STRUCTURE REPORTS

OPEN ACCESS

ISSN 1600-5368

Crystal structure of (*E*)-*N*-phenyl-*N*′-[1-(thiophen-2-yl)ethylidene]formohydrazide

C. S. Dileep,[a] K. R. Raghavendra,[b] N. K. Lokanath,[a] K. Ajay Kumar[c] and M. A. Sridhar[a]*

[a]Department of Studies in Physics, Manasagangotri, University of Mysore, Mysore 570 006, India, [b]Department of Chemistry, SBRR Mahajana College, Mysore 570 006, India, and [c]Post Graduate Department of Chemistry, Yuvaraja College, University of Mysore, Mysore 570 005, India. *Correspondence e-mail: mas@physics.uni-mysore.ac.in

Received 9 July 2014; accepted 16 July 2014

Edited by H. Ishida, Okayama University, Japan

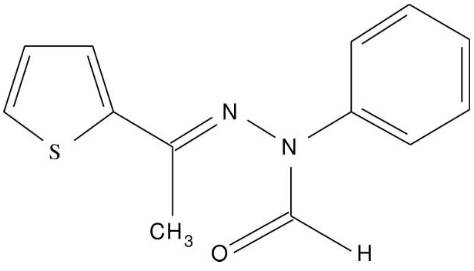
In the title compound, $C_{13}H_{12}N_2OS$, the planes of the thiophene and phenyl rings are nearly perpendicular to each other, making a dihedral angle of 86.42 (12)°. In the crystal, molecules are linked by C—H⋯O hydrogen bonds, forming a helical chain along the *b*-axis direction.

Keywords: crystal structure; thiophene derivative; hydrogen bonding.

CCDC reference: 1014287

1. Related literature

For the biological activity of thiophene derivatives, see: Bondock *et al.* (2010); Bellina *et al.* (2007); Konstantinova *et al.* (2009); Al-Said *et al.* (2011). For the crystal structure of a similar compound, *viz.* (*E*)-*N*′-[1-(thiophen-2-yl)ethylidene]-benzohydrazide, see: Shan *et al.* (2011). For a description of the Cambridge Structural Database, see: Allen (2002).



2. Experimental

2.1. Crystal data

$C_{13}H_{12}N_2OS$
$M_r = 244.32$
Orthorhombic, $P2_12_12_1$
$a = 5.4960$ (7) Å
$b = 11.0177$ (13) Å
$c = 20.249$ (2) Å
$V = 1226.1$ (2) Å^3
$Z = 4$
Cu *Kα* radiation
$\mu = 2.22$ mm^{-1}
$T = 296$ K
0.25 × 0.22 × 0.20 mm

2.2. Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{\min} = 0.604$, $T_{\max} = 0.662$
6298 measured reflections
2010 independent reflections
1904 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.042$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.102$
$S = 1.10$
2010 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983), 805 Friedel pairs
Absolute structure parameter: 0.02 (2)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C1—H1⋯O1$^{\rm i}$	0.93	2.39	3.202 (3)	145

Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2009).

Acknowledgements

The authors would like to thank the University of Mysore for providing the diffractometer facility under the Institution of Excellence. CSD would like to thank the University of Mysore for the award of an RFSMS fellowship under the head DV5/Physics/389/RFSMS/2009–2010/10.07.2012.

Supporting information for this paper is available from the IUCr electronic archives (Reference: IS5369).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Al-Said, M. S., Bashandy, M. S., Al-qasoumi, S. I. & Ghorab, M. M. (2011). *Eur. J. Med. Chem.* **46**, 137–141.
Bellina, F., Cauteruccio, S. & Rossi, R. (2007). *Tetrahedron*, **63**, 4571–4624.
Bondock, S., Fadaly, W. & Metwally, M. A. (2010). *Eur. J. Med. Chem.* **45**, 3692–3701.
Bruker (2013). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Konstantinova, L. S., Bolshakov, O. I., Obruchnikova, N. V., Laborie, H., Tanga, A., Sopena, V., Lanneluc, I., Picot, L., Sable, S., Thiery, V. & Rakitin, O. A. (2009). *Bioorg. Med. Chem. Lett.* **19**, 136–141.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.

Shan, S., Huang, Y.-L., Guo, H.-Q., Li, D.-F. & Sun, J. (2011). *Acta Cryst.* E**67**, o2498.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Acta Cryst. (2014). E**70**, o928–o929 Dileep *et al.* • $C_{13}H_{12}N_2OS$ **o929**

supporting information

Acta Cryst. (2014). E**70**, o928–o929 [doi:10.1107/S1600536814016511]

Crystal structure of (*E*)-*N*-phenyl-*N*′-[1-(thiophen-2-yl)ethylidene]formohydrazide

C. S. Dileep, K. R. Raghavendra, N. K. Lokanath, K. Ajay Kumar and M. A. Sridhar

S1. Comment

In medicinal chemistry, thiophene derivatives have been very well known for their therapeutic applications. Many thiophene derivatives have been developed as chemotherapeutic agents and are extensively used. Thiophene nucleus is one of the most important heterocycles exhibiting remarkable pharmacological activities. The great interest in the synthesis of thiophene derivatives due to their diverse biological and chemical properties. Thiophene, as a prominent structural motif, is found in numerous active compounds, which contain 5-membered heterocyclic structure have attracted a lot of interests in many fields, and its rich biological activity in medicinal chemistry owing to their biological properties. Thiophene and thiazole derivatives are known to possess interesting biological properties like anticancer (Bondock *et al.*, 2010; Bellina *et al.*, 2007; Konstantinova *et al.*, 2009). Thiophene or benzothiophene moieties due to the well documented anti-cancer activity of these moieties to study their SAR and their anti-breast cancer activity (Al-Said *et al.*, 2011). In view of their importance as discussed above, thiophene derivatives were taken for their conformational studies to get better structural activity correlation.

In the title compound (Fig. 1), the bond lengths do not show much variation in the core structure of the derivatives, and are similar to the standard values (Allen *et al.*, 2002). The thiophene (S1/C1–C4) and phenyl (C8–C13) rings are nearly perpendicular with a dihedral angle of 86.42 (12)° between their mean planes. The bond lengths and bond angles do not show large deviations and are comparable with those reported for a similar structure (Shan *et al.*, 2011). The conformation of the attachment of the thiophene and phenyl rings can also be characterized by torsion angles of (C4—C5—N1—N2), (C5—N1—N2—C8), (O1—C7—N2—C8) and (S1—C4—C5—C6) being 178.38, 127.73, 171.34 and -170.31°, respectively. The crystal structure has an intermolecular C—H···O hydrogen bond. The molecular packing viewed down the *a* axis is shown in Fig. 2.

S2. Experimental

A mixture of (*E*)-1-phenyl-2-[(1-thiophen-2-yl)ethylidene]hydrazine (0.176 mmol) were added to the Vilsmeier-Haack reagent prepared by drop-wise addition of $POCl_3$ (1.2 ml) in ice cooled DMF (5 ml). The mixture was stirred at 60–65 °C for 6 h. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was poured into ice cold water, neutralized with $NaHCO_3$, the solid separated was filtered, washed with water and recrystallized from ethanol to get the compound in 93% yield.

S3. Refinement

All H atoms were located from difference maps and were positioned geometrically and refined using a riding model with C—H = 0.93–0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

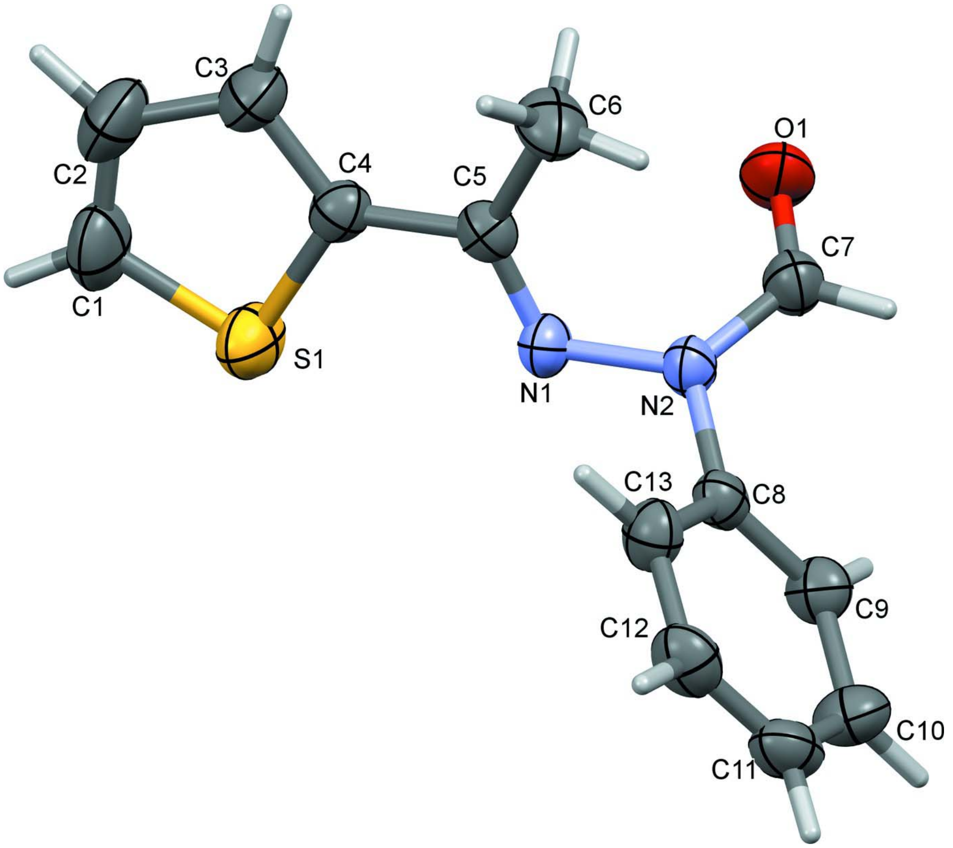


Figure 1

ORTEP view of the molecule with the atom-labeling scheme. The displacement ellipsoids are drawn at the 50% probability level.

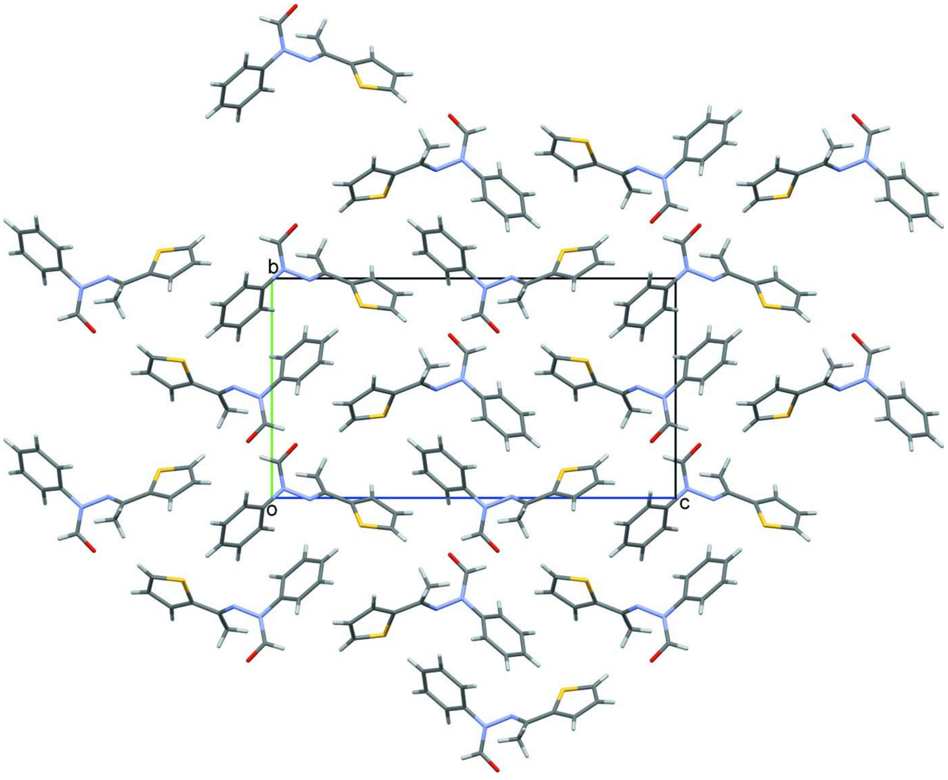


Figure 2

A molecular packing view of the title compound down the *a*-axis.

(*E*)-*N*-Phenyl-*N′*-[1-(thiophen-2-yl)ethylidene]formohydrazide.

Crystal data

$C_{13}H_{12}N_2OS$
$M_r = 244.32$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.4960$ (7) Å
$b = 11.0177$ (13) Å
$c = 20.249$ (2) Å
$V = 1226.1$ (2) Å^3
$Z = 4$
$F(000) = 512$
$D_x = 1.324$ Mg m^{-3}
Cu *Kα* radiation, $\lambda = 1.54178$ Å
Cell parameters from 6298 reflections
$\theta = 4.4$–64.6°
$\mu = 2.22$ mm^{-1}
$T = 296$ K
Block, pale yellow
0.25 × 0.22 × 0.20 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 10.7 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{\min} = 0.604$, $T_{\max} = 0.662$
6298 measured reflections
2010 independent reflections
1904 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.042$
$\theta_{\max} = 64.6°$, $\theta_{\min} = 4.4°$
$h = -2 \rightarrow 6$
$k = -12 \rightarrow 12$
$l = -23 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.102$
$S = 1.10$
2010 reflections
165 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.0861P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL*, $FC^*=KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Extinction coefficient: 0.0158 (16)
Absolute structure: Flack (1983), 805 Friedel pairs
Absolute structure parameter: 0.02 (2)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.05502 (12)	0.12455 (6)	0.72104 (3)	0.0539 (2)
O1	0.0434 (4)	−0.24121 (15)	0.55887 (8)	0.0526 (6)
N1	0.0078 (3)	0.00557 (16)	0.59394 (8)	0.0348 (5)
N2	0.0086 (3)	−0.04249 (15)	0.52882 (8)	0.0344 (5)
C1	−0.0682 (6)	0.1399 (3)	0.79702 (12)	0.0623 (10)
C2	−0.2635 (6)	0.0712 (3)	0.80591 (13)	0.0648 (10)
C3	−0.3227 (5)	0.0016 (2)	0.74907 (11)	0.0513 (8)
C4	−0.1639 (4)	0.02325 (18)	0.69777 (10)	0.0358 (6)
C5	−0.1724 (4)	−0.02503 (18)	0.63061 (10)	0.0331 (6)
C6	−0.3905 (4)	−0.0972 (2)	0.61062 (13)	0.0509 (8)
C7	0.0373 (4)	−0.16179 (19)	0.51733 (10)	0.0410 (7)
C8	0.0373 (4)	0.04484 (17)	0.47710 (9)	0.0325 (6)
C9	0.2273 (4)	0.0366 (2)	0.43330 (11)	0.0397 (6)
C10	0.2429 (4)	0.1192 (2)	0.38203 (12)	0.0479 (7)
C11	0.0710 (5)	0.2091 (2)	0.37516 (11)	0.0469 (7)
C12	−0.1151 (5)	0.2185 (2)	0.41975 (12)	0.0463 (8)
C13	−0.1322 (4)	0.1365 (2)	0.47156 (11)	0.0413 (6)
H1	−0.00560	0.19130	0.82930	0.0750*
H2	−0.35200	0.06880	0.84500	0.0780*
H3	−0.45300	−0.05220	0.74680	0.0620*
H6A	−0.39060	−0.10770	0.56360	0.0760*
H6B	−0.53550	−0.05510	0.62380	0.0760*

H6C	−0.38540	−0.17530	0.63170	0.0760*
H7	0.05420	−0.18570	0.47350	0.0490*
H9	0.34410	−0.02390	0.43800	0.0480*
H10	0.37060	0.11400	0.35200	0.0570*
H11	0.08130	0.26340	0.34010	0.0560*
H12	−0.23010	0.27990	0.41530	0.0560*
H13	−0.25720	0.14340	0.50230	0.0500*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0546 (4)	0.0703 (5)	0.0368 (3)	−0.0147 (3)	−0.0029 (3)	−0.0071 (3)
O1	0.0729 (11)	0.0375 (9)	0.0474 (9)	0.0049 (8)	0.0049 (8)	0.0096 (7)
N1	0.0402 (10)	0.0379 (9)	0.0263 (8)	−0.0024 (8)	−0.0012 (7)	−0.0037 (6)
N2	0.0452 (10)	0.0313 (8)	0.0266 (8)	0.0006 (8)	−0.0005 (7)	−0.0022 (7)
C1	0.085 (2)	0.0689 (17)	0.0330 (12)	0.0037 (16)	−0.0059 (13)	−0.0095 (11)
C2	0.0757 (18)	0.0819 (19)	0.0368 (13)	0.0055 (16)	0.0152 (13)	0.0000 (13)
C3	0.0524 (14)	0.0590 (15)	0.0426 (13)	−0.0041 (12)	0.0127 (11)	−0.0022 (11)
C4	0.0373 (10)	0.0370 (11)	0.0332 (10)	0.0038 (9)	0.0009 (8)	0.0026 (8)
C5	0.0317 (10)	0.0314 (10)	0.0362 (11)	0.0037 (8)	−0.0030 (8)	0.0008 (8)
C6	0.0372 (12)	0.0564 (15)	0.0592 (14)	−0.0111 (10)	0.0008 (11)	−0.0119 (12)
C7	0.0511 (12)	0.0353 (11)	0.0366 (11)	0.0020 (9)	0.0024 (10)	−0.0018 (9)
C8	0.0388 (10)	0.0296 (10)	0.0291 (9)	−0.0017 (8)	−0.0051 (8)	−0.0012 (8)
C9	0.0355 (10)	0.0392 (11)	0.0444 (12)	0.0049 (9)	0.0011 (9)	0.0016 (10)
C10	0.0456 (12)	0.0511 (13)	0.0469 (13)	−0.0056 (11)	0.0089 (10)	0.0094 (11)
C11	0.0604 (14)	0.0365 (11)	0.0439 (12)	−0.0080 (11)	−0.0054 (11)	0.0088 (9)
C12	0.0536 (14)	0.0350 (12)	0.0503 (13)	0.0084 (11)	−0.0087 (11)	0.0024 (10)
C13	0.0429 (11)	0.0416 (11)	0.0395 (11)	0.0083 (10)	0.0012 (10)	−0.0035 (10)

Geometric parameters (Å, º)

S1—C1	1.690 (3)	C10—C11	1.376 (3)
S1—C4	1.707 (2)	C11—C12	1.368 (4)
O1—C7	1.214 (3)	C12—C13	1.388 (3)
N1—N2	1.421 (2)	C1—H1	0.9300
N1—C5	1.283 (3)	C2—H2	0.9300
N2—C7	1.344 (3)	C3—H3	0.9300
N2—C8	1.431 (2)	C6—H6A	0.9600
C1—C2	1.326 (5)	C6—H6B	0.9600
C2—C3	1.421 (4)	C6—H6C	0.9600
C3—C4	1.378 (3)	C7—H7	0.9300
C4—C5	1.461 (3)	C9—H9	0.9300
C5—C6	1.494 (3)	C10—H10	0.9300
C8—C9	1.373 (3)	C11—H11	0.9300
C8—C13	1.379 (3)	C12—H12	0.9300
C9—C10	1.383 (3)	C13—H13	0.9300
C1—S1—C4	91.96 (13)	C2—C1—H1	123.00

N2—N1—C5	116.20 (17)	C1—C2—H2	124.00
N1—N2—C7	121.70 (16)	C3—C2—H2	124.00
N1—N2—C8	115.40 (15)	C2—C3—H3	124.00
C7—N2—C8	121.19 (16)	C4—C3—H3	124.00
S1—C1—C2	113.0 (2)	C5—C6—H6A	109.00
C1—C2—C3	112.6 (2)	C5—C6—H6B	109.00
C2—C3—C4	111.9 (2)	C5—C6—H6C	109.00
S1—C4—C3	110.58 (16)	H6A—C6—H6B	109.00
S1—C4—C5	121.16 (16)	H6A—C6—H6C	109.00
C3—C4—C5	128.2 (2)	H6B—C6—H6C	109.00
N1—C5—C4	114.73 (19)	O1—C7—H7	117.00
N1—C5—C6	127.04 (19)	N2—C7—H7	117.00
C4—C5—C6	118.13 (19)	C8—C9—H9	120.00
O1—C7—N2	126.02 (19)	C10—C9—H9	120.00
N2—C8—C9	120.81 (18)	C9—C10—H10	120.00
N2—C8—C13	118.53 (18)	C11—C10—H10	120.00
C9—C8—C13	120.65 (19)	C10—C11—H11	120.00
C8—C9—C10	119.2 (2)	C12—C11—H11	120.00
C9—C10—C11	120.5 (2)	C11—C12—H12	120.00
C10—C11—C12	120.1 (2)	C13—C12—H12	120.00
C11—C12—C13	120.0 (2)	C8—C13—H13	120.00
C8—C13—C12	119.5 (2)	C12—C13—H13	120.00
S1—C1—H1	124.00		

C4—S1—C1—C2	0.9 (3)	C2—C3—C4—C5	−176.1 (2)
C1—S1—C4—C3	−1.3 (2)	C2—C3—C4—S1	1.4 (3)
C1—S1—C4—C5	176.4 (2)	S1—C4—C5—N1	6.3 (3)
C5—N1—N2—C7	−67.0 (2)	C3—C4—C5—N1	−176.4 (2)
C5—N1—N2—C8	127.7 (2)	C3—C4—C5—C6	7.0 (3)
N2—N1—C5—C4	178.38 (16)	S1—C4—C5—C6	−170.31 (16)
N2—N1—C5—C6	−5.3 (3)	N2—C8—C9—C10	176.94 (19)
C7—N2—C8—C13	135.3 (2)	C13—C8—C9—C10	−2.1 (3)
N1—N2—C7—O1	6.9 (3)	N2—C8—C13—C12	−176.7 (2)
C8—N2—C7—O1	171.3 (2)	C9—C8—C13—C12	2.3 (3)
N1—N2—C8—C9	121.6 (2)	C8—C9—C10—C11	0.4 (3)
C7—N2—C8—C9	−43.7 (3)	C9—C10—C11—C12	1.0 (4)
N1—N2—C8—C13	−59.4 (2)	C10—C11—C12—C13	−0.7 (4)
S1—C1—C2—C3	−0.3 (4)	C11—C12—C13—C8	−0.9 (3)
C1—C2—C3—C4	−0.7 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1^{i}	0.93	2.39	3.202 (3)	145

Symmetry code: (i) −*x*, *y*+1/2, −*z*+3/2.