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Crystal structure of 2-methyl-*N*-{[2-(pyridin-2-yl)ethyl]carbamothioyl}-benzamide

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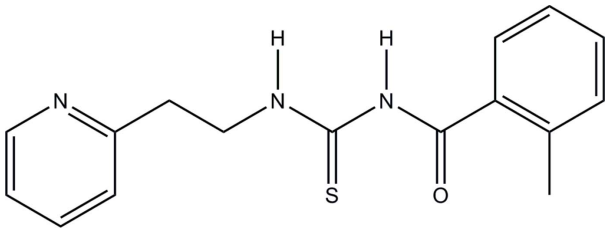
In the title compound, $C_{16}H_{17}N_3OS$, a benzoyl thiourea derivative, the planes of the pyridine and benzene rings are inclined to one another by 66.54 (9)°. There is an intramolecular N—H···O hydrogen bond present forming an *S*(6) ring motif. In the crystal, molecules are linked *via* pairs of N—H···N hydrogen bonds, forming inversion dimers, which are reinforced by pairs of C—H···S hydrogen bonds. The dimers are linked *via* C—H···π interactions, forming ribbons along [010].

Keywords: crystal structure; benzamide; carbonyl thiourea; inversion dimers; hydrogen bonding.

CCDC reference: 1412857

1. Related literature

For the crystal structure of the 4-methyl derivative, 4-methyl-*N*-{[2-(pyridin-2-yl)ethyl]carbamothioyl}benzamide, see: Adam *et al.* (2014). For the crystal structure of *N*-carbamothioyl-2-methylbenzamide, see: Adam *et al.* (2015).



2. Experimental

2.1. Crystal data

$C_{16}H_{17}N_3OS$
M_r = 299.38
Triclinic, $P\overline{1}$
a = 8.5434 (4) Å
b = 8.7477 (4) Å
c = 11.0530 (5) Å
α = 86.1868 (13)°
β = 83.3739 (13)°
γ = 73.8746 (13)°
V = 787.73 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 100 K
0.53 × 0.42 × 0.22 mm

2.2. Data collection

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.670, T_{max} = 0.867
30520 measured reflections
4698 independent reflections
3412 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.129
S = 1.03
4698 reflections
199 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*2 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O1	0.842 (17)	1.971 (18)	2.6550 (16)	137.8 (16)
N1—H1*N*1···N3^{i}	0.875 (17)	2.061 (17)	2.9366 (17)	180 (3)
C16—H16*A*···S1^{i}	0.96	2.86	3.782 (2)	162
C12—H12*A*···*Cg*2^{ii}	0.93	2.92	3.7724 (19)	153

Symmetry codes: (i) $-x+1, -y+2, -z$; (ii) $-x+1, -y+1, -z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* and *PLATON* (Spek, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5173).

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supporting information

Acta Cryst. (2015). E**71**, o636 [https://doi.org/10.1107/S2056989015013559]

Crystal structure of 2-methyl-*N*-{[2-(pyridin-2-yl)ethyl]carbamothioyl}benzamide

Nadiah Ameram and Farook Adam

S1. Synthesis and crystallization

Ortho Benzoyl chloride (13 mmol) was added drop wise to a stirred acetone solution (30 ml) of ammonium thiocyanate (13 mmol). Stirring was continued for 10 min. A solution of ethyl pyridine in acetone was then added and the reaction mixture was refluxed for 3 h, after which the solution was poured into a beaker containing some ice cubes. The resulting precipitate was collected by titration, washed several times with a cold ethanol/water mixture and purified by recrystallization from an ethanol solution, yielding colourless plate-like crystals.

S1.1. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The NH H-atoms were located in a difference Fourier map and freely refined. The C-bound H atoms were positioned geometrically and refined using a riding model: C—H = 0.93–0.96 Å with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

S2. Structural commentary

The title compound, Fig. 1, is a benzoyl thiourea derivative with the pyridine and benzene rings being inclined to one another by 66.54 (9) °. The bond lengths and angles are very similar to those observed for the analogous structure, 4-methyl-N-[2-(pyridin-2-yl)ethylcarbamothioyl]benzamide (Adam *et al.*, 2014). However, in the 4-methyl derivative the pyridine and benzene rings are inclined to one another by 71.33 (15) °. There is an intramolecular N—H···O hydrogen bond present in both molecules forming an S(6) ring motif (for the title compound, see Table 1 and Fig. 1).

In the crystal, molecules are linked *via* pairs of N—H···N hydrogen bonds forming inversion dimers which are reinforced by pairs of C—H···S hydrogen bonds (Fig. 2 and Table 1). The dimers are linked *via* C—H···π interactions forming ribbons along [010]; Table 1 and Fig. 2.

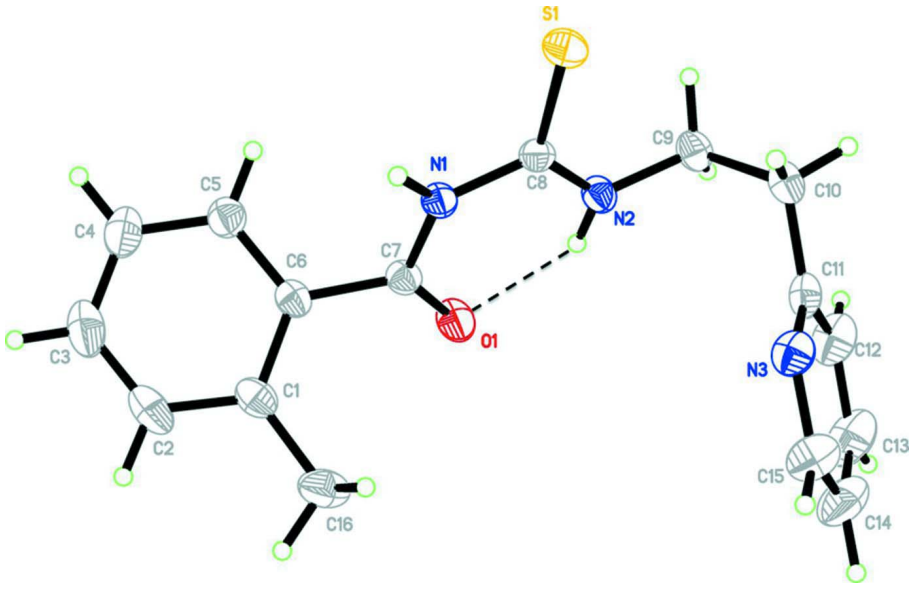

Figure 1

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular N-H···O hydrogen bond is shown as a dashed line (see Table 1 for details).

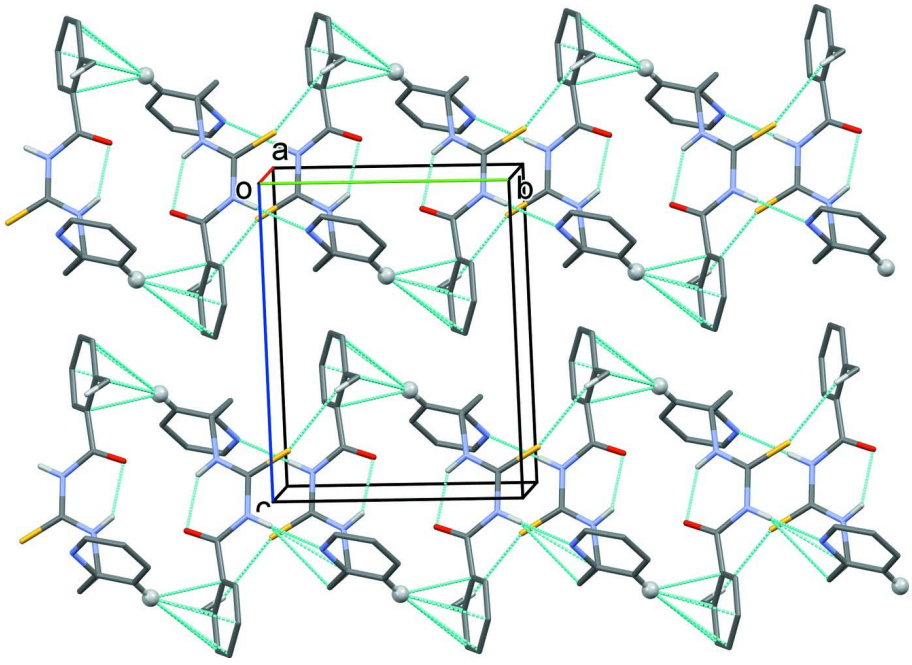

Figure 2

A view along the *a* axis of the crystal packing of the title compound. The hydrogen bonds and C-H···π interactions (H atom a grey ball) are shown as dashed lines (see Table 1 for details).

2-Methyl-*N*-{[2-(pyridin-2-yl)ethyl]carbamothioyl}benzamide

Crystal data

$C_{16}H_{17}N_3OS$
$M_r = 299.38$
Triclinic, $P\overline{1}$
$a = 8.5434$ (4) Å
$b = 8.7477$ (4) Å
$c = 11.0530$ (5) Å
$\alpha = 86.1868$ (13)°
$\beta = 83.3739$ (13)°
$\gamma = 73.8746$ (13)°
$V = 787.73$ (6) Å^3
$Z = 2$
$F(000) = 316$
$D_x = 1.262$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8347 reflections
$\theta = 2.4$–$28.3°$
$\mu = 0.21$ mm^{-1}
$T = 100$ K
Plate, colourless
0.53 × 0.42 × 0.22 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.670$, $T_{max} = 0.867$
30520 measured reflections
4698 independent reflections
3412 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 30.3°$, $\theta_{min} = 1.9°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.129$
$S = 1.03$
4698 reflections
199 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.2342P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.29858 (5)	1.05179 (5)	−0.14484 (4)	0.05473 (14)
O1	0.46415 (15)	0.62159 (12)	0.10945 (10)	0.0552 (3)
N1	0.32417 (14)	0.87622 (13)	0.05803 (10)	0.0367 (2)
N2	0.47718 (15)	0.75403 (15)	−0.11337 (11)	0.0408 (3)
N3	0.87338 (16)	0.81820 (15)	−0.15598 (13)	0.0499 (3)
C1	0.3515 (2)	0.72438 (18)	0.36636 (14)	0.0482 (3)
C2	0.2522 (3)	0.7379 (2)	0.47681 (15)	0.0654 (5)
H2A	0.3012	0.7083	0.5488	0.079*

C3	0.0847 (3)	0.7934 (3)	0.48282 (18)	0.0731 (6)
H3A	0.0226	0.7999	0.5581	0.088*
C4	0.0088 (2)	0.8393 (2)	0.37890 (18)	0.0638 (5)
H4A	−0.1046	0.8769	0.3828	0.077*
C5	0.10295 (19)	0.82906 (18)	0.26820 (15)	0.0478 (3)
H5A	0.0522	0.8619	0.1972	0.057*
C6	0.27224 (18)	0.77065 (15)	0.26075 (12)	0.0392 (3)
C7	0.36489 (17)	0.74777 (16)	0.13728 (12)	0.0384 (3)
C8	0.37507 (16)	0.88350 (16)	−0.06595 (12)	0.0359 (3)
C9	0.5275 (2)	0.73791 (19)	−0.24280 (13)	0.0462 (3)
H9A	0.4348	0.7906	−0.2875	0.055*
H9B	0.5589	0.6259	−0.2610	0.055*
C10	0.6698 (2)	0.80764 (19)	−0.28709 (14)	0.0487 (3)
H10A	0.6926	0.7974	−0.3746	0.058*
H10B	0.6371	0.9203	−0.2709	0.058*
C11	0.82339 (18)	0.73009 (17)	−0.22889 (14)	0.0445 (3)
C12	0.9112 (2)	0.5738 (2)	−0.2508 (2)	0.0661 (5)
H12A	0.8745	0.5138	−0.3019	0.079*
C13	1.0517 (3)	0.5084 (2)	−0.1970 (3)	0.0805 (7)
H13A	1.1105	0.4032	−0.2102	0.097*
C14	1.1056 (2)	0.5992 (3)	−0.1232 (2)	0.0779 (6)
H14A	1.2021	0.5582	−0.0867	0.093*
C15	1.0123 (2)	0.7523 (2)	−0.1053 (2)	0.0676 (5)
H15A	1.0477	0.8142	−0.0549	0.081*
C16	0.5335 (2)	0.6686 (3)	0.36477 (18)	0.0661 (5)
H16A	0.5808	0.7494	0.3264	0.099*
H16B	0.5743	0.5728	0.3200	0.099*
H16C	0.5624	0.6477	0.4468	0.099*
H1N2	0.508 (2)	0.676 (2)	−0.0647 (16)	0.049 (5)*
H1N1	0.265 (2)	0.967 (2)	0.0876 (16)	0.049 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0565 (2)	0.0474 (2)	0.0519 (2)	−0.00366 (17)	−0.00764 (18)	0.01618 (17)
O1	0.0692 (7)	0.0369 (5)	0.0446 (6)	0.0061 (5)	0.0018 (5)	0.0038 (4)
N1	0.0395 (6)	0.0309 (5)	0.0360 (6)	−0.0043 (4)	−0.0032 (4)	0.0010 (4)
N2	0.0463 (6)	0.0384 (6)	0.0355 (6)	−0.0096 (5)	−0.0016 (5)	0.0026 (5)
N3	0.0483 (7)	0.0395 (6)	0.0586 (8)	−0.0079 (5)	−0.0005 (6)	−0.0052 (6)
C1	0.0641 (9)	0.0415 (7)	0.0405 (7)	−0.0166 (7)	−0.0103 (7)	0.0054 (6)
C2	0.0994 (15)	0.0627 (11)	0.0355 (8)	−0.0253 (10)	−0.0080 (9)	0.0049 (7)
C3	0.0886 (15)	0.0695 (12)	0.0523 (10)	−0.0179 (11)	0.0201 (10)	−0.0027 (9)
C4	0.0601 (10)	0.0592 (10)	0.0635 (11)	−0.0100 (8)	0.0140 (9)	−0.0032 (8)
C5	0.0506 (8)	0.0436 (8)	0.0458 (8)	−0.0086 (6)	−0.0015 (6)	−0.0008 (6)
C6	0.0499 (8)	0.0310 (6)	0.0357 (7)	−0.0099 (5)	−0.0038 (6)	0.0006 (5)
C7	0.0430 (7)	0.0331 (6)	0.0368 (7)	−0.0067 (5)	−0.0054 (5)	0.0006 (5)
C8	0.0345 (6)	0.0375 (6)	0.0369 (6)	−0.0111 (5)	−0.0072 (5)	0.0028 (5)
C9	0.0559 (9)	0.0488 (8)	0.0360 (7)	−0.0176 (7)	−0.0025 (6)	−0.0056 (6)

C10	0.0598 (9)	0.0483 (8)	0.0374 (7)	−0.0171 (7)	0.0017 (6)	0.0012 (6)
C11	0.0474 (8)	0.0380 (7)	0.0449 (8)	−0.0131 (6)	0.0112 (6)	−0.0009 (6)
C12	0.0633 (11)	0.0428 (9)	0.0883 (14)	−0.0116 (8)	0.0089 (10)	−0.0158 (9)
C13	0.0581 (11)	0.0424 (9)	0.128 (2)	0.0000 (8)	0.0105 (12)	−0.0077 (11)
C14	0.0437 (9)	0.0661 (12)	0.1145 (18)	−0.0032 (9)	−0.0051 (10)	0.0084 (12)
C15	0.0534 (10)	0.0612 (11)	0.0871 (14)	−0.0109 (8)	−0.0126 (9)	−0.0062 (10)
C16	0.0702 (12)	0.0708 (12)	0.0607 (11)	−0.0217 (9)	−0.0261 (9)	0.0174 (9)

Geometric parameters (Å, º)

S1—C8	1.6675 (13)	C5—H5A	0.9300
O1—C7	1.2236 (16)	C6—C7	1.4926 (19)
N1—C7	1.3686 (17)	C9—C10	1.525 (2)
N1—C8	1.3919 (17)	C9—H9A	0.9700
N1—H1N1	0.874 (18)	C9—H9B	0.9700
N2—C8	1.3203 (18)	C10—C11	1.496 (2)
N2—C9	1.4499 (18)	C10—H10A	0.9700
N2—H1N2	0.841 (19)	C10—H10B	0.9700
N3—C11	1.331 (2)	C11—C12	1.387 (2)
N3—C15	1.336 (2)	C12—C13	1.364 (3)
C1—C2	1.396 (2)	C12—H12A	0.9300
C1—C6	1.397 (2)	C13—C14	1.372 (3)
C1—C16	1.494 (3)	C13—H13A	0.9300
C2—C3	1.372 (3)	C14—C15	1.369 (3)
C2—H2A	0.9300	C14—H14A	0.9300
C3—C4	1.366 (3)	C15—H15A	0.9300
C3—H3A	0.9300	C16—H16A	0.9600
C4—C5	1.378 (2)	C16—H16B	0.9600
C4—H4A	0.9300	C16—H16C	0.9600
C5—C6	1.388 (2)		

C7—N1—C8	127.33 (12)	N2—C9—H9A	108.9
C7—N1—H1N1	118.0 (12)	C10—C9—H9A	108.9
C8—N1—H1N1	114.5 (12)	N2—C9—H9B	108.9
C8—N2—C9	123.54 (13)	C10—C9—H9B	108.9
C8—N2—H1N2	116.3 (12)	H9A—C9—H9B	107.7
C9—N2—H1N2	120.0 (12)	C11—C10—C9	113.91 (13)
C11—N3—C15	118.03 (15)	C11—C10—H10A	108.8
C2—C1—C6	116.77 (16)	C9—C10—H10A	108.8
C2—C1—C16	120.27 (16)	C11—C10—H10B	108.8
C6—C1—C16	122.91 (15)	C9—C10—H10B	108.8
C3—C2—C1	122.25 (17)	H10A—C10—H10B	107.7
C3—C2—H2A	118.9	N3—C11—C12	121.26 (16)
C1—C2—H2A	118.9	N3—C11—C10	117.10 (13)
C4—C3—C2	120.40 (17)	C12—C11—C10	121.64 (16)
C4—C3—H3A	119.8	C13—C12—C11	119.68 (19)
C2—C3—H3A	119.8	C13—C12—H12A	120.2
C3—C4—C5	118.99 (18)	C11—C12—H12A	120.2

C3—C4—H4A	120.5	C12—C13—C14	119.45 (18)
C5—C4—H4A	120.5	C12—C13—H13A	120.3
C4—C5—C6	121.16 (16)	C14—C13—H13A	120.3
C4—C5—H5A	119.4	C15—C14—C13	117.6 (2)
C6—C5—H5A	119.4	C15—C14—H14A	121.2
C5—C6—C1	120.41 (14)	C13—C14—H14A	121.2
C5—C6—C7	118.19 (13)	N3—C15—C14	123.9 (2)
C1—C6—C7	121.25 (13)	N3—C15—H15A	118.0
O1—C7—N1	123.59 (13)	C14—C15—H15A	118.0
O1—C7—C6	121.89 (12)	C1—C16—H16A	109.5
N1—C7—C6	114.47 (11)	C1—C16—H16B	109.5
N2—C8—N1	117.23 (12)	H16A—C16—H16B	109.5
N2—C8—S1	124.66 (11)	C1—C16—H16C	109.5
N1—C8—S1	118.08 (10)	H16A—C16—H16C	109.5
N2—C9—C10	113.56 (12)	H16B—C16—H16C	109.5

C6—C1—C2—C3	0.1 (3)	C9—N2—C8—N1	173.37 (12)
C16—C1—C2—C3	177.89 (19)	C9—N2—C8—S1	−4.5 (2)
C1—C2—C3—C4	−0.6 (3)	C7—N1—C8—N2	−1.7 (2)
C2—C3—C4—C5	0.0 (3)	C7—N1—C8—S1	176.26 (11)
C3—C4—C5—C6	1.1 (3)	C8—N2—C9—C10	84.03 (18)
C4—C5—C6—C1	−1.6 (2)	N2—C9—C10—C11	61.39 (18)
C4—C5—C6—C7	174.02 (15)	C15—N3—C11—C12	0.8 (2)
C2—C1—C6—C5	0.9 (2)	C15—N3—C11—C10	−178.50 (15)
C16—C1—C6—C5	−176.76 (16)	C9—C10—C11—N3	−113.16 (15)
C2—C1—C6—C7	−174.53 (14)	C9—C10—C11—C12	67.54 (19)
C16—C1—C6—C7	7.8 (2)	N3—C11—C12—C13	−0.1 (3)
C8—N1—C7—O1	5.5 (2)	C10—C11—C12—C13	179.20 (17)
C8—N1—C7—C6	−171.85 (12)	C11—C12—C13—C14	−1.0 (3)
C5—C6—C7—O1	−128.16 (16)	C12—C13—C14—C15	1.2 (3)
C1—C6—C7—O1	47.4 (2)	C11—N3—C15—C14	−0.5 (3)
C5—C6—C7—N1	49.22 (18)	C13—C14—C15—N3	−0.5 (4)
C1—C6—C7—N1	−135.22 (14)		

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O1	0.842 (17)	1.971 (18)	2.6550 (16)	137.8 (16)
N1—H1*N*1···N3^{i}	0.875 (17)	2.061 (17)	2.9366 (17)	180 (3)
C16—H16*A*···S1^{i}	0.96	2.86	3.782 (2)	162
C12—H12*A*···*Cg*2^{ii}	0.93	2.92	3.7724 (19)	153

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