Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Jahyr E. Theodoro,[a] Yvonne Mascarenhas,[b] Javier Ellena,[a] Osvaldo Estévez-Hernández[c] and Julio Duque[c]*

[a]Grupo de Cristalografía, Instituto de Física de São Carlos, Universidade de São Paulo, São Carlos, Brazil, [b]Instituto de Física de São Carlos, Universidade de São Paulo, São Carlos, Brazil, and [c]Department of Structure Analysis, Institute of Materials, University of Havana, Cuba
Correspondence e-mail: duque@imre.oc.uh.cu

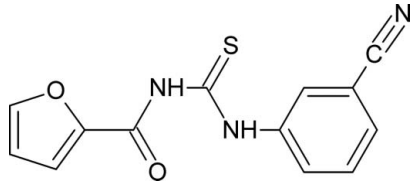
Received 16 May 2008; accepted 27 May 2008

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; R factor = 0.071; wR factor = 0.209; data-to-parameter ratio = 15.6.

The title compound, $C_{13}H_9N_3O_2S$, was synthesized from furoyl isothiocyanate and 3-aminobenzonitrile in dry acetone. The thiourea group is in the thioamide form. The thiourea fragment makes dihedral angles of 3.91 (16) and 37.83 (12)° with the ketofuran group and the benzene ring, respectively. The molecular geometry is stabilized by N—H···O hydrogen bonds. In the crystal structure, centrosymmetrically related molecules are linked by two intermolecular N—H···S hydrogen bonds to form dimers.

Related literature

For general background, see: Aly *et al.* (2007); Koch (2001). For related structures, see: Dago *et al.* (1987); Otazo-Sánchez *et al.* (2001); Pérez *et al.* (2008); Duque *et al.* (2008). For the synthesis, see: Otazo-Sánchez *et al.* (2001).



Experimental

Crystal data

$C_{13}H_9N_3O_2S$
M_r = 271.29
Monoclinic, $P2_1/n$
a = 16.7375 (5) Å
b = 3.8789 (1) Å
c = 19.6739 (5) Å
β = 96.956 (1)°
V = 1267.89 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 K
0.16 × 0.04 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
4807 measured reflections
2684 independent reflections
1908 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.208
S = 1.08
2684 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 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···O2	0.86	2.28	2.701 (5)	110
N1—H1···S1^{i}	0.86	2.80	3.629 (4)	163
N2—H2···O1	0.86	1.90	2.622 (4)	141

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

Data collection: *COLLECT* (Enraf–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).

The authors thank the Crystallography Group, São Carlos Physics Institute, USP, and acknowledge financial support from the Brazilian agency CNPq.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2219).

References

Aly, A. A., Ahmed, E. K., El-Mokadem, K. M. & Hegazy, M. E. F. (2007). *J. Sulfur Chem.* **28**, 73–93.
Dago, A., Simonov, M. A., Pobedimskaya, E. A., Martín, A. & Macías, A. (1987). *Kristallografiya*, **32**, 1024–1026.
Duque, J., Estevez-Hernandez, O., Reguera, E., Corrêa, R. S. & Gutierrez Maria, P. (2008). *Acta Cryst.* E**64**, o1068.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Koch, K. R. (2001). *Coord. Chem. Rev.* **216–217**, 473–488.
Otazo-Sánchez, E., Pérez-Marín, L., Estévez-Hernández, O., Rojas-Lima, S. & Alonso-Chamorro, J. (2001). *J. Chem. Soc. Perkin Trans. 2*, pp. 2211–2218.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Pérez, H., Mascarenhas, Y., Estévez-Hernández, O., Santos, S. Jr & Duque, J. (2008). *Acta Cryst.* E**64**, o695.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, o1193 [doi:10.1107/S1600536808016012]

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

Jahyr E. Theodoro, Yvonne Mascarenhas, Javier Ellena, Osvaldo Estévez-Hernández and Julio Duque

S1. Comment

The importance of aroylthioureas is found largely in heterocyclic syntheses and many of these substrates have interesting biological activities. Aroylthioureas have also been found to have applications in metal complexes and molecular electronics (Aly *et al.*, 2007). Structural determinations of this kind of derivatives shed more light on the chemistry of aroylthiourea compounds and their wide variety of applications. The title compound (Fig. 1) is another example of our newly synthesized furoylthiourea derivatives.

The title compound crystallizes in the thioamide form. The bond lengths are within the ranges observed for similar compounds (Koch, 2001). The C2—S1 and C1—O1 bonds (Table 1) both show the expected double-bond character. The short values of the C2—N1 (1.347 (5) Å), C2—N2 (1.348 (5) Å) and C1—N1 (1.368 (5) Å) bonds indicate partial double bond character. These results can be explained by the existence of resonance in this part of the molecule. The furan carbonyl group is nearly coplanar with the plane of the thiourea fragment (dihedral angle 3.39(16°), whereas the benzene ring is inclined by 37.83 (12)°. The geometry in the thiourea group is stabilized by the N2—H2···O1 and N1—H1···O2 intramolecular hydrogen bonds (Fig. 1 and Table 2). The crystal structure is stabilized by two intermolecular N1—H1···S1 hydrogen bonds (Fig. 2 and Table 2) between centrosymmetrically related molecules forming dimers stacked along the [010] direction.

S2. Experimental

The title compound was synthesized according to a previous report (Otazo-Sánchez *et al.*, 2001), by converting furoyl chloride into furoyl isothiocyanate and then condensing with 3-aminobenzonitrile. The resulting solid product was crystallized from ethanol yielding X-ray quality single crystals (m.p 148–149 °C). Elemental analysis (%) for $C_{13}H_9N_3O_2S$ calculated: C 57.56, H 3.32, N 15.50, S 11.81; found: C 57.77, H 3.34, N 15.79, S 11.73.

S3. Refinement

H atoms were placed in calculated positions with N—H = 0.88 Å and C—H = 0.95 Å or 0.98 Å (methylene), and refined in riding-model, with U_{iso}(H) = 1.5U_{eq}(C) for methylene or 1.2U_{eq}(C,N) for others.

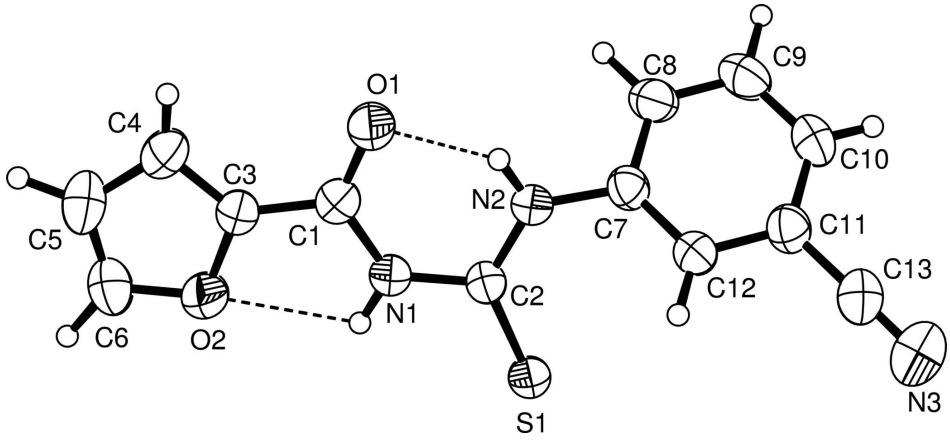


Figure 1

View of the molecule (50% probability displacement ellipsoids) Intramolecular hydrogen bonds are shown as dashed lines.

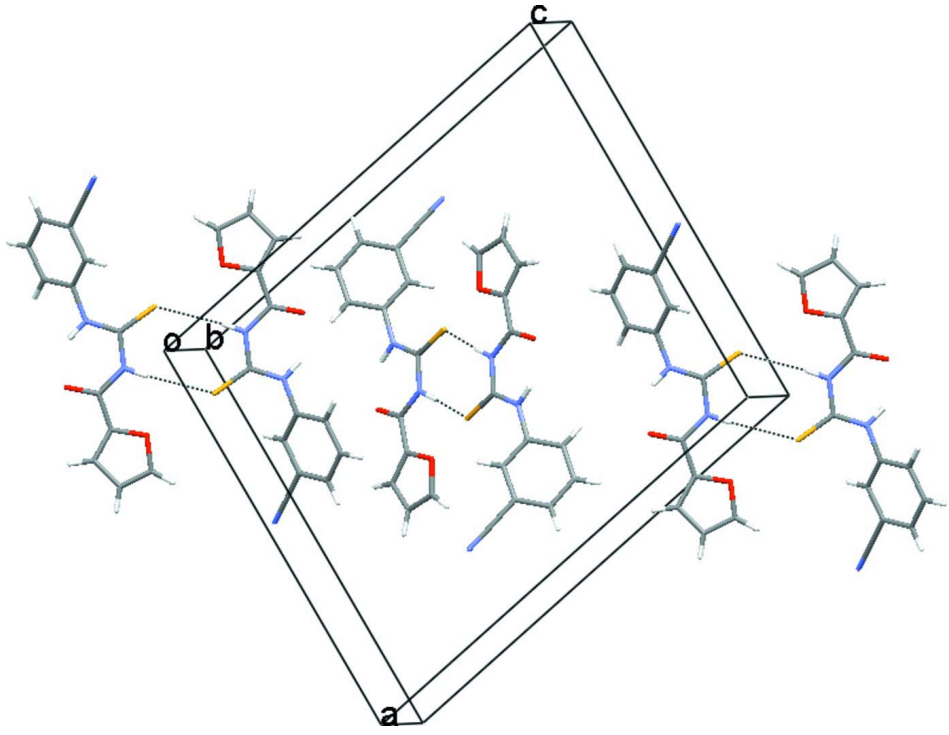


Figure 2

View of the crystal packing of the title compound. Intermolecular hydrogen bonds are shown as dashed lines.

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

Crystal data

$C_{13}H_9N_3O_2S$
$M_r = 271.29$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 16.7375\ (5)$ Å
$b = 3.8789\ (1)$ Å
$c = 19.6739\ (5)$ Å
$\beta = 96.956\ (1)°$
$V = 1267.89\ (6)$ Å^3
$Z = 4$

$F(000) = 560$
$D_x = 1.421$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3051 reflections
$\theta = 2.9$–$26.7°$

$\mu = 0.26$ mm^{-1}
$T = 294$ K
Prism, colourless
0.16 × 0.04 × 0.03 mm

Data collection

Enraf–Nonius KappaCCD diffractometer
CCD rotation images, thick slices scans
4807 measured reflections
2684 independent reflections
1908 reflections with $I > 2\sigma(I)$

$R_{int} = 0.040$
$\theta_{max} = 26.9°$, $\theta_{min} = 3.9°$
$h = -20 \rightarrow 21$
$k = -4 \rightarrow 4$
$l = -25 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.208$
$S = 1.08$
2684 reflections
172 parameters

0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 3.4802P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

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}
S1	0.10993 (6)	0.7197 (3)	−0.00840 (5)	0.0465 (3)
O1	0.08926 (19)	0.9883 (11)	0.21366 (15)	0.0699 (11)
C13	0.3735 (3)	1.3099 (15)	−0.0369 (3)	0.0617 (13)
N3	0.3872 (3)	1.4227 (17)	−0.0884 (3)	0.0909 (17)
N1	0.0452 (2)	0.7949 (10)	0.10538 (16)	0.0457 (9)
H1	0.0031	0.7186	0.0805	0.055*
O2	−0.10121 (19)	0.6665 (10)	0.14431 (15)	0.0643 (10)
N2	0.17909 (19)	0.9396 (11)	0.11374 (17)	0.0482 (9)
H2	0.1712	0.991	0.1549	0.058*
C7	0.2586 (2)	0.9846 (11)	0.0976 (2)	0.0415 (9)
C5	−0.1517 (3)	0.7102 (15)	0.2424 (3)	0.0676 (15)
H5	−0.1872	0.6986	0.2752	0.081*
C2	0.1143 (2)	0.8261 (11)	0.07249 (19)	0.0398 (9)
C9	0.3556 (2)	1.1694 (12)	0.0263 (2)	0.0474 (10)
C8	0.2756 (2)	1.1209 (12)	0.0366 (2)	0.0456 (10)
H8	0.2342	1.1798	0.0026	0.055*
C3	−0.0430 (2)	0.7960 (12)	0.1925 (2)	0.0471 (10)
C10	0.4180 (3)	1.0819 (14)	0.0767 (2)	0.0571 (12)
H10	0.4714	1.1093	0.069	0.068*
C1	0.0355 (3)	0.8694 (13)	0.1719 (2)	0.0495 (11)

C11	0.3993 (3)	0.9541 (15)	0.1381 (3)	0.0633 (14)
H11	0.4403	0.9026	0.1729	0.076*
C4	−0.0724 (3)	0.8274 (14)	0.2531 (2)	0.0581 (13)
H4	−0.0452	0.9105	0.2938	0.07*
C6	−0.1676 (3)	0.6183 (17)	0.1772 (3)	0.0705 (15)
H6	−0.2168	0.5335	0.1569	0.085*
C12	0.3204 (3)	0.9022 (13)	0.1483 (2)	0.0535 (11)
H12	0.3084	0.8109	0.1895	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0410 (6)	0.0603 (8)	0.0382 (5)	−0.0046 (5)	0.0050 (4)	−0.0056 (5)
O1	0.0528 (19)	0.112 (3)	0.0457 (17)	−0.021 (2)	0.0093 (14)	−0.0222 (19)
C13	0.049 (3)	0.074 (4)	0.063 (3)	−0.004 (2)	0.015 (2)	0.004 (3)
N3	0.088 (4)	0.115 (5)	0.073 (3)	−0.013 (3)	0.025 (3)	0.017 (3)
N1	0.0389 (17)	0.061 (2)	0.0367 (17)	−0.0055 (17)	0.0040 (14)	−0.0029 (17)
O2	0.0536 (18)	0.096 (3)	0.0440 (16)	−0.0177 (19)	0.0072 (14)	−0.0081 (18)
N2	0.0393 (18)	0.069 (3)	0.0355 (17)	−0.0083 (18)	0.0028 (13)	−0.0001 (17)
C7	0.039 (2)	0.041 (2)	0.045 (2)	−0.0045 (18)	0.0045 (16)	0.0001 (18)
C5	0.058 (3)	0.075 (4)	0.075 (3)	0.001 (3)	0.031 (3)	−0.004 (3)
C2	0.0358 (19)	0.043 (2)	0.040 (2)	−0.0019 (17)	0.0049 (16)	0.0019 (18)
C9	0.044 (2)	0.046 (3)	0.053 (2)	−0.0032 (19)	0.0071 (18)	0.003 (2)
C8	0.039 (2)	0.052 (3)	0.044 (2)	−0.0024 (19)	0.0022 (17)	0.0050 (19)
C3	0.042 (2)	0.058 (3)	0.042 (2)	−0.004 (2)	0.0073 (17)	−0.003 (2)
C10	0.036 (2)	0.071 (3)	0.064 (3)	−0.005 (2)	0.005 (2)	0.002 (3)
C1	0.047 (2)	0.060 (3)	0.042 (2)	−0.005 (2)	0.0072 (18)	−0.001 (2)
C11	0.043 (2)	0.081 (4)	0.062 (3)	−0.001 (2)	−0.011 (2)	0.010 (3)
C4	0.059 (3)	0.073 (4)	0.045 (2)	−0.003 (3)	0.016 (2)	−0.009 (2)
C6	0.045 (3)	0.094 (4)	0.073 (3)	−0.014 (3)	0.013 (2)	0.009 (3)
C12	0.051 (2)	0.060 (3)	0.047 (2)	−0.008 (2)	−0.0041 (19)	0.006 (2)

Geometric parameters (Å, º)

S1—C2	1.637 (4)	C5—C4	1.395 (7)
O1—C1	1.233 (5)	C5—H5	0.93
C13—N3	1.153 (6)	C9—C8	1.392 (6)
C13—C9	1.422 (6)	C9—C10	1.392 (6)
N1—C1	1.368 (5)	C8—H8	0.93
N1—C2	1.397 (5)	C3—C4	1.348 (6)
N1—H1	0.86	C3—C1	1.450 (6)
O2—C6	1.365 (6)	C10—C11	1.377 (7)
O2—C3	1.369 (5)	C10—H10	0.93
N2—C2	1.348 (5)	C11—C12	1.375 (6)
N2—C7	1.416 (5)	C11—H11	0.93
N2—H2	0.86	C4—H4	0.93
C7—C8	1.373 (6)	C6—H6	0.93
C7—C12	1.383 (6)	C12—H12	0.93

C5—C6	1.326 (7)		
N3—C13—C9	179.3 (6)	C9—C8—H8	120.5
C1—N1—C2	128.7 (3)	C4—C3—O2	109.9 (4)
C1—N1—H1	115.6	C4—C3—C1	132.0 (4)
C2—N1—H1	115.6	O2—C3—C1	118.1 (4)
C6—O2—C3	105.9 (4)	C11—C10—C9	118.9 (4)
C2—N2—C7	128.0 (3)	C11—C10—H10	120.6
C2—N2—H2	116	C9—C10—H10	120.6
C7—N2—H2	116	O1—C1—N1	123.7 (4)
C8—C7—C12	120.2 (4)	O1—C1—C3	120.0 (4)
C8—C7—N2	122.9 (4)	N1—C1—C3	116.3 (4)
C12—C7—N2	116.8 (4)	C12—C11—C10	120.3 (4)
C6—C5—C4	108.0 (4)	C12—C11—H11	119.9
C6—C5—H5	126	C10—C11—H11	119.9
C4—C5—H5	126	C3—C4—C5	106.3 (4)
N2—C2—N1	113.6 (3)	C3—C4—H4	126.9
N2—C2—S1	127.3 (3)	C5—C4—H4	126.9
N1—C2—S1	119.1 (3)	C5—C6—O2	110.0 (4)
C8—C9—C10	121.1 (4)	C5—C6—H6	125
C8—C9—C13	119.1 (4)	O2—C6—H6	125
C10—C9—C13	119.8 (4)	C11—C12—C7	120.6 (4)
C7—C8—C9	118.9 (4)	C11—C12—H12	119.7
C7—C8—H8	120.5	C7—C12—H12	119.7
C2—N2—C7—C8	41.1 (7)	C2—N1—C1—C3	−177.3 (4)
C2—N2—C7—C12	−142.4 (5)	C4—C3—C1—O1	−1.2 (9)
C7—N2—C2—N1	176.9 (4)	O2—C3—C1—O1	179.5 (5)
C7—N2—C2—S1	−1.9 (7)	C4—C3—C1—N1	177.9 (5)
C1—N1—C2—N2	1.3 (7)	O2—C3—C1—N1	−1.3 (7)
C1—N1—C2—S1	−179.8 (4)	C9—C10—C11—C12	2.4 (8)
C12—C7—C8—C9	0.9 (7)	O2—C3—C4—C5	0.5 (6)
N2—C7—C8—C9	177.3 (4)	C1—C3—C4—C5	−178.8 (5)
C10—C9—C8—C7	0.1 (7)	C6—C5—C4—C3	−0.7 (7)
C13—C9—C8—C7	179.9 (5)	C4—C5—C6—O2	0.7 (7)
C6—O2—C3—C4	−0.1 (6)	C3—O2—C6—C5	−0.4 (6)
C6—O2—C3—C1	179.3 (5)	C10—C11—C12—C7	−1.5 (8)
C8—C9—C10—C11	−1.7 (8)	C8—C7—C12—C11	−0.3 (7)
C13—C9—C10—C11	178.4 (5)	N2—C7—C12—C11	−176.8 (5)
C2—N1—C1—O1	1.8 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	2.28	2.701 (5)	110

N1—H1···S1^{i}	0.86	2.80	3.629 (4)	163
N2—H2···O1	0.86	1.90	2.622 (4)	141

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