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

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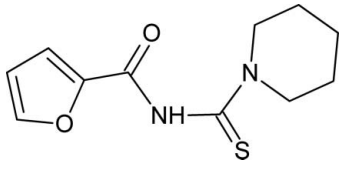
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.068; $wR$ factor = 0.205; data-to-parameter ratio = 16.5.

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
$\mu$ = 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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# supporting information

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

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

**J. Duque, O. Estévez-Hernández, Yvonne Mascarenhas, J. Ellena and Rodrigo S. Corrêa**

### S1. 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).

### S2. 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.

### S3. Refinement

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

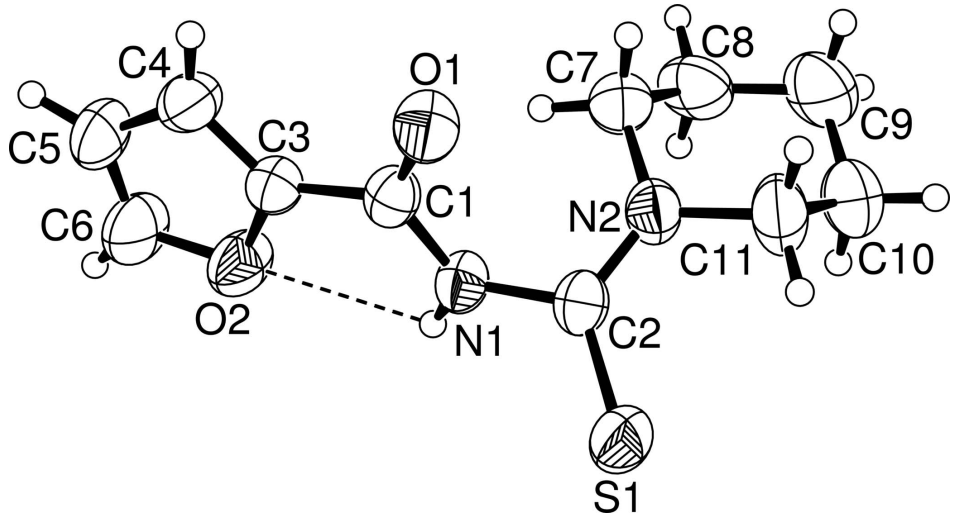


**Figure 1**

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

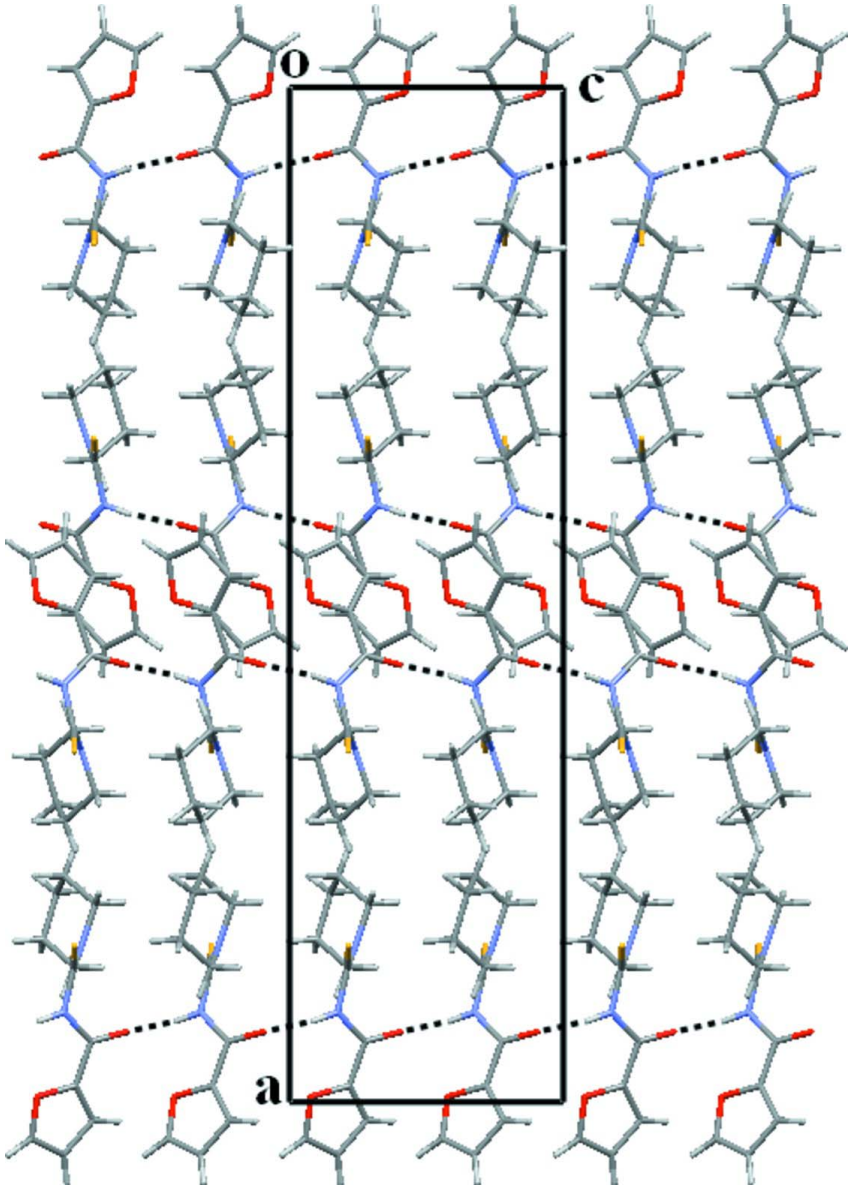


**Figure 2**

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*

$C_{11}H_{14}N_2O_2S$
$M_r = 238.3$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 31.6377\ (15)$ Å
$b = 8.6787\ (4)$ Å
$c = 8.5308\ (3)$ Å
$V = 2342.34\ (18)$ Å$^3$
$Z = 8$

$F(000) = 1008$
$D_x = 1.352$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2684 reflections
$\theta = 2.9–26.4°$
$\mu = 0.26$ mm$^{-1}$
$T = 294$ K
Prism, colourless
0.15 × 0.13 × 0.06 mm

*Data collection*

Nonius KappaCCD diffractometer
CCD rotation images, thick slices scans
4308 measured reflections
2387 independent reflections
1550 reflections with $I > 2\sigma(I)$

$R_{int} = 0.039$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.4°$
$h = -39 \rightarrow 39$
$k = -10 \rightarrow 10$
$l = -10 \rightarrow 10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.205$
$S = 1.11$
2387 reflections
145 parameters

0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1017P)^2 + 1.0533P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å$^{-3}$
$\Delta\rho_{min} = -0.35$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

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

| | | | |
|---|---|---|---|
| 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, º)*

| *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) $x$, $-y+1/2$, $z+1/2$.