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1-Benzyl-3-(2-furoyl)thiourea

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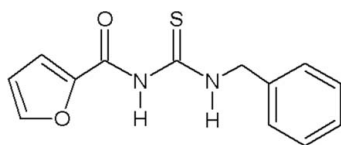
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.036; wR factor = 0.085; data-to-parameter ratio = 13.0.

In the title compound, $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$, the dihedral angle between the two aromatic ring planes is $87.52(12)^\circ$. The molecule shows an intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond. The crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{S}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding.

Related literature

For general background, see: Estévez-Hernández *et al.* (2007); Otazo *et al.* (2001). For related structures, see: Arslan *et al.* (2004); Khawar Rauf *et al.* (2007). For the synthesis, see: Otazo *et al.* (2001).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$
 $M_r = 260.31$
 Tetragonal, $P4_12_12$
 $a = 9.445(3)$ Å
 $c = 27.107(6)$ Å
 $V = 2418.2(12)$ Å³

$Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.26$ mm⁻¹
 $T = 150(2)$ K
 $0.3 \times 0.1 \times 0.08$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: none
 12492 measured reflections
 2120 independent reflections
 1922 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.092$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.085$
 $S = 1.06$
 2120 reflections
 163 parameters
 H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³
 Absolute structure: Flack (1983),
 802 Friedel pairs
 Flack parameter: $-0.16(10)$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}$	0.88	2.00	2.697 (3)	135
$\text{N2}-\text{H2}\cdots\text{S1}^i$	0.88	2.70	3.578 (2)	174
$\text{C7}-\text{H7}\cdots\text{O1}^{ii}$	0.95	2.58	3.423 (3)	148

Symmetry codes: (i) $y, x, -z$; (ii) $y + \frac{1}{2}, -x + \frac{3}{2}, z - \frac{1}{4}$.

Data collection: COLLECT (Nonius, 2000); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO (Otwinowski & Minor, 1997) and SCALEPACK; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2401).

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supplementary materials

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1-Benzyl-3-(2-furoyl)thiourea

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Comment

Substituted *N*-acylthioureas have been a subject of investigations, due to their ability to form stable metal complexes and as ionophores in potentiometric and amperometric sensors for Cd(II), Hg(II) and Pb(II) (Otazo *et al.*, 2001; Estévez-Hernández *et al.*, 2007). The title compound, (I) (Fig. 1), is another example of our newly synthesized furoylthiourea derivatives, which show outstanding complexation properties.

Compound (I) is a typical *N,N*-disubstituted thiourea derivative with normal geometric parameters. The C2—S1 and C3—O1 bonds (Table 1) both show the expected double-bond character. The short values of the C2—N1, C2—N2 and C3—N2 bonds indicate partial double bond character.

The dihedral angle between the aromatic rings is 87.52 (12)°, and the angles with the thiourea plane are 86.67 (19)° for the benzene ring and 4.81 (12)° for the furan ring. An intramolecular N—H···O hydrogen bond is present (Table 2), forming a six-membered ring commonly observed in this type of compounds (Arslan *et al.*, 2004; Khawar Rauf *et al.*, 2007). The crystal structure of (I) is stabilized by intermolecular N—H···S and C—H···O hydrogen bonding (Table 2).

Experimental

The title compound, (I), was synthesized according to a procedure described by Otazo *et al.* (2001) by converting furoyl chloride into furoyl isothiocyanate and then condensing with the appropriate amine. The resulting solid product was crystallized from a dichlorometane-methanol (1:1) mixture yielding X-ray quality single crystals. Elemental analysis for C₁₃H₁₂N₂O₂S found: C 67.73, H 4.75, N 8.23, S 9.34%; calculated: C 67.86, H 4.46, N 8.33, S 9.52%

Refinement

H atoms were placed in calculated positions with C—H = 0.95 Å (aromatic), N—H = 0.88 Å and C—H = 0.99 Å (methylene), and refined in riding model, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

Figures

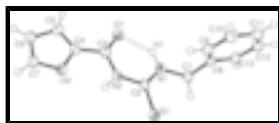


Fig. 1. The molecular structure of title compound. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular N—H···O hydrogen bond is shown as a dashed line.

1-Benzyl-3-(2-furoyl)thiourea

Crystal data

$C_{13}H_{12}N_2O_2S$	$Z = 8$
$M_r = 260.31$	$F_{000} = 1088$
Tetragonal, $P4_12_12$	$D_x = 1.43 \text{ Mg m}^{-3}$
Hall symbol: P 4abw 2nw	Melting point: 402.5 K
$a = 9.445 (3) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.445 (3) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$c = 27.107 (6) \text{ \AA}$	Cell parameters from 9761 reflections
$\alpha = 90^\circ$	$\theta = 2.9\text{--}26.0^\circ$
$\beta = 90^\circ$	$\mu = 0.26 \text{ mm}^{-1}$
$\gamma = 90^\circ$	$T = 150 (2) \text{ K}$
$V = 2418.2 (12) \text{ \AA}^3$	Block, colourless
	$0.3 \times 0.1 \times 0.08 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer	$R_{\text{int}} = 0.092$
ω scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: none	$\theta_{\text{min}} = 3.7^\circ$
12492 measured reflections	$h = -11 \rightarrow 9$
2120 independent reflections	$k = -8 \rightarrow 11$
1922 reflections with $I > 2\sigma(I)$	$l = -32 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.4478P]$
Least-squares matrix: full	where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$wR(F^2) = 0.085$	$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$
$S = 1.06$	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
2120 reflections	Extinction correction: none
163 parameters	Absolute structure: Flack (1983), 802 Friedel pairs
H-atom parameters constrained	Flack parameter: $-0.16 (10)$

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.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7270 (2)	0.4402 (2)	0.16180 (8)	0.0311 (6)
H1A	0.7104	0.3494	0.1791	0.037*
H1B	0.6351	0.4904	0.1597	0.037*
C2	0.7259 (2)	0.4740 (2)	0.07225 (7)	0.0251 (5)
C3	0.8761 (2)	0.3236 (2)	0.01767 (8)	0.0260 (5)
C4	0.9003 (2)	0.2913 (2)	-0.03448 (8)	0.0270 (5)
C5	0.9858 (2)	0.1962 (2)	-0.05694 (8)	0.0316 (5)
H5	1.0486	0.1313	-0.0415	0.038*
C6	0.9623 (2)	0.2131 (2)	-0.10836 (8)	0.0336 (6)
H6	1.0069	0.1616	-0.1341	0.04*
C7	0.8651 (2)	0.3157 (2)	-0.11361 (8)	0.0330 (6)
H7	0.8296	0.3482	-0.1444	0.04*
C8	0.8284 (2)	0.5288 (2)	0.19216 (8)	0.0272 (5)
C9	0.9223 (2)	0.6243 (2)	0.17099 (9)	0.0320 (5)
H9	0.9287	0.6313	0.1361	0.038*
C10	1.0065 (3)	0.7093 (3)	0.20046 (10)	0.0389 (6)
H10	1.0706	0.7739	0.1855	0.047*
C11	0.9987 (3)	0.7014 (3)	0.25126 (10)	0.0388 (6)
H11	1.0563	0.7606	0.2713	0.047*
C12	0.9063 (3)	0.6063 (3)	0.27252 (9)	0.0388 (6)
H12	0.9001	0.6	0.3074	0.047*
C13	0.8222 (2)	0.5198 (3)	0.24330 (8)	0.0333 (6)
H13	0.7598	0.4539	0.2584	0.04*
O1	0.94160 (17)	0.26029 (17)	0.04999 (6)	0.0314 (4)
O2	0.82422 (16)	0.36671 (15)	-0.06877 (5)	0.0309 (4)
N1	0.7763 (2)	0.41033 (19)	0.11201 (6)	0.0275 (4)
H1	0.8432	0.3465	0.1081	0.033*
N2	0.77589 (19)	0.42680 (19)	0.02701 (6)	0.0262 (4)
H2	0.739	0.4679	0.0009	0.031*
S1	0.60657 (6)	0.60581 (6)	0.072955 (19)	0.03259 (18)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0337 (12)	0.0375 (13)	0.0220 (11)	-0.0027 (11)	0.0039 (9)	0.0008 (10)
C2	0.0253 (11)	0.0269 (11)	0.0231 (11)	-0.0052 (9)	0.0015 (9)	-0.0028 (9)
C3	0.0227 (11)	0.0265 (11)	0.0289 (12)	-0.0054 (10)	0.0013 (9)	-0.0005 (9)
C4	0.0289 (12)	0.0274 (12)	0.0248 (11)	-0.0019 (10)	-0.0021 (9)	0.0017 (9)
C5	0.0310 (13)	0.0319 (12)	0.0318 (12)	0.0041 (10)	0.0005 (10)	-0.0019 (10)
C6	0.0396 (14)	0.0353 (13)	0.0260 (12)	0.0033 (11)	0.0041 (10)	-0.0041 (10)
C7	0.0411 (14)	0.0380 (13)	0.0200 (11)	0.0024 (11)	0.0016 (10)	-0.0032 (10)
C8	0.0304 (12)	0.0266 (12)	0.0246 (11)	0.0077 (10)	0.0000 (9)	-0.0018 (9)
C9	0.0354 (13)	0.0324 (12)	0.0283 (12)	0.0043 (11)	0.0009 (10)	0.0006 (11)
C10	0.0381 (14)	0.0358 (13)	0.0427 (15)	-0.0012 (12)	-0.0005 (11)	-0.0003 (11)

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C11	0.0399 (14)	0.0381 (13)	0.0385 (14)	0.0034 (12)	-0.0058 (12)	-0.0074 (12)
C12	0.0451 (15)	0.0452 (15)	0.0261 (12)	0.0097 (13)	-0.0045 (11)	-0.0035 (11)
C13	0.0376 (13)	0.0347 (13)	0.0277 (13)	0.0048 (11)	0.0023 (11)	0.0020 (10)
O1	0.0327 (9)	0.0354 (9)	0.0262 (8)	0.0035 (7)	-0.0005 (7)	0.0009 (7)
O2	0.0354 (9)	0.0319 (9)	0.0253 (8)	0.0060 (7)	-0.0011 (7)	-0.0017 (7)
N1	0.0303 (10)	0.0287 (10)	0.0236 (9)	0.0027 (8)	0.0016 (8)	-0.0010 (8)
N2	0.0288 (10)	0.0291 (10)	0.0208 (9)	0.0010 (8)	-0.0008 (8)	-0.0012 (8)
S1	0.0371 (3)	0.0324 (3)	0.0283 (3)	0.0066 (3)	0.0013 (3)	-0.0021 (2)

Geometric parameters (Å, °)

C1—N1	1.456 (3)	C7—O2	1.363 (2)
C1—C8	1.515 (3)	C7—H7	0.95
C1—H1A	0.99	C8—C9	1.389 (3)
C1—H1B	0.99	C8—C13	1.390 (3)
C2—N1	1.323 (3)	C9—C10	1.384 (3)
C2—N2	1.388 (3)	C9—H9	0.95
C2—S1	1.679 (2)	C10—C11	1.381 (4)
C3—O1	1.228 (3)	C10—H10	0.95
C3—N2	1.382 (3)	C11—C12	1.379 (4)
C3—C4	1.464 (3)	C11—H11	0.95
C4—C5	1.353 (3)	C12—C13	1.387 (4)
C4—O2	1.374 (2)	C12—H12	0.95
C5—C6	1.420 (3)	C13—H13	0.95
C5—H5	0.95	N1—H1	0.88
C6—C7	1.342 (3)	N2—H2	0.88
C6—H6	0.95		
N1—C1—C8	114.11 (18)	C9—C8—C1	122.5 (2)
N1—C1—H1A	108.7	C13—C8—C1	118.8 (2)
C8—C1—H1A	108.7	C10—C9—C8	120.3 (2)
N1—C1—H1B	108.7	C10—C9—H9	119.8
C8—C1—H1B	108.7	C8—C9—H9	119.8
H1A—C1—H1B	107.6	C11—C10—C9	120.9 (2)
N1—C2—N2	116.84 (18)	C11—C10—H10	119.6
N1—C2—S1	124.70 (16)	C9—C10—H10	119.6
N2—C2—S1	118.46 (15)	C12—C11—C10	119.1 (2)
O1—C3—N2	123.90 (19)	C12—C11—H11	120.5
O1—C3—C4	120.59 (19)	C10—C11—H11	120.5
N2—C3—C4	115.51 (18)	C11—C12—C13	120.5 (2)
C5—C4—O2	110.61 (18)	C11—C12—H12	119.8
C5—C4—C3	131.8 (2)	C13—C12—H12	119.8
O2—C4—C3	117.63 (18)	C12—C13—C8	120.6 (2)
C4—C5—C6	105.9 (2)	C12—C13—H13	119.7
C4—C5—H5	127.1	C8—C13—H13	119.7
C6—C5—H5	127.1	C7—O2—C4	105.78 (17)
C7—C6—C5	107.0 (2)	C2—N1—C1	123.51 (18)
C7—C6—H6	126.5	C2—N1—H1	118.2
C5—C6—H6	126.5	C1—N1—H1	118.2
C6—C7—O2	110.8 (2)	C3—N2—C2	128.43 (18)

C6—C7—H7	124.6	C3—N2—H2	115.8
O2—C7—H7	124.6	C2—N2—H2	115.8
C9—C8—C13	118.6 (2)		
O1—C3—C4—C5	1.4 (4)	C10—C11—C12—C13	0.0 (4)
N2—C3—C4—C5	-178.1 (2)	C11—C12—C13—C8	-0.8 (3)
O1—C3—C4—O2	-179.53 (19)	C9—C8—C13—C12	1.1 (3)
N2—C3—C4—O2	1.0 (3)	C1—C8—C13—C12	-175.5 (2)
O2—C4—C5—C6	0.2 (3)	C6—C7—O2—C4	-0.1 (2)
C3—C4—C5—C6	179.3 (2)	C5—C4—O2—C7	-0.1 (2)
C4—C5—C6—C7	-0.2 (3)	C3—C4—O2—C7	-179.4 (2)
C5—C6—C7—O2	0.2 (3)	N2—C2—N1—C1	-175.32 (19)
N1—C1—C8—C9	27.7 (3)	S1—C2—N1—C1	4.4 (3)
N1—C1—C8—C13	-155.8 (2)	C8—C1—N1—C2	-104.7 (2)
C13—C8—C9—C10	-0.6 (3)	O1—C3—N2—C2	-3.1 (3)
C1—C8—C9—C10	175.9 (2)	C4—C3—N2—C2	176.4 (2)
C8—C9—C10—C11	-0.3 (4)	N1—C2—N2—C3	-2.3 (3)
C9—C10—C11—C12	0.6 (4)	S1—C2—N2—C3	178.05 (17)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O1	0.88	2.00	2.697 (3)	135
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Fig. 1

