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N-(2-Furoyl)-*N*′-(6-methyl-2-pyridyl)thiourea

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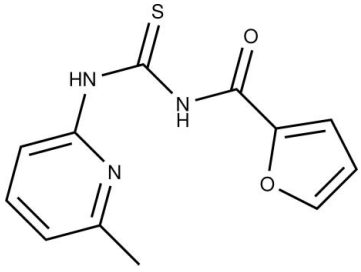
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.130; data-to-parameter ratio = 14.4.

The title molecule, $C_{12}H_{11}N_3O_2S$, adopts a *cis–trans* configuration of the furoyl and 6-methyl-2-pyridyl groups with respect to the thiono S atom across the thiourea C—N bonds. Intramolecular N—H···O, N—H···N and C—H···O hydrogen bonds contribute to the essential planarity of the molecular skeleton. In the crystal structure, weak intermolecular N—H···S hydrogen bonds link the molecules into centrosymmetric dimers.

Related literature

For related crystal structures, see: Yusof, Tajuddin *et al.* (2006); Yusof, Soh *et al.* (2006); Yusof *et al.* (2007).



Experimental

Crystal data

$C_{12}H_{11}N_3O_2S$
M_r = 261.30
Monoclinic, $P2_1/c$
a = 7.3189 (16) Å
b = 18.187 (4) Å
c = 9.534 (2) Å
β = 108.482 (4)°
V = 1203.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 298 (2) K
0.42 × 0.32 × 0.31 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.896, T_{max} = 0.922
6575 measured reflections
2352 independent reflections
1959 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
S = 0.97
2352 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*A*···O1	0.86	2.26	2.685 (2)	111
N1—H1*A*···N3	0.86	1.93	2.645 (2)	140
C13—H13*A*···O1	0.96	2.56	3.490 (3)	163
N2—H2*A*···S1^{i}	0.86	2.56	3.4118 (19)	169

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2308).

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supplementary materials

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N-(2-Furoyl)-*N*'-(6-methyl-2-pyridyl)thiourea

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Comment

The title compound, (I), is similar to 1-(3-methylbenzoyl)-3-(6-methyl-2-pyridyl)-thiourea, (Yusof, Tajuddin *et al.*, 2006*a*), except that the 3-methylbenzoyl group is replaced by furoyl (Fig. 1). The molecule also adopts *cis-trans* configuration with respect to the position of the furoyl and 6-methyl-pyridine-2-yl groups relative to the thiono S1 atom across their C—N bonds, respectively. The bond lengths and angles in (I) and comparable with those observed in other thiourea derivatives (Yusof, Soh *et al.*, 2006*b*; Yusof *et al.*, 2007). The (6-methyl-pyridine-2-yl)thiourea (S1/N1—N3/C6—C7/C9—C13) and furoyl (O1/O2/C1—C5) fragments are essentially planar with a maximum deviation of 0.066 (1)Å for atom S1 from the least-squares plane. The dihedral angle between the planes is 13.79 (10)°.

There are three intramolecular hydrogen bonds, N—H···N, N—H···O, and C—H···O (Table 1), forming two pseudo-six- and a pseudo-five-membered rings. In the crystal structure, the weak intermolecular N—H···S hydrogen bonds (Table 1) link the molecules into centrosymmetric dimers (Fig. 2).

Experimental

To a stirring acetone solution (75 ml) of 2-furoyl chloride (2.0 g, 15 mmol) and ammoniumthiocyanate (1.17 g, 15 mmol), 2-amino-6-methylpyridine (1.66 g, 15 mmol) in 40 ml of acetone was added dropwise. The solution mixture was refluxed for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered off and washed with distilled water and cold ethanol before dried under vacuo. Good quality crystals were obtained by recrystallization from DMSO. Yield 82% (2.31 g).

Refinement

After their location in the difference map, all H-atoms were fixed geometrically at ideal positions (C—H 0.93–0.97 Å, N—H = 0.86 Å), and allowed to ride on the parent atoms with U_{iso}(H)= 1.2–1.5U_{eq}(C, N)

Figures

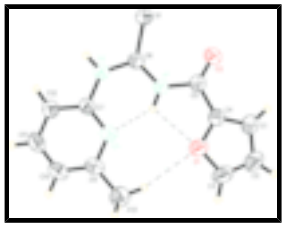

Fig. 1. The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

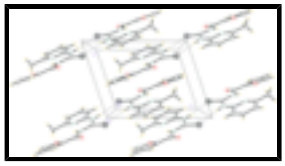

Fig. 2. Packing diagram viewed down the *b* axis. The dashed lines denote the N—H···S hydrogen bonds.

_N_-(2-Furoyl)-_N_'-(6-methyl-2-pyridyl)thiourea

Crystal data

$C_{12}H_{11}N_3O_2S$	F_{000} = 544
M_r = 261.30	D_x = 1.442 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1001 reflections
a = 7.3189 (16) Å	θ = 2.2–26º
b = 18.187 (4) Å	μ = 0.27 mm^{-1}
c = 9.534 (2) Å	T = 298 (2) K
β = 108.482 (4)º	Block, colourless
V = 1203.6 (4) Å^3	0.42 × 0.32 × 0.31 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2352 independent reflections
Radiation source: fine-focus sealed tube	1959 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.018
Detector resolution: 83.66 pixels mm^{-1}	θ_{max} = 26.0º
T = 298(2) K	θ_{min} = 2.2º
ω scans	h = −9→9
Absorption correction: multi-scan (SADABS; Bruker, 2000)	k = −15→22
T_{min} = 0.896, T_{max} = 0.922	l = −10→11
6575 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
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H-atom parameters constrained
$wR(F^2)$ = 0.130	$w = 1/[\sigma^2(F_o^2) + (0.0777P)^2 + 0.5195P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.97	$(\Delta/\sigma)_{max} < 0.001$
2352 reflections	$\Delta\rho_{max}$ = 0.44 e Å^{-3}
163 parameters	$\Delta\rho_{min}$ = −0.29 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 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.16238 (9)	0.40704 (3)	0.10547 (5)	0.0557 (2)
O1	0.4366 (2)	0.43047 (8)	0.69045 (16)	0.0586 (4)
O2	0.3268 (3)	0.31529 (9)	0.37716 (17)	0.0668 (5)
N1	0.2635 (2)	0.43838 (9)	0.39697 (16)	0.0417 (4)
H1A	0.2757	0.4724	0.4620	0.050*
N2	0.0981 (2)	0.52668 (8)	0.23383 (17)	0.0443 (4)
H2A	0.0385	0.5377	0.1431	0.053*
C1	0.4787 (3)	0.30973 (13)	0.7043 (2)	0.0563 (6)
H1B	0.4811	0.2606	0.6777	0.068*
C2	0.5363 (4)	0.33824 (14)	0.8479 (3)	0.0639 (6)
H2B	0.5841	0.3118	0.9354	0.077*
C3	0.5096 (4)	0.41022 (14)	0.8351 (3)	0.0694 (7)
H3A	0.5370	0.4427	0.9145	0.083*
C4	0.4191 (3)	0.36716 (11)	0.6121 (2)	0.0427 (4)
C5	0.3350 (3)	0.36956 (10)	0.4507 (2)	0.0428 (4)
C6	0.1765 (3)	0.45834 (10)	0.2534 (2)	0.0401 (4)
C7	0.0966 (3)	0.58277 (10)	0.3350 (2)	0.0390 (4)
N3	0.1815 (2)	0.57018 (9)	0.47800 (17)	0.0416 (4)
C9	0.1824 (3)	0.62420 (11)	0.5746 (2)	0.0446 (5)
C10	0.0970 (3)	0.69141 (12)	0.5268 (3)	0.0531 (5)
H10A	0.0999	0.7284	0.5949	0.064*
C11	0.0080 (3)	0.70341 (12)	0.3784 (3)	0.0536 (5)
H11A	−0.0504	0.7484	0.3457	0.064*
C12	0.0058 (3)	0.64870 (11)	0.2788 (2)	0.0475 (5)
H12A	−0.0538	0.6554	0.1779	0.057*
C13	0.2778 (3)	0.60720 (14)	0.7347 (2)	0.0583 (6)
H13A	0.3273	0.5579	0.7447	0.087*
H13B	0.1856	0.6117	0.7869	0.087*
H13C	0.3817	0.6411	0.7754	0.087*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0802 (4)	0.0441 (3)	0.0339 (3)	0.0130 (3)	0.0054 (3)	−0.0058 (2)
O1	0.0881 (11)	0.0418 (8)	0.0379 (8)	−0.0036 (7)	0.0088 (7)	0.0007 (6)
O2	0.1077 (14)	0.0407 (8)	0.0467 (9)	0.0189 (8)	0.0171 (9)	−0.0029 (7)
N1	0.0557 (9)	0.0323 (8)	0.0330 (8)	0.0031 (7)	0.0084 (7)	−0.0022 (6)
N2	0.0580 (10)	0.0360 (8)	0.0322 (8)	0.0055 (7)	0.0050 (7)	−0.0005 (6)
C1	0.0697 (14)	0.0466 (12)	0.0497 (12)	0.0083 (10)	0.0149 (11)	0.0094 (10)
C2	0.0792 (16)	0.0652 (16)	0.0411 (12)	0.0048 (12)	0.0102 (11)	0.0149 (10)
C3	0.1002 (19)	0.0636 (16)	0.0343 (11)	−0.0034 (14)	0.0069 (12)	0.0001 (10)
C4	0.0485 (11)	0.0381 (10)	0.0414 (10)	0.0013 (8)	0.0142 (8)	0.0025 (8)
C5	0.0511 (11)	0.0361 (10)	0.0399 (10)	0.0041 (8)	0.0125 (8)	0.0018 (8)
C6	0.0455 (10)	0.0350 (9)	0.0364 (10)	−0.0005 (8)	0.0083 (8)	−0.0015 (7)
C7	0.0422 (10)	0.0330 (9)	0.0406 (10)	−0.0014 (7)	0.0116 (8)	−0.0022 (7)
N3	0.0465 (9)	0.0381 (8)	0.0383 (9)	−0.0010 (7)	0.0107 (7)	−0.0042 (6)
C9	0.0443 (11)	0.0443 (11)	0.0463 (11)	−0.0049 (8)	0.0158 (8)	−0.0098 (9)
C10	0.0591 (13)	0.0421 (11)	0.0598 (13)	−0.0001 (9)	0.0212 (10)	−0.0148 (10)
C11	0.0593 (13)	0.0360 (10)	0.0647 (14)	0.0070 (9)	0.0185 (11)	−0.0018 (9)
C12	0.0531 (12)	0.0402 (10)	0.0456 (11)	0.0048 (9)	0.0106 (9)	0.0011 (9)
C13	0.0646 (14)	0.0626 (14)	0.0451 (12)	−0.0004 (11)	0.0137 (10)	−0.0130 (10)

Geometric parameters (Å, °)

S1—C6	1.6669 (19)	C3—H3A	0.9300
O1—C4	1.356 (2)	C4—C5	1.466 (3)
O1—C3	1.363 (3)	C7—N3	1.326 (2)
O2—C5	1.201 (2)	C7—C12	1.393 (3)
N1—C6	1.363 (2)	N3—C9	1.345 (2)
N1—C5	1.390 (2)	C9—C10	1.383 (3)
N1—H1A	0.8600	C9—C13	1.496 (3)
N2—C6	1.357 (2)	C10—C11	1.375 (3)
N2—C7	1.407 (2)	C10—H10A	0.9300
N2—H2A	0.8600	C11—C12	1.372 (3)
C1—C4	1.345 (3)	C11—H11A	0.9300
C1—C2	1.399 (3)	C12—H12A	0.9300
C1—H1B	0.9300	C13—H13A	0.9600
C2—C3	1.323 (4)	C13—H13B	0.9600
C2—H2B	0.9300	C13—H13C	0.9600
C4—O1—C3	105.45 (18)	N1—C6—S1	125.78 (14)
C6—N1—C5	127.69 (16)	N3—C7—C12	123.70 (17)
C6—N1—H1A	116.2	N3—C7—N2	118.49 (16)
C5—N1—H1A	116.2	C12—C7—N2	117.82 (17)
C6—N2—C7	131.65 (16)	C7—N3—C9	118.47 (17)
C6—N2—H2A	114.2	N3—C9—C10	121.1 (2)
C7—N2—H2A	114.2	N3—C9—C13	116.57 (19)
C4—C1—C2	106.6 (2)	C10—C9—C13	122.34 (19)

C4—C1—H1B	126.7	C11—C10—C9	119.78 (19)
C2—C1—H1B	126.7	C11—C10—H10A	120.1
C3—C2—C1	106.6 (2)	C9—C10—H10A	120.1
C3—C2—H2B	126.7	C12—C11—C10	119.7 (2)
C1—C2—H2B	126.7	C12—C11—H11A	120.2
C2—C3—O1	111.1 (2)	C10—C11—H11A	120.2
C2—C3—H3A	124.5	C11—C12—C7	117.29 (19)
O1—C3—H3A	124.5	C11—C12—H12A	121.4
C1—C4—O1	110.18 (19)	C7—C12—H12A	121.4
C1—C4—C5	130.6 (2)	C9—C13—H13A	109.5
O1—C4—C5	119.19 (17)	C9—C13—H13B	109.5
O2—C5—N1	125.52 (18)	H13A—C13—H13B	109.5
O2—C5—C4	121.01 (18)	C9—C13—H13C	109.5
N1—C5—C4	113.43 (16)	H13A—C13—H13C	109.5
N2—C6—N1	115.14 (16)	H13B—C13—H13C	109.5
N2—C6—S1	119.07 (14)		
C4—C1—C2—C3	−0.2 (3)	C5—N1—C6—N2	171.73 (18)
C1—C2—C3—O1	0.3 (3)	C5—N1—C6—S1	−9.0 (3)
C4—O1—C3—C2	−0.2 (3)	C6—N2—C7—N3	−1.4 (3)
C2—C1—C4—O1	0.1 (3)	C6—N2—C7—C12	178.5 (2)
C2—C1—C4—C5	−176.6 (2)	C12—C7—N3—C9	−0.8 (3)
C3—O1—C4—C1	0.0 (3)	N2—C7—N3—C9	179.10 (16)
C3—O1—C4—C5	177.2 (2)	C7—N3—C9—C10	0.0 (3)
C6—N1—C5—O2	−1.8 (4)	C7—N3—C9—C13	179.34 (17)
C6—N1—C5—C4	−179.67 (18)	N3—C9—C10—C11	0.6 (3)
C1—C4—C5—O2	−7.6 (4)	C13—C9—C10—C11	−178.7 (2)
O1—C4—C5—O2	175.9 (2)	C9—C10—C11—C12	−0.5 (3)
C1—C4—C5—N1	170.4 (2)	C10—C11—C12—C7	−0.3 (3)
O1—C4—C5—N1	−6.1 (3)	N3—C7—C12—C11	0.9 (3)
C7—N2—C6—N1	3.1 (3)	N2—C7—C12—C11	−178.99 (18)
C7—N2—C6—S1	−176.21 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1	0.86	2.26	2.685 (2)	111
N1—H1A···N3	0.86	1.93	2.645 (2)	140
C13—H13A···O1	0.96	2.56	3.490 (3)	163
N2—H2A···S1^{i}	0.86	2.56	3.4118 (19)	169

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

Fig. 1

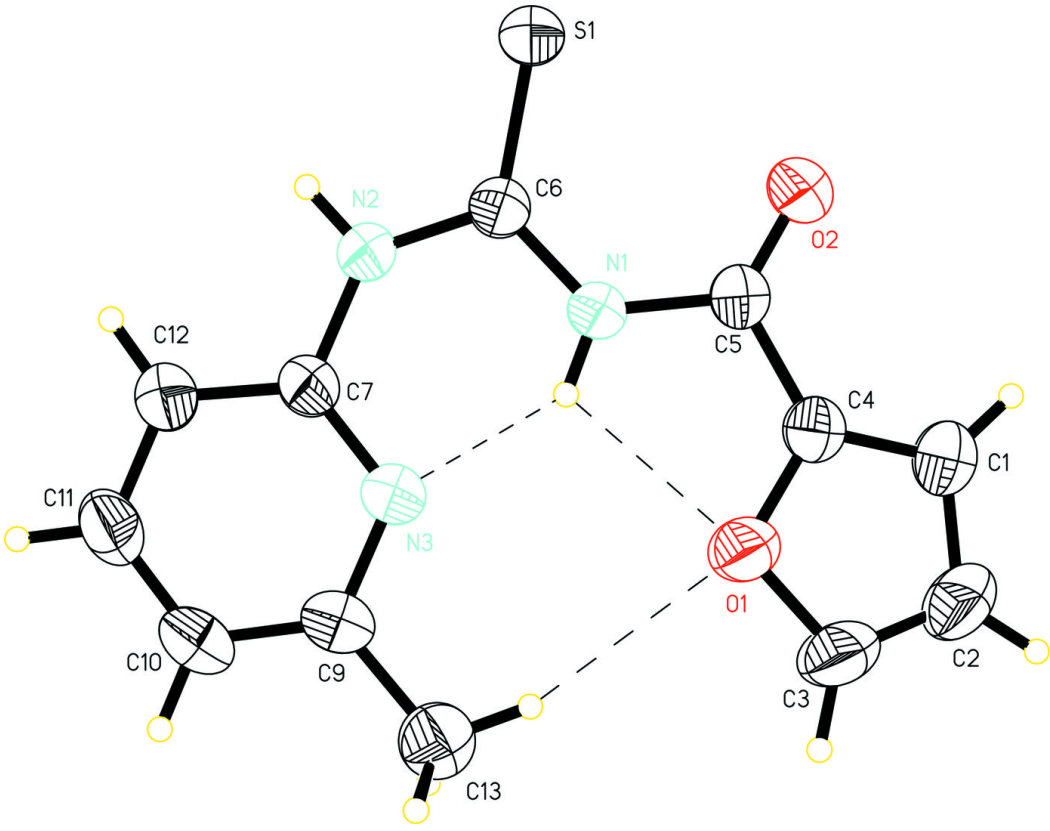

Fig. 2