_2N_2OS residue are coplanar (r.m.s. deviation = 0.002 Å), which facilitates the formation of an intramolecular N—H···O hydrogen bond, which closes an *S*(6) loop. The terminal phenyl ring is inclined with respect to the central plane [dihedral angle = 42.10 (6)°]. The most prominent feature of the crystal packing is the formation of $\{\cdots HNCS\}_2$ synthons resulting in centrosymmetric dimers.

Keywords: crystal structure; thiourea; conformation; hydrogen bonding.

CCDC reference: 1038725

1. Related literature

For use of thioureas as building blocks in the synthesis of various organic compounds, see: Burgeson *et al.* (2012); Vega-Pérez *et al.* (2012); Yao *et al.* (2012); Shantharam *et al.* (2013); Yang *et al.* (2013). For use of thiourea-containing compounds in medicinal applications, see: Rodriguez-Fernandez *et al.* (2005); Rauf *et al.* (2012).



2. Experimental

2.1. Crystal data

$C_{11}H_{14}N_2OS$
M_r = 222.30
Monoclinic, $P2_1/c$
a = 11.2147 (4) Å
b = 5.3988 (2) Å
c = 19.6834 (7) Å
β = 102.031 (4)°
V = 1165.57 (7) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.27 mm^{-1}
T = 293 K
0.28 × 0.22 × 0.18 mm

2.2. Data collection

Agilent Xcalibur, Eos, Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.828, T_{max} = 1.000
3844 measured reflections
2189 independent reflections
1944 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.146
S = 1.08
2189 reflections
146 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···S1^{i}	0.81 (2)	2.66 (2)	3.4439 (19)	165 (2)
N2—H2*N*···O1	0.87 (2)	2.00 (3)	2.662 (2)	132 (2)

Symmetry code: (i) $-x+1, -y+2, -z$.

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2014* (Gruene *et al.*, 2014); program(s) used to refine structure: *SHELXL2014* (Gruene *et al.*, 2014); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009).

Acknowledgements

JPJ acknowledges the NSF–MRI program (grant No. CHE-1039027) for funds to purchase the X-ray diffractometer. SKM would like to thank Keene State College for providing the X-ray data and Manchester Metropolitan University for supporting this study.

Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5351).

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supporting information

Acta Cryst. (2015). E**71**, o56–o57 [doi:10.1107/S2056989014027133]

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S1. Structural commentary

Compounds containing thiourea linkage are very useful building blocks for the synthesis of a wide range of multiheterocyclic and macromolecular compounds. Thioureas have proved to be useful substances in drug research in recent years (Burgeson *et al.*, 2012; Vega-Pérez *et al.*, 2012; Yao *et al*., 2012; Shantharam *et al.*, 2013; Yang *et al.*, 2013). Symmetrical and unsymmetrical thioureas have shown anti-fungal activity against the plant pathogens like *Penicillium expansum* and *Fusarium oxysporu*m (Rodriguez-Fernandez *et al.*, 2005). Also, 1,3-dialkyl or diaryl thioureas exhibited significant anti-fungal activity against *Pyricularia oryzae* and *Drechslera oryzae* (Rauf *et al.*, 2012). In light of this, and following to our on-going study in synthesis of bio-active molecules, we report here the synthesis and crystal structure of the title compound.

In the structure of the title compound, Fig. 1, intramolecular N—H···O and intermolecular N—H···S interactions are noted (Table 1).

S2. Synthesis and crystallization

Freshly prepared benzoyl chloride 5 ml (0.043 mol) was added drop wise to a solution of 3.2 g (0.042 mol) of ammonium thiocyanate in 20 ml dry acetone with stirring. The reaction mixture was refluxed for 3 h. The obtained solid precipitate ammonium chloride was filtered off. The formed benzoyl isothiocyanate in the filtrate was added to a solution of 3.1 ml (0.0425 mol) of 2-amino-isopropane in 20 ml dry acetone. The reaction mixture was heated under reflux for 5 h, then poured into a beaker containing some ice cubes. The resulting precipitate was collected by filtration, washed several times with cold ethanol/water and purified by recrystallization from ethanol/dichloromethane mixture (1:1). Yield (63%); colourless solid, m.p 418 K.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.93 to 0.98 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = 1.2–1.5U_{eq}(C). The hydrogen atoms attached to N1 and N2 were found from difference Fourier maps and were refined with the distance contratin N—H = 0.86±0.02 Å with unrestrained U_{iso}.

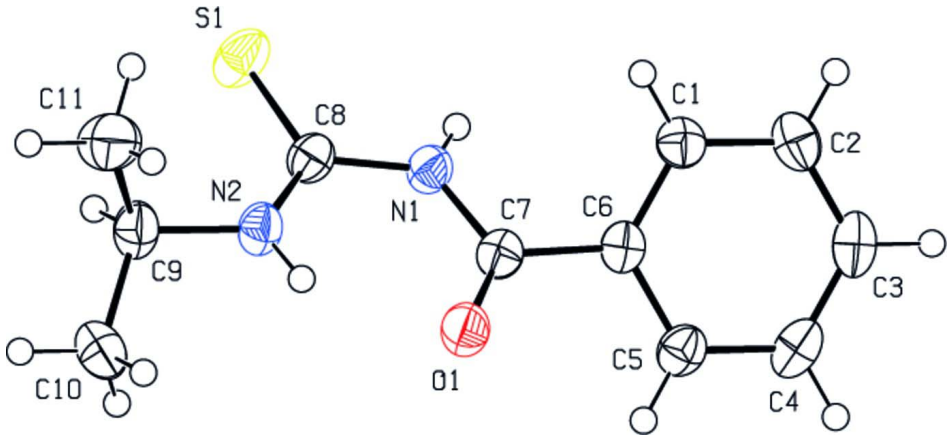

Figure 1

Perspective view of the title molecule with atom labeling scheme and 50% probability ellipsoids.

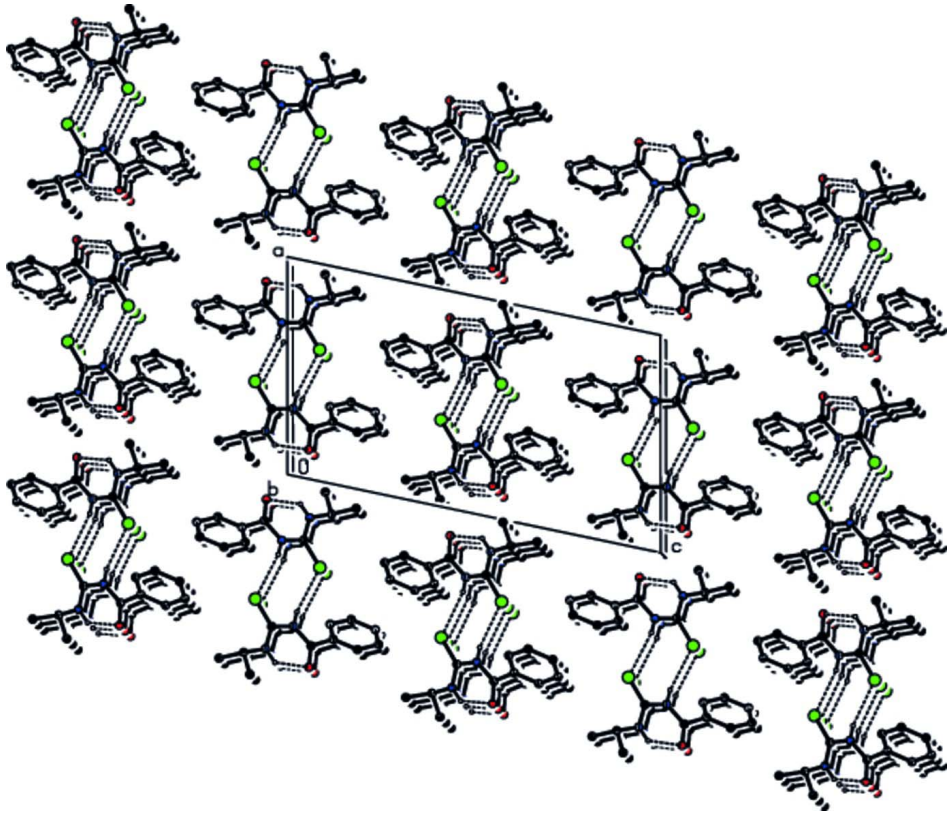

Figure 2

Packing viewed down the *b* axis showing stacks of pairs of molecules connected by N—H···S interactions.

N-(Propan-2-ylcarbamothioyl)benzamide

Crystal data

$C_{11}H_{14}N_2OS$	a = 11.2147 (4) Å
M_r = 222.30	b = 5.3988 (2) Å
Monoclinic, $P2_1/c$	c = 19.6834 (7) Å
Hall symbol: -P 2ybc	β = 102.031 (4)°

V = 1165.57 (7) Å^3
Z = 4
F(000) = 472
D_x = 1.267 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 1804 reflections

θ = 4.0–71.4°
μ = 2.27 mm^{-1}
T = 293 K
Prism, colourless
0.28 × 0.22 × 0.18 mm

Data collection

Agilent Xcalibur, Eos, Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.828, T_{max} = 1.000

3844 measured reflections
2189 independent reflections
1944 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 71.3°, θ_{min} = 4.0°
h = −12→13
k = −6→5
l = −18→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.146
S = 1.08
2189 reflections
146 parameters
2 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0871P)^2 + 0.3862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.60730 (5)	0.80543 (14)	0.08368 (3)	0.0464 (2)
O1	0.86531 (13)	1.0046 (3)	−0.05679 (8)	0.0392 (5)
N1	0.69070 (16)	1.0345 (3)	−0.01498 (9)	0.0321 (5)
N2	0.82704 (16)	0.7717 (3)	0.05595 (9)	0.0337 (5)
C1	0.63795 (19)	1.4665 (4)	−0.10695 (11)	0.0334 (6)
C2	0.5930 (2)	1.6272 (4)	−0.16095 (12)	0.0392 (6)
C3	0.6171 (2)	1.5865 (4)	−0.22650 (11)	0.0414 (7)
C4	0.6865 (2)	1.3853 (5)	−0.23779 (11)	0.0402 (7)
C5	0.73328 (19)	1.2248 (4)	−0.18386 (11)	0.0342 (6)
C6	0.70827 (17)	1.2641 (4)	−0.11803 (10)	0.0293 (5)
C7	0.76319 (18)	1.0900 (4)	−0.06135 (10)	0.0310 (6)
C8	0.71668 (19)	0.8697 (4)	0.04084 (10)	0.0322 (6)

C9	0.8684 (2)	0.5923 (4)	0.11200 (11)	0.0389 (7)
C10	0.9666 (2)	0.4305 (4)	0.09293 (13)	0.0452 (7)
C11	0.9154 (3)	0.7308 (6)	0.17988 (13)	0.0592 (9)
H1	0.62120	1.49370	−0.06320	0.0400*
H1N	0.6216 (16)	1.085 (4)	−0.0236 (11)	0.022 (5)*
H2	0.54640	1.76300	−0.15340	0.0470*
H2N	0.880 (2)	0.814 (5)	0.0317 (14)	0.048 (8)*
H3	0.58660	1.69470	−0.26270	0.0500*
H4	0.70190	1.35740	−0.28180	0.0480*
H5	0.78120	1.09110	−0.19140	0.0410*
H9	0.79950	0.48790	0.11730	0.0470*
H10A	0.93400	0.34290	0.05070	0.0680*
H10B	0.99470	0.31390	0.12960	0.0680*
H10C	1.03360	0.53220	0.08640	0.0680*
H11A	0.98180	0.83660	0.17480	0.0890*
H11B	0.94320	0.61370	0.21650	0.0890*
H11C	0.85090	0.82920	0.19110	0.0890*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0297 (3)	0.0743 (5)	0.0364 (3)	0.0061 (2)	0.0096 (2)	0.0129 (3)
O1	0.0309 (8)	0.0465 (9)	0.0415 (8)	0.0072 (6)	0.0106 (6)	0.0113 (7)
N1	0.0278 (8)	0.0390 (9)	0.0293 (8)	0.0046 (7)	0.0056 (6)	0.0054 (7)
N2	0.0306 (9)	0.0383 (10)	0.0314 (9)	0.0010 (7)	0.0046 (7)	0.0076 (7)
C1	0.0350 (10)	0.0309 (10)	0.0331 (10)	−0.0033 (8)	0.0041 (8)	−0.0023 (8)
C2	0.0400 (11)	0.0299 (10)	0.0448 (12)	−0.0012 (9)	0.0019 (9)	0.0025 (9)
C3	0.0418 (12)	0.0405 (12)	0.0377 (11)	−0.0059 (9)	−0.0014 (9)	0.0124 (9)
C4	0.0369 (11)	0.0544 (14)	0.0291 (10)	−0.0081 (10)	0.0064 (8)	0.0062 (9)
C5	0.0284 (10)	0.0408 (11)	0.0341 (10)	−0.0027 (8)	0.0080 (8)	0.0032 (8)
C6	0.0260 (9)	0.0304 (9)	0.0300 (9)	−0.0055 (7)	0.0022 (7)	0.0023 (8)
C7	0.0321 (10)	0.0314 (10)	0.0286 (9)	−0.0038 (8)	0.0043 (7)	−0.0007 (8)
C8	0.0336 (10)	0.0363 (10)	0.0255 (9)	−0.0020 (8)	0.0033 (7)	−0.0004 (8)
C9	0.0367 (11)	0.0412 (12)	0.0379 (11)	0.0022 (9)	0.0059 (9)	0.0114 (9)
C10	0.0487 (13)	0.0347 (11)	0.0506 (13)	0.0075 (10)	0.0065 (10)	0.0043 (10)
C11	0.0607 (16)	0.079 (2)	0.0334 (12)	0.0280 (14)	−0.0003 (11)	0.0010 (12)

Geometric parameters (Å, º)

S1—C8	1.664 (2)	C9—C11	1.526 (3)
O1—C7	1.220 (3)	C9—C10	1.513 (3)
N1—C7	1.376 (3)	C1—H1	0.9300
N1—C8	1.397 (3)	C2—H2	0.9300
N2—C8	1.322 (3)	C3—H3	0.9300
N2—C9	1.469 (3)	C4—H4	0.9300
C1—C2	1.383 (3)	C5—H5	0.9300
C1—C6	1.391 (3)	C9—H9	0.9800
N1—H1N	0.806 (19)	C10—H10A	0.9600

N2—H2N	0.87 (2)	C10—H10B	0.9600
C2—C3	1.390 (3)	C10—H10C	0.9600
C3—C4	1.381 (3)	C11—H11A	0.9600
C4—C5	1.386 (3)	C11—H11B	0.9600
C5—C6	1.398 (3)	C11—H11C	0.9600
C6—C7	1.490 (3)		

C7—N1—C8	127.26 (18)	C6—C1—H1	120.00
C8—N2—C9	124.57 (18)	C1—C2—H2	120.00
C2—C1—C6	120.0 (2)	C3—C2—H2	120.00
C7—N1—H1N	117.5 (15)	C2—C3—H3	120.00
C8—N1—H1N	114.5 (15)	C4—C3—H3	120.00
C1—C2—C3	120.3 (2)	C3—C4—H4	120.00
C8—N2—H2N	119.2 (17)	C5—C4—H4	120.00
C9—N2—H2N	116.2 (17)	C4—C5—H5	120.00
C2—C3—C4	119.9 (2)	C6—C5—H5	120.00
C3—C4—C5	120.3 (2)	N2—C9—H9	109.00
C4—C5—C6	119.9 (2)	C10—C9—H9	109.00
C1—C6—C7	122.51 (18)	C11—C9—H9	109.00
C5—C6—C7	117.82 (18)	C9—C10—H10A	109.00
C1—C6—C5	119.63 (19)	C9—C10—H10B	110.00
O1—C7—N1	122.96 (19)	C9—C10—H10C	109.00
O1—C7—C6	121.90 (18)	H10A—C10—H10B	110.00
N1—C7—C6	115.14 (17)	H10A—C10—H10C	109.00
S1—C8—N1	118.47 (16)	H10B—C10—H10C	109.00
S1—C8—N2	123.87 (16)	C9—C11—H11A	110.00
N1—C8—N2	117.65 (19)	C9—C11—H11B	109.00
N2—C9—C11	109.39 (19)	C9—C11—H11C	109.00
C10—C9—C11	111.3 (2)	H11A—C11—H11B	109.00
N2—C9—C10	109.06 (18)	H11A—C11—H11C	109.00
C2—C1—H1	120.00	H11B—C11—H11C	110.00

C7—N1—C8—N2	7.1 (3)	C2—C1—C6—C7	177.6 (2)
C7—N1—C8—S1	−172.18 (17)	C1—C2—C3—C4	−0.2 (3)
C8—N1—C7—O1	−3.3 (3)	C2—C3—C4—C5	−0.6 (3)
C8—N1—C7—C6	176.95 (19)	C3—C4—C5—C6	1.2 (3)
C9—N2—C8—S1	0.4 (3)	C4—C5—C6—C7	−178.5 (2)
C9—N2—C8—N1	−178.83 (18)	C4—C5—C6—C1	−0.9 (3)
C8—N2—C9—C10	151.8 (2)	C1—C6—C7—O1	−140.9 (2)
C8—N2—C9—C11	−86.2 (3)	C5—C6—C7—O1	36.7 (3)
C6—C1—C2—C3	0.4 (3)	C5—C6—C7—N1	−143.55 (19)
C2—C1—C6—C5	0.1 (3)	C1—C6—C7—N1	38.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···S1^{i}	0.81 (2)	2.66 (2)	3.4439 (19)	165 (2)

N2—H2*N*···O1	0.87 (2)	2.00 (3)	2.662 (2)	132 (2)

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