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N-(2-Furylcarbonyl)piperidine-1-carbothioamide

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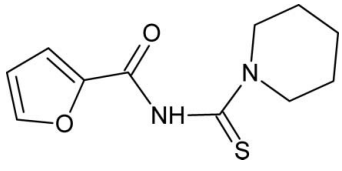
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.068; wR factor = 0.205; data-to-parameter ratio = 16.5.

The title compound, $C_{11}H_{14}N_2O_2S$, was synthesized from furoyl isothiocyanate and piperidine in dry acetone. The thiourea group is in the thioamide form. The thiourea group makes a dihedral angle of 53.9 (1)° with the furan carbonyl group. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds, forming one-dimensional chains along the *c* axis. An intramolecular N—H···O hydrogen bond is also present.

Related literature

For general background, see: Aly *et al.* (2007); Estévez-Hernández *et al.* (2006, 2007); Koch (2001). For related structures, see: Dago *et al.* (1987); Plutin *et al.* (2000); Pérez *et al.* (2008); Duque *et al.* (2008). For the synthesis, see: Otazo-Sánchez *et al.* (2001).



Experimental

Crystal data

$C_{11}H_{14}N_2O_2S$
M_r = 238.3
Orthorhombic, *Pbca*
a = 31.6377 (15) Å
b = 8.6787 (4) Å
c = 8.5308 (3) Å
V = 2342.34 (18) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 K
0.15 × 0.13 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
4308 measured reflections
2387 independent reflections
1550 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.205
S = 1.10
2387 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 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	2.38	2.756 (3)	107
N1—H1···O1^{i}	0.86	2.18	2.994 (4)	157

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WW2121).

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supplementary materials

Acta Cryst. (2008). E**64**, o1457 [**doi:10.1107/S1600536808020977**]

N-(2-Furylcarbonyl)piperidine-1-carbothioamide

J. Duque, O. Estévez-Hernández, Y. Mascarenhas, J. Ellena and R. S. Corrêa

Comment

Thiourea and its derivatives form a versatile family of ligands that are suitable to form complexes with ions of transition and post-transition metal through the S atom (Koch *et al.*, 2001; Aly *et al.*, 2007). The title compound shows outstanding complexation properties (Estévez-Hernández *et al.*, 2006). The potential applications of this class of ligands as ionophores or chemical modifiers in amperometric sensors (Estévez-Hernández *et al.*, 2007) have stimulated our interest in their crystal structure. The title compound crystallizes in the thioamide form. The main bond lengths and torsion angles are within the ranges obtained for similar compounds (Dago *et al.*, 1987; Plutin *et al.*, 2000). All the C–N bonds of thiourea fragment C1–N1, C2–N1 and C2–N2 (Table1) are in the range 1.415 (4)–1.327 (4) Å, intermediate between those expected for single and double C–N bonds (1.47 and 1.27 Å respectively). These results can be explained by the existence of resonance in this part of molecule (Pérez *et al.*, 2008; Duque *et al.*, 2008). The central thiourea fragment (N1—C2—S1—N2) makes dihedral angle of 53.9 (1)° with the furan carbonyl (C1—C3—C4—C5—C6—O2) group. The *trans-cis* geometry in the thiourea moiety is stabilized by the N1–H1···O2 intramolecular hydrogen bond (Fig.1 and Table 2). In the crystal structure symmetry related molecules are linked by N1–H1···O1 intermolecular hydrogen bonds to form one-dimensional chains along *c* axis (Figs. 2 and Table 2).

Experimental

The title compound was synthesized according to a previous report (Otazo-Sánchez *et al.*, 2001), by converting furoyl choride into furoyl isothiocyanate and then condensing with piperidine. The resulting solid product was crystallized from ethanol yielding X-ray quality single crystals (m.p 120–121°C). Elemental analysis (%) for $C_{11}H_{14}N_2O_2S$ calculated: C 55.46, H 5.88, N 11.76, S 13.45; found: C 55.23, H 5.90, N 11.63, S 13.32.

Refinement

All H atoms were refined with $U_{iso}(H)=1.2U_{eq}(C/N)$.

Figures

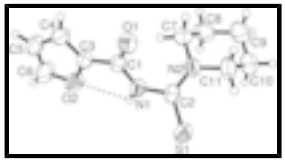

Fig. 1. View of the molecular structure of the title compound (50% probability displacement ellipsoids). Intramolecular Hydrogen bonds (N1–H1···O2) are shown as dashed lines.

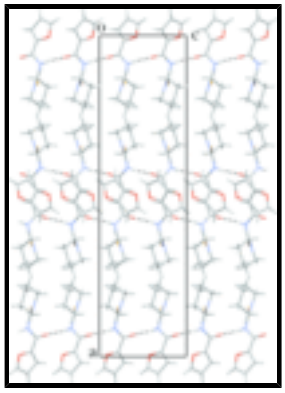

Fig. 2. View of the crystal packing of the title compound projected down the *b* axis. Intermolecular hydrogen bonds (N1–H1···O1) form one-dimensional chains along *c* axis. The hydrogen bonds are shown as dotted lines.

N-(2-Furylcarbonyl)piperidine-1-carbothioamide

Crystal data

$C_{11}H_{14}N_2O_2S_1$	F_{000} = 1008
M_r = 238.3	D_x = 1.352 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 2684 reflections
a = 31.6377 (15) Å	θ = 2.9–26.4º
b = 8.6787 (4) Å	μ = 0.26 mm^{-1}
c = 8.5308 (3) Å	*T* = 294 K
V = 2342.34 (18) Å^3	Prism, colourless
Z = 8	0.15 × 0.13 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.039
CCD rotation images, thick slices scans	θ_{max} = 26.4º
Absorption correction: none	θ_{min} = 3.4º
4308 measured reflections	*h* = −39→39
2387 independent reflections	*k* = −10→10
1550 reflections with $I > 2\sigma(I)$	*l* = −10→10

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.1017P)^2 + 1.0533P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.067	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.205	$\Delta\rho_{max}$ = 0.35 e Å^{-3}
S = 1.11	$\Delta\rho_{min}$ = −0.35 e Å^{-3}
2387 reflections	Extinction correction: none
145 parameters	

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.05964 (9)	0.3587 (4)	0.2251 (4)	0.0455 (7)
C2	0.13205 (9)	0.2817 (4)	0.2882 (3)	0.0456 (8)
C3	0.01610 (9)	0.3667 (3)	0.2812 (3)	0.0442 (7)
C4	−0.01794 (10)	0.4368 (4)	0.2179 (4)	0.0561 (9)
H4	−0.0191	0.4904	0.1237	0.067*
C5	−0.05161 (11)	0.4118 (4)	0.3246 (5)	0.0678 (11)
H5	−0.0792	0.4468	0.314	0.081*
C6	−0.03631 (12)	0.3293 (5)	0.4421 (5)	0.0776 (12)
H6	−0.0521	0.2957	0.5275	0.093*
C7	0.13358 (12)	0.5686 (4)	0.2859 (5)	0.0627 (10)
H7A	0.1051	0.5591	0.3268	0.075*
H7B	0.1321	0.6226	0.1865	0.075*
C8	0.16045 (13)	0.6593 (4)	0.3996 (5)	0.0743 (11)
H8A	0.1586	0.6123	0.5026	0.089*
H8B	0.1495	0.7633	0.4075	0.089*
C9	0.20640 (14)	0.6656 (5)	0.3497 (6)	0.0890 (13)
H9A	0.209	0.7254	0.2541	0.107*
H9B	0.223	0.7157	0.4305	0.107*
C10	0.22286 (12)	0.5045 (5)	0.3226 (6)	0.0849 (13)
H10A	0.2515	0.5098	0.2827	0.102*
H10B	0.2235	0.4492	0.4213	0.102*
C11	0.19565 (11)	0.4186 (5)	0.2081 (5)	0.0703 (11)
H11A	0.197	0.4688	0.1065	0.084*
H11B	0.2062	0.3143	0.1962	0.084*
N1	0.08854 (7)	0.2942 (3)	0.3243 (3)	0.0458 (7)
H1	0.0799	0.2594	0.4131	0.055*
N2	0.15184 (8)	0.4140 (3)	0.2619 (3)	0.0527 (7)
O1	0.06909 (7)	0.4111 (3)	0.0970 (2)	0.0569 (7)
O2	0.00546 (7)	0.3001 (3)	0.4214 (3)	0.0651 (7)
S1	0.15400 (3)	0.10814 (10)	0.28587 (13)	0.0661 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0424 (16)	0.0478 (17)	0.0462 (19)	0.0006 (13)	−0.0034 (13)	−0.0070 (15)
C2	0.0397 (15)	0.056 (2)	0.0412 (17)	−0.0007 (14)	−0.0028 (13)	−0.0054 (15)
C3	0.0435 (16)	0.0484 (17)	0.0406 (16)	0.0015 (13)	−0.0001 (13)	0.0006 (14)

C4	0.0547 (19)	0.0541 (19)	0.060 (2)	0.0076 (16)	−0.0086 (16)	0.0046 (17)
C5	0.0423 (18)	0.070 (2)	0.091 (3)	0.0139 (17)	0.0037 (18)	−0.004 (2)
C6	0.050 (2)	0.092 (3)	0.090 (3)	0.018 (2)	0.023 (2)	0.017 (2)
C7	0.057 (2)	0.049 (2)	0.082 (3)	0.0016 (16)	0.0006 (18)	0.0000 (18)
C8	0.089 (3)	0.048 (2)	0.086 (3)	−0.0092 (19)	−0.006 (2)	−0.005 (2)
C9	0.082 (3)	0.074 (3)	0.111 (3)	−0.027 (2)	−0.018 (3)	0.002 (3)
C10	0.053 (2)	0.084 (3)	0.118 (4)	−0.017 (2)	−0.011 (2)	0.009 (3)
C11	0.0438 (19)	0.079 (3)	0.088 (3)	−0.0093 (18)	0.0112 (18)	−0.006 (2)
N1	0.0374 (13)	0.0569 (17)	0.0430 (14)	0.0020 (11)	0.0016 (10)	0.0006 (12)
N2	0.0419 (15)	0.0509 (16)	0.0655 (18)	−0.0043 (12)	0.0023 (12)	−0.0071 (13)
O1	0.0537 (13)	0.0760 (17)	0.0411 (13)	−0.0015 (11)	0.0017 (10)	0.0051 (11)
O2	0.0520 (13)	0.0813 (18)	0.0619 (15)	0.0160 (12)	0.0121 (11)	0.0164 (13)
S1	0.0485 (5)	0.0536 (6)	0.0962 (8)	0.0069 (4)	−0.0001 (4)	−0.0080 (5)

Geometric parameters (Å, °)

C1—O1	1.221 (4)	C7—H7A	0.97
C1—N1	1.366 (4)	C7—H7B	0.97
C1—C3	1.460 (4)	C8—C9	1.516 (6)
C2—N2	1.327 (4)	C8—H8A	0.97
C2—N1	1.415 (4)	C8—H8B	0.97
C2—S1	1.659 (3)	C9—C10	1.510 (7)
C3—C4	1.349 (4)	C9—H9A	0.97
C3—O2	1.371 (4)	C9—H9B	0.97
C4—C5	1.418 (5)	C10—C11	1.500 (5)
C4—H4	0.93	C10—H10A	0.97
C5—C6	1.323 (5)	C10—H10B	0.97
C5—H5	0.93	C11—N2	1.461 (4)
C6—O2	1.357 (4)	C11—H11A	0.97
C6—H6	0.93	C11—H11B	0.97
C7—N2	1.475 (4)	N1—H1	0.86
C7—C8	1.511 (5)		
O1—C1—N1	122.9 (3)	C9—C8—H8B	109.2
O1—C1—C3	120.4 (3)	H8A—C8—H8B	107.9
N1—C1—C3	116.6 (3)	C10—C9—C8	109.9 (3)
N2—C2—N1	115.5 (3)	C10—C9—H9A	109.7
N2—C2—S1	125.9 (2)	C8—C9—H9A	109.7
N1—C2—S1	118.6 (2)	C10—C9—H9B	109.7
C4—C3—O2	110.1 (3)	C8—C9—H9B	109.7
C4—C3—C1	130.1 (3)	H9A—C9—H9B	108.2
O2—C3—C1	119.8 (3)	C11—C10—C9	111.2 (3)
C3—C4—C5	105.9 (3)	C11—C10—H10A	109.4
C3—C4—H4	127	C9—C10—H10A	109.4
C5—C4—H4	127	C11—C10—H10B	109.4
C6—C5—C4	107.1 (3)	C9—C10—H10B	109.4
C6—C5—H5	126.5	H10A—C10—H10B	108
C4—C5—H5	126.5	N2—C11—C10	110.7 (3)
C5—C6—O2	111.0 (3)	N2—C11—H11A	109.5
C5—C6—H6	124.5	C10—C11—H11A	109.5

O2—C6—H6	124.5	N2—C11—H11B	109.5
N2—C7—C8	110.0 (3)	C10—C11—H11B	109.5
N2—C7—H7A	109.7	H11A—C11—H11B	108.1
C8—C7—H7A	109.7	C1—N1—C2	123.2 (3)
N2—C7—H7B	109.7	C1—N1—H1	118.4
C8—C7—H7B	109.7	C2—N1—H1	118.4
H7A—C7—H7B	108.2	C2—N2—C11	121.6 (3)
C7—C8—C9	112.2 (4)	C2—N2—C7	125.4 (3)
C7—C8—H8A	109.2	C11—N2—C7	113.0 (3)
C9—C8—H8A	109.2	C6—O2—C3	105.9 (3)
C7—C8—H8B	109.2		
O1—C1—C3—C4	−5.9 (5)	N2—C2—N1—C1	59.9 (4)
N1—C1—C3—C4	172.5 (3)	S1—C2—N1—C1	−121.4 (3)
O1—C1—C3—O2	176.5 (3)	N1—C2—N2—C11	−173.8 (3)
N1—C1—C3—O2	−5.2 (4)	S1—C2—N2—C11	7.6 (5)
O2—C3—C4—C5	−0.2 (4)	N1—C2—N2—C7	7.8 (4)
C1—C3—C4—C5	−178.0 (3)	S1—C2—N2—C7	−170.8 (3)
C3—C4—C5—C6	−0.5 (5)	C10—C11—N2—C2	−120.6 (4)
C4—C5—C6—O2	1.0 (5)	C10—C11—N2—C7	58.1 (4)
N2—C7—C8—C9	54.0 (5)	C8—C7—N2—C2	122.3 (4)
C7—C8—C9—C10	−53.7 (5)	C8—C7—N2—C11	−56.3 (4)
C8—C9—C10—C11	54.5 (5)	C5—C6—O2—C3	−1.1 (5)
C9—C10—C11—N2	−56.8 (5)	C4—C3—O2—C6	0.8 (4)
O1—C1—N1—C2	−0.1 (5)	C1—C3—O2—C6	178.8 (3)
C3—C1—N1—C2	−178.4 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	2.38	2.756 (3)	107
N1—H1···O1^{i}	0.86	2.18	2.994 (4)	157

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

Fig. 1

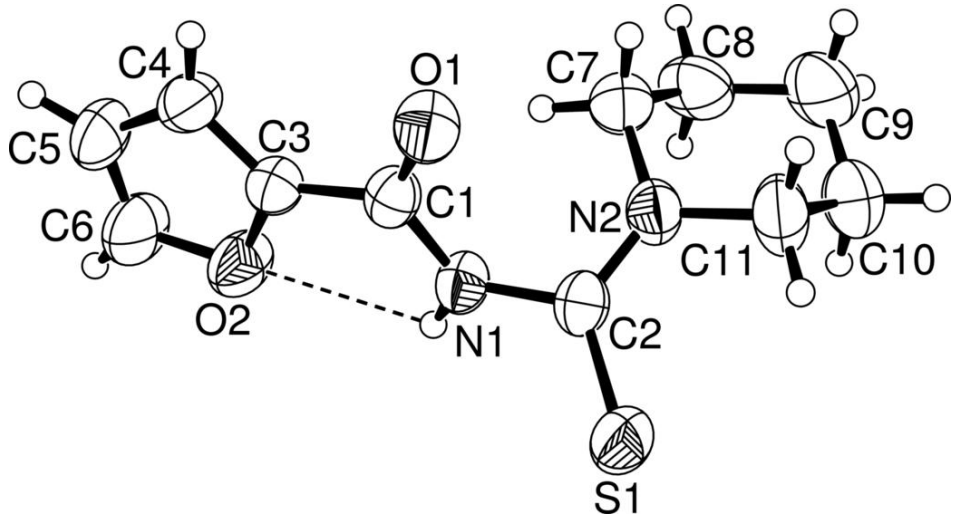

Fig. 2