Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-[*N*-(2-Pyridylmethyl)thiocarbamoyl]-benzamide

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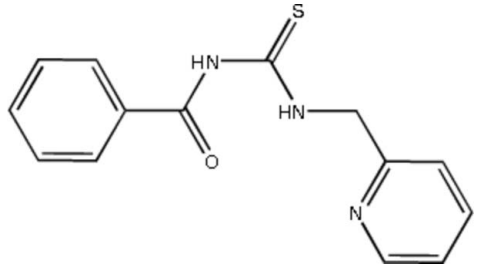
Received 29 October 2007; accepted 10 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.057; wR factor = 0.171; data-to-parameter ratio = 13.6.

In the title compound, $C_{14}H_{13}N_3OS$, the phenyl ring forms a dihedral angle of 33.22 (12)° with the remaining part of the molecule, which is approximately planar. The *trans–cis* geometry of the thiourea fragment is stabilized by an intramolecular N—H···O hydrogen bond involving the O atom of the benzoyl group and the H atom of the *trans*-thioamide unit. Intermolecular N—H···S hydrogen bonds between the *cis*-thioamide fragments connect molecules into centrosymmetric dimers.

Related literature

For the crystal structure of the analogue in which pyridyl is replaced by phenyl, see: Sabino *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{13}N_3OS$
M_r = 271.33
Triclinic, $P\overline{1}$
a = 8.533 (3) Å
b = 8.813 (4) Å
c = 9.028 (4) Å
α = 98.904 (6)°
β = 96.978 (8)°
γ = 90.197 (7)°
V = 665.6 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 298 (2) K
0.50 × 0.34 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.890, T_{max} = 0.953
6204 measured reflections
2336 independent reflections
2172 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.171
S = 1.37
2336 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1	0.86	1.96	2.641 (4)	135
N2—H2···N3	0.86	2.24	2.644 (4)	109
N1—H1···S1^{i}	0.86	2.60	3.426 (3)	162

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

The authors thank the Ministry of Higher Education of Malaysia for a Fundamental Research Grant (No. UKM-OUP-BTT-28/2007) and Universiti Kebangsaan Malaysian for research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2114).

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  doi:10.1107/S1600536807057789

supplementary materials

Acta Cryst. (2007). E**63**, o4842 [**doi:10.1107/S1600536807057789**]

N-[*N*-(2-Pyridylmethyl)thiocarbamoyl]benzamide

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Comment

The title compound (I) is an analog of *N*-(benzylaminothiocarbonyl)benzamide (II) (Sabino *et al.*, 2006), which like the title compound has the tiourea fragment in *trans-cis* configuration (Fig.1). The bond lengths and angles are in normal ranges. In contrast to (II), the molecule of (I) is more planar with only the phenyl ring of the benzoyl group twisted with respect to the plane defined by the remaining atoms of the molecule. This planar conformation is due to two intramolecular hydrogen bonds, one relatively strong (N2—H2···O1) and one weak (N2—H2···N3). The N1—H1···S1 hydrogen bonds between the *cis*-thioamide fragments of (I) connect molecules into centrosymmetric dimers (Fig.2).

Experimental

The mixture of benzoyl chloride (7.03 g, 0.05 mol), ammonium thiocyanate (3.8 g, 0.05 mol) and 2-picolylamine (5.4 g, 0.05 mol) in 30 ml dry acetone was refluxed with stirring for 4 h. The solution was filtered and left to evaporate at room temperature. The colourless solid obtained after a few days was washed with water and cold ethanol (yield 80%; m.p 424.4- 427.2 K). Crystals suitablefor X-ray investigation were obtained by recrystallization from chloroform.

Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å, N—H = 0.86Å and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(parent atom).

Figures

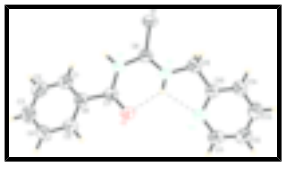

Fig. 1. The molecular structure of (I), with displacement ellipsods are drawn at the 50% probability level.

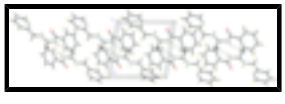

Fig. 2. A packing diagram of (I) viewed down the *a* axis. Hydrogen bonds are shown by dashed lines.

N-[*N*-(2-Pyridylmethyl)thiocarbamoyl]benzamide

Crystal data

$C_{14}H_{13}N_3OS$	$Z = 2$
$M_r = 271.33$	$F_{000} = 284$
Triclinic, $P\overline{1}$	$D_x = 1.354$ Mg m^{-3}

Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.533 (3) Å	Cell parameters from 3526 reflections
b = 8.813 (4) Å	θ = 2.3–25.0º
c = 9.028 (4) Å	μ = 0.24 mm^{-1}
α = 98.904 (6)º	T = 298 (2) K
β = 96.978 (8)º	Block, colourless
γ = 90.197 (7)º	0.50 × 0.34 × 0.20 mm
V = 665.6 (4) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2336 independent reflections
Radiation source: fine-focus sealed tube	2172 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
Detector resolution: 83.66 pixels mm^{-1}	θ_{max} = 25.0º
T = 298(2) K	θ_{min} = 2.3º
ω scan	h = −10→10
Absorption correction: multi-scan (SADABS; Bruker, 2000)	k = −10→10
T_{min} = 0.890, T_{max} = 0.953	l = −10→10
6204 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	H-atom parameters constrained
$wR(F^2)$ = 0.171	$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.2921P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.37	$(\Delta/\sigma)_{max}$ = 0.001
2336 reflections	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
172 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	1.04589 (10)	0.52568 (10)	0.23600 (9)	0.0589 (3)
O1	0.6410 (3)	0.8417 (3)	0.1405 (2)	0.0636 (7)
N1	0.8063 (3)	0.6462 (3)	0.0833 (3)	0.0485 (6)
H1	0.8340	0.5833	0.0088	0.058*
N2	0.8620 (3)	0.7491 (3)	0.3356 (3)	0.0471 (6)
H2	0.7860	0.8104	0.3189	0.056*
N3	0.7395 (4)	0.9462 (4)	0.5396 (3)	0.0676 (9)
C1	0.5694 (4)	0.5341 (4)	−0.1708 (3)	0.0497 (7)
H1A	0.6330	0.4600	−0.1329	0.060*
C2	0.4673 (4)	0.4948 (4)	−0.3016 (4)	0.0597 (9)
H2A	0.4621	0.3941	−0.3518	0.072*
C3	0.3730 (4)	0.6038 (5)	−0.3584 (4)	0.0656 (10)
H3	0.3039	0.5767	−0.4466	0.079*
C4	0.3810 (4)	0.7525 (5)	−0.2849 (4)	0.0654 (10)
H4	0.3181	0.8265	−0.3241	0.078*
C5	0.4815 (4)	0.7924 (4)	−0.1536 (4)	0.0552 (8)
H5	0.4851	0.8930	−0.1032	0.066*
C6	0.5777 (3)	0.6835 (3)	−0.0957 (3)	0.0439 (7)
C7	0.6763 (4)	0.7318 (3)	0.0517 (3)	0.0455 (7)
C8	0.8989 (3)	0.6492 (3)	0.2221 (3)	0.0441 (7)
C9	0.9426 (4)	0.7609 (4)	0.4868 (3)	0.0524 (8)
H9A	1.0525	0.7893	0.4872	0.063*
H9B	0.9387	0.6620	0.5208	0.063*
C10	0.8672 (4)	0.8796 (3)	0.5936 (3)	0.0468 (7)
C11	0.6733 (5)	1.0521 (5)	0.6354 (4)	0.0749 (11)
H11	0.5833	1.0999	0.5979	0.090*
C12	0.7290 (4)	1.0929 (4)	0.7814 (4)	0.0638 (9)
H12	0.6788	1.1662	0.8437	0.077*
C13	0.8605 (6)	1.0240 (6)	0.8352 (4)	0.0936 (16)
H13	0.9033	1.0503	0.9358	0.112*
C14	0.9302 (5)	0.9150 (5)	0.7405 (4)	0.0830 (13)
H14	1.0199	0.8658	0.7766	0.100*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0578 (5)	0.0722 (6)	0.0469 (5)	0.0331 (4)	0.0069 (4)	0.0094 (4)
O1	0.0709 (15)	0.0614 (14)	0.0520 (13)	0.0340 (12)	−0.0037 (11)	−0.0030 (11)
N1	0.0484 (14)	0.0553 (15)	0.0395 (13)	0.0201 (12)	0.0043 (11)	0.0010 (11)
N2	0.0449 (13)	0.0522 (14)	0.0421 (13)	0.0165 (11)	0.0020 (10)	0.0036 (11)
N3	0.0681 (19)	0.077 (2)	0.0521 (16)	0.0328 (16)	−0.0010 (14)	−0.0019 (14)
C1	0.0469 (17)	0.0496 (17)	0.0531 (17)	0.0073 (14)	0.0109 (14)	0.0060 (14)

C2	0.0520 (19)	0.063 (2)	0.060 (2)	−0.0034 (16)	0.0101 (16)	−0.0051 (16)
C3	0.0518 (19)	0.090 (3)	0.0504 (19)	0.0006 (18)	−0.0022 (15)	0.0017 (18)
C4	0.060 (2)	0.081 (2)	0.055 (2)	0.0182 (18)	−0.0033 (16)	0.0170 (18)
C5	0.0586 (19)	0.0554 (19)	0.0499 (17)	0.0167 (15)	0.0013 (14)	0.0075 (14)
C6	0.0423 (15)	0.0501 (17)	0.0410 (15)	0.0089 (13)	0.0091 (12)	0.0086 (13)
C7	0.0473 (16)	0.0459 (16)	0.0440 (16)	0.0142 (13)	0.0075 (13)	0.0073 (13)
C8	0.0431 (15)	0.0468 (16)	0.0435 (15)	0.0075 (13)	0.0080 (12)	0.0086 (13)
C9	0.0515 (17)	0.0586 (19)	0.0452 (16)	0.0154 (15)	0.0003 (13)	0.0068 (14)
C10	0.0455 (16)	0.0481 (16)	0.0457 (16)	0.0066 (13)	0.0044 (13)	0.0046 (13)
C11	0.075 (2)	0.078 (3)	0.067 (2)	0.036 (2)	0.0035 (19)	−0.0020 (19)
C12	0.067 (2)	0.062 (2)	0.059 (2)	0.0136 (18)	0.0142 (17)	−0.0054 (16)
C13	0.101 (3)	0.118 (4)	0.048 (2)	0.043 (3)	−0.009 (2)	−0.018 (2)
C14	0.077 (3)	0.104 (3)	0.055 (2)	0.044 (2)	−0.0145 (19)	−0.012 (2)

Geometric parameters (Å, °)

S1—C8	1.670 (3)	C4—C5	1.374 (5)
O1—C7	1.221 (3)	C4—H4	0.9300
N1—C7	1.370 (4)	C5—C6	1.388 (4)
N1—C8	1.394 (4)	C5—H5	0.9300
N1—H1	0.8600	C6—C7	1.487 (4)
N2—C8	1.314 (4)	C9—C10	1.510 (4)
N2—C9	1.440 (4)	C9—H9A	0.9700
N2—H2	0.8600	C9—H9B	0.9700
N3—C10	1.317 (4)	C10—C14	1.357 (5)
N3—C11	1.345 (4)	C11—C12	1.339 (5)
C1—C2	1.376 (5)	C11—H11	0.9300
C1—C6	1.382 (4)	C12—C13	1.352 (5)
C1—H1A	0.9300	C12—H12	0.9300
C2—C3	1.375 (5)	C13—C14	1.372 (5)
C2—H2A	0.9300	C13—H13	0.9300
C3—C4	1.372 (5)	C14—H14	0.9300
C3—H3	0.9300		
C7—N1—C8	127.6 (2)	O1—C7—C6	121.1 (3)
C7—N1—H1	116.2	N1—C7—C6	116.9 (2)
C8—N1—H1	116.2	N2—C8—N1	117.0 (2)
C8—N2—C9	123.3 (2)	N2—C8—S1	123.9 (2)
C8—N2—H2	118.4	N1—C8—S1	119.1 (2)
C9—N2—H2	118.4	N2—C9—C10	110.8 (2)
C10—N3—C11	117.6 (3)	N2—C9—H9A	109.5
C2—C1—C6	120.1 (3)	C10—C9—H9A	109.5
C2—C1—H1A	119.9	N2—C9—H9B	109.5
C6—C1—H1A	119.9	C10—C9—H9B	109.5
C3—C2—C1	120.3 (3)	H9A—C9—H9B	108.1
C3—C2—H2A	119.9	N3—C10—C14	121.7 (3)
C1—C2—H2A	119.9	N3—C10—C9	117.9 (3)
C4—C3—C2	119.9 (3)	C14—C10—C9	120.4 (3)
C4—C3—H3	120.0	C12—C11—N3	124.0 (3)
C2—C3—H3	120.0	C12—C11—H11	118.0

C3—C4—C5	120.2 (3)	N3—C11—H11	118.0
C3—C4—H4	119.9	C11—C12—C13	117.8 (3)
C5—C4—H4	119.9	C11—C12—H12	121.1
C4—C5—C6	120.4 (3)	C13—C12—H12	121.1
C4—C5—H5	119.8	C12—C13—C14	119.5 (4)
C6—C5—H5	119.8	C12—C13—H13	120.2
C1—C6—C5	119.1 (3)	C14—C13—H13	120.2
C1—C6—C7	123.1 (3)	C10—C14—C13	119.4 (3)
C5—C6—C7	117.6 (3)	C10—C14—H14	120.3
O1—C7—N1	122.0 (3)	C13—C14—H14	120.3
C6—C1—C2—C3	0.1 (5)	C9—N2—C8—S1	−1.5 (4)
C1—C2—C3—C4	0.2 (5)	C7—N1—C8—N2	−2.7 (5)
C2—C3—C4—C5	−0.8 (6)	C7—N1—C8—S1	176.1 (3)
C3—C4—C5—C6	1.1 (6)	C8—N2—C9—C10	−176.7 (3)
C2—C1—C6—C5	0.2 (5)	C11—N3—C10—C14	0.4 (6)
C2—C1—C6—C7	175.4 (3)	C11—N3—C10—C9	−179.9 (3)
C4—C5—C6—C1	−0.8 (5)	N2—C9—C10—N3	3.8 (4)
C4—C5—C6—C7	−176.2 (3)	N2—C9—C10—C14	−176.4 (4)
C8—N1—C7—O1	12.3 (5)	C10—N3—C11—C12	−0.3 (7)
C8—N1—C7—C6	−166.9 (3)	N3—C11—C12—C13	0.5 (7)
C1—C6—C7—O1	−152.7 (3)	C11—C12—C13—C14	−0.8 (8)
C5—C6—C7—O1	22.5 (5)	N3—C10—C14—C13	−0.7 (7)
C1—C6—C7—N1	26.5 (4)	C9—C10—C14—C13	179.5 (4)
C5—C6—C7—N1	−158.3 (3)	C12—C13—C14—C10	0.9 (8)
C9—N2—C8—N1	177.3 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.86	1.96	2.641 (4)	135
N2—H2···N3	0.86	2.24	2.644 (4)	109
N1—H1···S1^{i}	0.86	2.60	3.426 (3)	162

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

Fig. 1

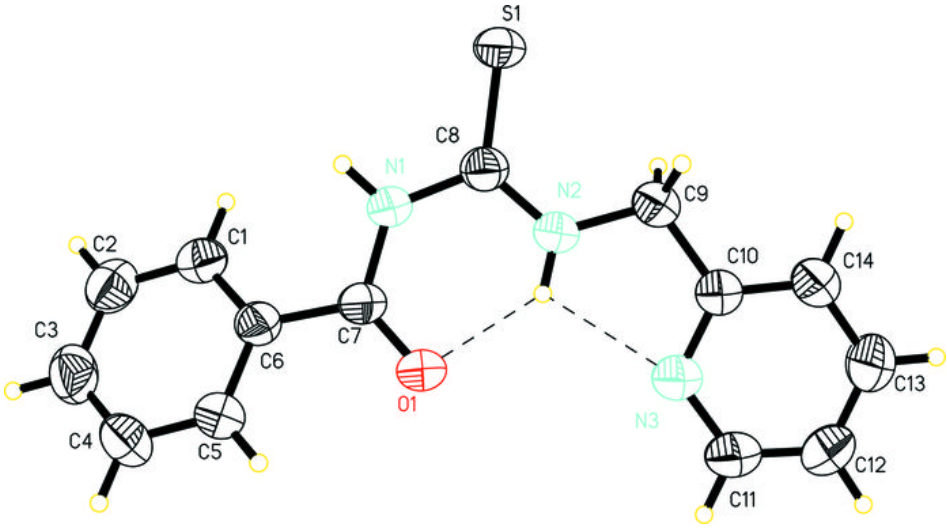

Fig. 2