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

**Hiram Pérez,[a]* Yvonne Mascarenhas,[b] Osvaldo Estévez-Hernández,[c] Sauli Santos Jr[d] and Julio Duque[c]**

[a]Departamento de Química Inorgánica, Facultad de Química, Universidad de la Habana, Habana 10400, Cuba, [b]Instituto de Física de São Carlos, Universidade de São Paulo, São Carlos, Brazil, [c]Instituto de Ciencia y Tecnología de Materiales, Universidad de la Habana, Habana 10400, Cuba, and [d]Laboratório de Física, Universidade Federal do Tocantins, CEP 77020-120, Palmas, Tocantins, Brazil
Correspondence e-mail: hperez@fq.uh.cu

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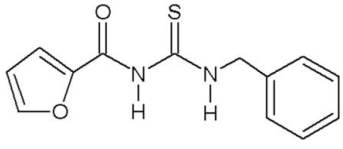
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.036; $wR$ factor = 0.085; data-to-parameter ratio = 13.0.

In the title compound, $C_{13}H_{12}N_2O_2S$, the dihedral angle between the two aromatic ring planes is 87.52 (12)°. The molecule shows an intramolecular N—H···O hydrogen bond. The crystal structure is stabilized by intermolecular N—H···S and C—H···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

$C_{13}H_{12}N_2O_2S$
$M_r$ = 260.31
Tetragonal, $P4_12_12$
$a$ = 9.445 (3) Å
$c$ = 27.107 (6) Å
$V$ = 2418.2 (12) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.26 mm$^{-1}$
$T$ = 150 (2) K
0.3 × 0.1 × 0.08 mm

### Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
12492 measured reflections
2120 independent reflections
1922 reflections with $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −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···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1 | 0.88 | 2.00 | 2.697 (3) | 135 |
| N2—H2···S1$^{i}$ | 0.88 | 2.70 | 3.578 (2) | 174 |
| C7—H7···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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# supporting information

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

**Hiram Pérez, Yvonne Mascarenhas, Osvaldo Estévez-Hernández, Sauli Santos Jr and Julio Duque**

### S1. Comment

Substituted *N*-acylthioureas have been a subject of investigations, due to their ability to form stable metal complexes and as ionophores in potenciometric 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).

### S2. 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 $C_{13}H_{12}N_2O_2S$ 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%

### S3. 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, $U_{iso}$(H) = 1.2$U_{eq}$(C,*N*).

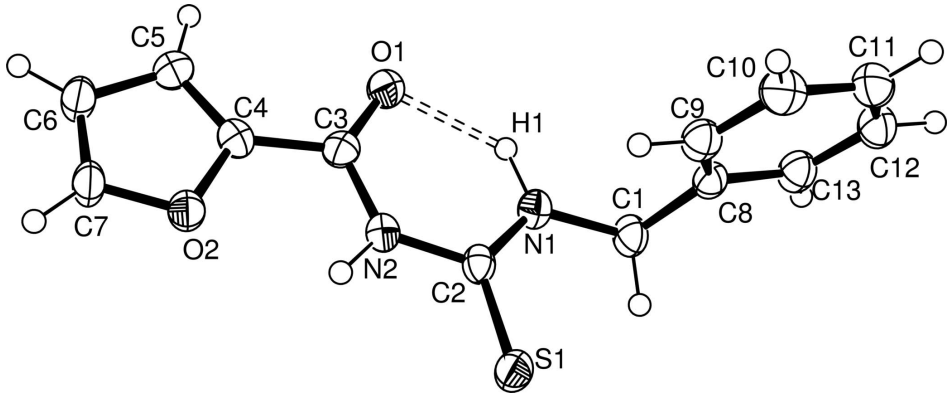


**Figure 1**

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*

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

*Data collection*

Nonius KappaCCD diffractometer
$\omega$ scans
12492 measured reflections
2120 independent reflections
1922 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.7°$
$h = -11\rightarrow9$
$k = -8\rightarrow11$
$l = -32\rightarrow24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.085$
$S = 1.06$
2120 reflections
163 parameters
0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.4478P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å$^{-3}$
$\Delta\rho_{min} = -0.19$ e Å$^{-3}$
Absolute structure: Flack (1983), 802 Friedel pairs
Absolute structure 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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) |
| 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, º)*

| | | | |
|---|---|---|---|
| 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 (Å, º)*

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

Symmetry codes: (i) *y*, *x*, −*z*; (ii) *y*+1/2, −*x*+3/2, *z*−1/4.