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(*E*)-1-(3-Nitrophenyl)-2-({5-[(1*E*)-2-(3-nitrophenyl)hydrazin-1-ylidenemethyl]-2-thienyl}methylidene)hydrazine

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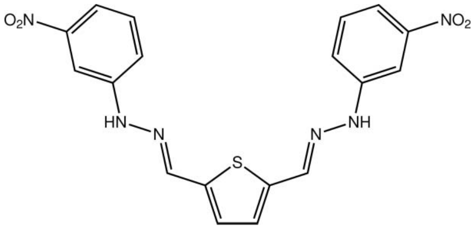
Key indicators: single-crystal X-ray study; *T* = 120 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.046; *wR* factor = 0.133; data-to-parameter ratio = 15.6.

The title molecule, $C_{18}H_{14}N_6O_4S$, adopts a U-shape with the aromatic groups lying *syn* and oriented in the same direction as the thiophene S atom. Twists away from planarity are evident with the maximum deviation being found for a terminal nitro group: C/C/N/O = 19.0 (3)°. The conformation about each of the C=N bonds is *E*. In the crystal, centrosymmetrically related molecules are connected *via* $\text{N}-\text{H}\cdots\text{O}_{\text{nitro}}$ hydrogen bonds, forming 14-membered $\{\cdots\text{HNC}_3\text{NO}\}_2$ synthons. These are linked into layers *via* $\text{C}-\text{H}\cdots\text{O}_{\text{nitro}}$ interactions with the primary interactions between layers being of the type $\text{C}-\text{H}\cdots\pi$, where the π-system is the thiophene ring.

Related literature

For the preparation of hydrazones of thiophenecarbaldehydes, see: Kwon *et al.* (2009); Wardell *et al.* (2007); Vaysse & Pastour (1964). For general uses of 2-substituted-thiophenes, see: Campaigne (1984). For their specific uses as materials, see: Michaleviciute *et al.* (2007, 2009); Kwon *et al.* (2009). For their specific uses as pharmacological agents, see: Kleemann *et al.* (2006); Sonar & Crooks (2009); Mellado *et al.* (2009); Satyanarayana *et al.* (2008); Lourenço *et al.* (2007). For related structures, see: Wardell *et al.* (2007, 2010); Ferreira *et al.* (2009); Nogueira *et al.* (2010).



Experimental

Crystal data

$C_{18}H_{14}N_6O_4S$
M_r = 410.41
Monoclinic, $P2_1/c$
a = 11.1790 (5) Å
b = 20.6993 (9) Å
c = 8.0334 (2) Å
β = 100.513 (2)°
V = 1827.70 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 120 K
0.62 × 0.10 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
$T_{\min}$ = 0.668, $T_{\max}$ = 0.746
21780 measured reflections
4183 independent reflections
3001 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.133
S = 1.08
4183 reflections
268 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.27 e Å^{-3}
$\Delta\rho_{\min}$ = −0.34 e Å^{-3}

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2n}\cdots\text{O2}^{\text{i}}$	0.89 (2)	2.27 (2)	3.103 (2)	156 (2)
$\text{C2}-\text{H2}\cdots\text{O3}^{\text{ii}}$	0.95	2.46	3.278 (3)	145
$\text{C18}-\text{H18}\cdots\text{O4}^{\text{iii}}$	0.95	2.48	3.241 (3)	137
$\text{C12}-\text{H12}\cdots Cg1^{\text{iv}}$	0.95	2.58	3.323 (2)	135

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $x-1, y, z+1$; (iii) $x, y, z+1$; (iv) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2635).

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References

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Campaigne, E. (1984). *Comprehensive Heterocyclic Chemistry*, Vol. 4, edited by A. R. Katritzky & C. W. Rees, pp. 863–934. Oxford: Pergamon.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Ferreira de Lima, M., de Souza, M. V. N., Tiekink, E. R. T., Wardell, J. L. & Wardell, S. M. S. V. (2009). *Acta Cryst.* E**65**, o3203.

Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Kleemann, A., Engel, J. B., Kutscher, B. & Reichert, D. (2006). In *Pharmaceutical Substances*. New York, Stuttgart: Georg Thieme Verlag.

Kwon, O.-P., Jazbinsek, M., Seo, J.-I., Kim, P.-J., Yun, H., Lee, Y. S. & Gunter, P. (2009). *J. Phys. Chem. C*, **113**, 15405–15411.

Lourenço, M. C. S., Vicente, F. R., Henriques, M., das, G. M. de O., Candéa, A. L. P., Gonçalves, R. S. B., Nogueira, T. C. M., Ferreira, M. de L. & de Souza, M. V. N. (2007). *Bioorg. Med. Chem. Lett.* **17**, 6895–6898.

Mellado, O. G. & Cortes, E. C. (2009). Mex. Pat. Appl. CODEN: MXXXA3 MX 2007012608 A 20090413.

Michaleviciute, A., Buika, G., Grazulevicius, J. V., Tran-Van, F., Chevrot, C. & Jankauskas, V. (2007). *Mol. Cryst. Liq. Cryst*. **468**, 459–470.

Michaleviciute, A., Lygaitis, R., Grazulevicius, J. V., Buika, G., Jankauskas, V., Undzenas, A. & Fataraite, E. (2009). *Synth. Met.* **159**, 223–227.

Nogueira, T. C. M., de Souza, M. V. N., Wardell, J. L., Wardell, S. M. S. V. & Tiekink, E. R. T. (2010). *Acta Cryst.* E**66**, o177.

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.

Satyanarayana, V. S. V., Sreevani, P., Sivakumar, A. & Vijayakumar, V. (2008). *ARKIVOC*, pp. 221–233.

Sheldrick, G. M. (2007). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

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

Sonar, V. N. & Crooks, P. A. (2009). *J. Enz. Inhib. Med. Chem.* **24**, 117–124

Vaysse, M. & Pastour, P. (1964). *Compt. Rend.* **259**, 2657–2659.

Wardell, S. M. S. V., de Lima, G. M., Tiekink, E. R. T. & Wardell, J. L. (2010). *Acta Cryst.* E**66**, o271–o272.

Wardell, J. L., Low, J. N. & Glidewell, C. (2007). *Acta Cryst.* E**63**, o1848–o1850.

Westrip, S. P. (2010). *publCIF*. In preparation.

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supporting information

Acta Cryst. (2010). E**66**, o457–o458   [https://doi.org/10.1107/S1600536810002771]

(*E*)-1-(3-Nitrophenyl)-2-({5-[(1*E*)-2-(3-nitrophenyl)hydrazin-1-ylidenemethyl]-2-thienyl}methylidene)hydrazine

Geraldo M. de Lima, William T. A. Harrison, Edward R. T. Tiekink, James L. Wardell and Solange M. S. V. Wardell

S1. Comment

The preparation of hydrazonederivatives of thiophenecarbaldehydes is well documented (Kwon, *et al.* 2009; Wardell *et al.*, 2007; Vaysse & Pastour, 1964). As a continuation of structural studies of this class of compound (Wardell *et al.*, 2007; Ferreira *et al.*, 2009; Nogueira *et al.*, 2010; Wardell *et al.*, 2010), the title compound (I) was synthesised and structurally investigated. 2-Substituted thiophenes in general have various uses, for example as dyestuffs, flavour agents, drugs, and inhibitors (Campaigne, 1984). Thiophenes are present in many natural and synthetic products with a wide range of pharmacological activities (Kleemann *et al.*, 2006; Sonar & Crooks, 2009; Mellado *et al.*, 2009; Satyanarayana *et al.*, 2008; Lourenço *et al.*, 2007). Specifically, hydrazone derivatives of thiophene have found uses in optoelectronic applications (Michaleviciute *et al.*, 2007), as optical non-linear materials (Kwon *et al.*, 2009), and as hole transporting materials (Michaleviciute *et al.*, 2009).

The overall molecular conformation in (I) is U-shaped as the two aromatic residues lie to the same side of the molecule and are syn, being orientated in the same direction as the thiophene-S atom, Fig. 1. Twists from planarity in the molecule are evident in each of the side-arms, i.e. about the N2–C6 and N4–N5 bonds; the N1/N2/C6/C7 and C12/N4/N5/C13 torsion angles are 168.57 (18) and -170.79 (19) °, respectively. In addition, the values of the C7/C8/N3/O1 and C14/C14/N6/O3 torsion angles of -161.0 (2) and 164.4 (2) °, respectively, indicate each of the nitro groups is twisted out of the plane of the benzene ring to which it is bonded. The conformation about each of the C5═N1 [1.288 (3) Å] and C12═N4 [1.285 (3) Å] double bonds is *E*.

In the crystal packing, centrosymmetrically related molecules associate via *N–H*···O_{nitro} hydrogen bonds to result in the formation of a 14-membered $\{\cdots HNC_3NO\}_2$ synthon, Table 1. The dimeric aggregates are linked into a supramolecular chain along the *c* axis via *C–H*···O_{nitro} interactions, Table 1 and Fig. 2. The chains in turn are linked into layers in the *ac* plane via further *C–H*···O_{nitro} interactions, Table 1. The layers thus formed stack along the *b* axis with the primary interactions between them being of the type C–H···*π* where the *π*-system is the thiophene ring [C12–H···ring centroid(S1,C1–C4)^i = 2.58 Å, C12···ring centroid^i = 3.323 (2) Å with an angle subtended at H = 135 ° for symmetry operation *i*: *x*, 3/2-*y*, -1/2+*z*]. The second N5-amine H does not participate in a formal hydrogen bond. It is noted that the N4–N5—H residues lie in the interlayer region and are in relative close proximity [e.g. N5–H···N4^{ii} = 2.87 Å] but steric constraints preclude a closer approach of these groups to allow a hydrogen bonding interaction.

S2. Experimental

Solutions of 3-nitrophenylhydrazine.hydrochloride (0.38 g, 2 mmol) in EtOH (10 ml) and 2,5-thiophenedicarbaldehyde (0.14 g, 1 mmol) in EtOH (10 ml) were mixed. The reaction mixture was refluxed for 1 h, and rotary evaporated. The solid residue was recrystallised twice from aq. EtOH (v:v 1:2), m.p. 497-479 K. ^{1}H NMR (400 MHz, DMSO-d$_6$): δ 7.26 (s,2*H*), 7.41 (dd, 2H, J = 1 & 8.1 Hz), 7.51 (t,2*H*, J = 8.0 Hz), 7.59 (dd, 2H, J = 1.3 & 8.0 Hz), 7.79 (t, 2H, 2.0 Hz), 8.10 (s, 2H), 10.91(s, 2H) p.p.m. ^{13}C (100 MHz, DMSO-d$_6$): δ 105.7, 113.10, 118.2, 128.5, 130.5, 134.3, 140.1, 145.9, 148.8 p.p.m.

S3. Refinement

The C-bound H atoms were geometrically placed (C–H = 0.95 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$. The N-bound H atoms were located from a difference map and refined with $U_{iso}(H) = 1.5U_{eq}(N)$.

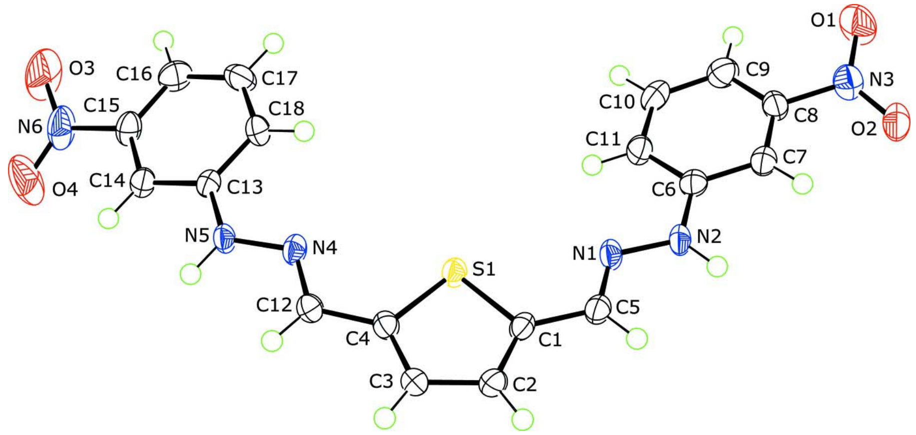

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 70% probability level.

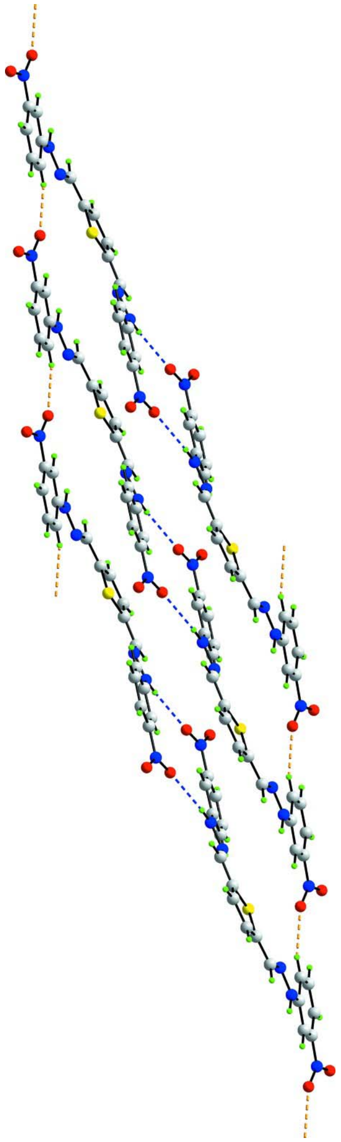

Figure 2

A view of the supramolecular chain in (I) whereby centrosymmetrically related dimeric aggregates, held together by *N*–*H*···O_{nitro} hydrogen bonds (blue dashed bonds), are linked via *C*–*H*···O_{nitro} interactions (orange dashed lines). Colour code: S, yellow; O, red; N, blue; C, grey; and H, green.

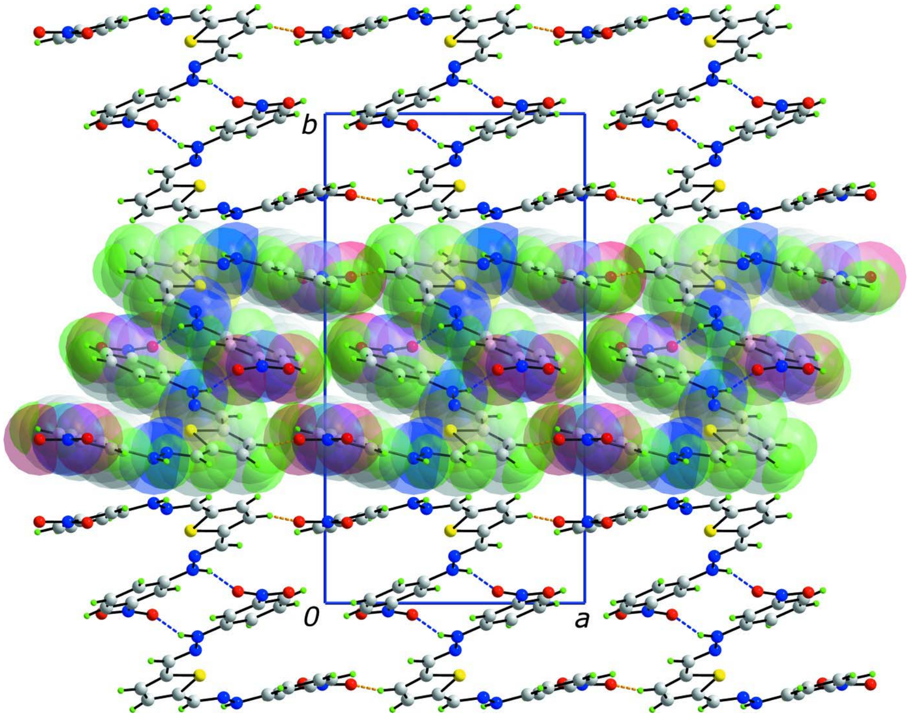

Figure 3

View of the stacking of layers in (I) with the central layer highlighted by a superimposed space filling representation. The N–H···O and C–H···O interactions are shown as blue and orange dashed lines, respectively. Colour code: S, yellow; O, red; N, blue; C, grey; and H, green.

(*E*)-1-(3-Nitrophenyl)-2-({5-[(1*E*)-2-(3-nitrophenyl)hydrazin-1- ylidenemethyl]-2-thienyl}methylidene)hydrazine

Crystal data

$C_{18}H_{14}N_6O_4S$
M_r = 410.41
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.1790 (5) Å
b = 20.6993 (9) Å
c = 8.0334 (2) Å
β = 100.513 (2)°
V = 1827.70 (12) Å^3
Z = 4

F(000) = 848
D_x = 1.492 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 13699 reflections
θ = 2.9–27.5°
μ = 0.22 mm^{-1}
T = 120 K
Rod, red
0.62 × 0.10 × 0.06 mm

Data collection

KappaCCD area-detector diffractometer
Radiation source: Enraf Nonius FR591 rotating anode
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)

$T_{\min}$ = 0.668, $T_{\max}$ = 0.746
21780 measured reflections
4183 independent reflections
3001 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
$\theta_{\max}$ = 27.5°, $\theta_{\min}$ = 3.1°
$h = -14 \rightarrow 14$
$k = -26 \rightarrow 26$
$l = -10 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.133$
$S = 1.08$
4183 reflections
268 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 0.3581P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor wR and goodness 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 threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) 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	0.51694 (5)	0.64771 (3)	0.10731 (6)	0.01767 (15)
O1	0.86026 (16)	0.47833 (10)	1.1894 (2)	0.0440 (5)
O2	0.66466 (16)	0.47631 (9)	1.16581 (19)	0.0344 (4)
O3	1.09960 (18)	0.66615 (11)	−0.6358 (3)	0.0553 (6)
O4	0.91528 (19)	0.66409 (11)	−0.7736 (2)	0.0519 (6)
N1	0.50139 (16)	0.59578 (9)	0.4425 (2)	0.0182 (4)
N2	0.50940 (17)	0.56838 (9)	0.5969 (2)	0.0191 (4)
H2N	0.446 (2)	0.5655 (12)	0.649 (3)	0.029*
N3	0.75746 (18)	0.48377 (10)	1.1061 (2)	0.0267 (5)
N4	0.60955 (16)	0.69295 (9)	−0.1986 (2)	0.0181 (4)
N5	0.65934 (16)	0.70405 (9)	−0.3383 (2)	0.0184 (4)
H5N	0.611 (2)	0.7106 (11)	−0.442 (3)	0.028*
N6	0.9889 (2)	0.66605 (11)	−0.6418 (3)	0.0340 (5)
C1	0.38972 (19)	0.64722 (10)	0.2032 (2)	0.0171 (4)
C2	0.29149 (19)	0.67487 (10)	0.1014 (3)	0.0194 (5)
H2	0.2137	0.6787	0.1322	0.023*
C3	0.31778 (19)	0.69709 (10)	−0.0543 (2)	0.0186 (4)
H3	0.2596	0.7175	−0.1389	0.022*
C4	0.43575 (19)	0.68609 (10)	−0.0706 (2)	0.0173 (4)
C5	0.3983 (2)	0.61919 (10)	0.3698 (2)	0.0189 (5)
H5	0.3297	0.6181	0.4242	0.023*
C6	0.6201 (2)	0.54271 (10)	0.6765 (3)	0.0184 (5)
C7	0.6332 (2)	0.52533 (10)	0.8470 (3)	0.0189 (5)
H7	0.5680	0.5303	0.9069	0.023*

C8	0.7441 (2)	0.50061 (11)	0.9255 (2)	0.0208 (5)
C9	0.8416 (2)	0.49104 (11)	0.8444 (3)	0.0245 (5)
H9	0.9165	0.4740	0.9028	0.029*
C10	0.8256 (2)	0.50743 (11)	0.6740 (3)	0.0240 (5)
H10	0.8903	0.5008	0.6140	0.029*
C11	0.7166 (2)	0.53338 (10)	0.5900 (3)	0.0216 (5)
H11	0.7076	0.5448	0.4738	0.026*
C12	0.49410 (19)	0.70092 (10)	−0.2128 (2)	0.0174 (4)
H12	0.4474	0.7164	−0.3159	0.021*
C13	0.77989 (19)	0.68714 (10)	−0.3355 (2)	0.0162 (4)
C14	0.8226 (2)	0.68469 (10)	−0.4875 (3)	0.0193 (5)
H14	0.7703	0.6936	−0.5921	0.023*
C15	0.9433 (2)	0.66899 (11)	−0.4818 (3)	0.0223 (5)
C16	1.0241 (2)	0.65534 (12)	−0.3337 (3)	0.0264 (5)
H16	1.1069	0.6452	−0.3345	0.032*
C17	0.9786 (2)	0.65709 (11)	−0.1840 (3)	0.0253 (5)
H17	1.0311	0.6473	−0.0802	0.030*
C18	0.8583 (2)	0.67280 (10)	−0.1833 (3)	0.0200 (5)
H18	0.8289	0.6738	−0.0796	0.024*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0173 (3)	0.0206 (3)	0.0161 (3)	0.0015 (2)	0.00585 (19)	0.0015 (2)
O1	0.0278 (11)	0.0719 (15)	0.0297 (10)	0.0132 (10)	−0.0019 (8)	0.0147 (9)
O2	0.0306 (10)	0.0526 (12)	0.0217 (8)	0.0002 (8)	0.0090 (7)	0.0075 (7)
O3	0.0333 (12)	0.0913 (18)	0.0497 (12)	0.0191 (11)	0.0300 (10)	0.0196 (11)
O4	0.0481 (13)	0.0905 (17)	0.0198 (10)	0.0207 (11)	0.0136 (9)	0.0044 (9)
N1	0.0241 (10)	0.0195 (10)	0.0122 (8)	−0.0030 (8)	0.0065 (7)	−0.0013 (7)
N2	0.0215 (10)	0.0240 (10)	0.0132 (8)	−0.0002 (8)	0.0069 (7)	0.0020 (7)
N3	0.0290 (12)	0.0286 (11)	0.0230 (10)	0.0034 (9)	0.0061 (9)	0.0030 (8)
N4	0.0212 (10)	0.0197 (10)	0.0152 (9)	−0.0012 (8)	0.0080 (7)	−0.0006 (7)
N5	0.0165 (9)	0.0272 (10)	0.0126 (9)	0.0029 (8)	0.0052 (7)	0.0040 (7)
N6	0.0326 (13)	0.0449 (14)	0.0297 (12)	0.0129 (10)	0.0192 (10)	0.0116 (9)
C1	0.0188 (11)	0.0167 (11)	0.0171 (10)	−0.0050 (9)	0.0070 (8)	−0.0024 (8)
C2	0.0161 (11)	0.0241 (12)	0.0186 (11)	−0.0013 (9)	0.0051 (8)	−0.0013 (8)
C3	0.0172 (11)	0.0227 (11)	0.0154 (10)	−0.0010 (9)	0.0020 (8)	0.0006 (8)
C4	0.0204 (11)	0.0164 (11)	0.0148 (10)	−0.0018 (9)	0.0025 (8)	−0.0001 (8)
C5	0.0208 (11)	0.0198 (11)	0.0174 (10)	−0.0039 (9)	0.0068 (8)	−0.0021 (8)
C6	0.0212 (12)	0.0151 (11)	0.0190 (10)	−0.0044 (9)	0.0042 (8)	−0.0020 (8)
C7	0.0219 (12)	0.0176 (11)	0.0181 (10)	−0.0018 (9)	0.0063 (8)	−0.0009 (8)
C8	0.0258 (13)	0.0207 (12)	0.0156 (10)	−0.0034 (9)	0.0032 (9)	0.0009 (8)
C9	0.0202 (12)	0.0260 (13)	0.0274 (12)	0.0003 (10)	0.0049 (9)	0.0012 (9)
C10	0.0211 (12)	0.0261 (13)	0.0274 (12)	−0.0029 (10)	0.0112 (9)	0.0001 (9)
C11	0.0245 (12)	0.0225 (12)	0.0186 (11)	−0.0053 (9)	0.0059 (9)	0.0009 (8)
C12	0.0190 (11)	0.0183 (11)	0.0150 (10)	0.0006 (9)	0.0033 (8)	−0.0009 (8)
C13	0.0173 (11)	0.0163 (11)	0.0162 (10)	−0.0012 (8)	0.0061 (8)	0.0001 (8)
C14	0.0200 (12)	0.0201 (11)	0.0186 (10)	0.0004 (9)	0.0060 (8)	0.0031 (8)

C15	0.0237 (12)	0.0255 (12)	0.0207 (11)	0.0030 (9)	0.0116 (9)	0.0019 (8)
C16	0.0149 (11)	0.0340 (14)	0.0313 (12)	0.0040 (10)	0.0070 (9)	0.0018 (10)
C17	0.0193 (12)	0.0349 (14)	0.0196 (11)	0.0007 (10)	−0.0016 (9)	−0.0011 (9)
C18	0.0234 (12)	0.0236 (12)	0.0137 (10)	−0.0002 (9)	0.0050 (8)	−0.0002 (8)

Geometric parameters (Å, º)

S1—C1	1.737 (2)	C5—H5	0.9500
S1—C4	1.739 (2)	C6—C11	1.399 (3)
O1—N3	1.225 (3)	C6—C7	1.398 (3)
O2—N3	1.229 (2)	C7—C8	1.382 (3)
O3—N6	1.230 (3)	C7—H7	0.9500
O4—N6	1.217 (3)	C8—C9	1.382 (3)
N1—C5	1.288 (3)	C9—C10	1.390 (3)
N1—N2	1.352 (2)	C9—H9	0.9500
N2—C6	1.391 (3)	C10—C11	1.389 (3)
N2—H2N	0.89 (3)	C10—H10	0.9500
N3—C8	1.473 (3)	C11—H11	0.9500
N4—C12	1.285 (3)	C12—H12	0.9500
N4—N5	1.360 (2)	C13—C14	1.391 (3)
N5—C13	1.389 (3)	C13—C18	1.400 (3)
N5—H5N	0.92 (2)	C14—C15	1.380 (3)
N6—C15	1.468 (3)	C14—H14	0.9500
C1—C2	1.369 (3)	C15—C16	1.385 (3)
C1—C5	1.445 (3)	C16—C17	1.388 (3)
C2—C3	1.413 (3)	C16—H16	0.9500
C2—H2	0.9500	C17—C18	1.385 (3)
C3—C4	1.368 (3)	C17—H17	0.9500
C3—H3	0.9500	C18—H18	0.9500
C4—C12	1.449 (3)		

C1—S1—C4	91.17 (10)	C6—C7—H7	121.1
C5—N1—N2	118.56 (18)	C9—C8—C7	123.82 (19)
N1—N2—C6	119.09 (18)	C9—C8—N3	118.9 (2)
N1—N2—H2N	122.5 (16)	C7—C8—N3	117.29 (19)
C6—N2—H2N	118.4 (16)	C8—C9—C10	117.3 (2)
O1—N3—O2	123.37 (19)	C8—C9—H9	121.4
O1—N3—C8	118.43 (19)	C10—C9—H9	121.4
O2—N3—C8	118.20 (19)	C9—C10—C11	121.2 (2)
C12—N4—N5	117.41 (17)	C9—C10—H10	119.4
N4—N5—C13	119.06 (17)	C11—C10—H10	119.4
N4—N5—H5N	121.3 (16)	C10—C11—C6	119.9 (2)
C13—N5—H5N	117.1 (16)	C10—C11—H11	120.0
O4—N6—O3	123.4 (2)	C6—C11—H11	120.0
O4—N6—C15	118.4 (2)	N4—C12—C4	119.47 (18)
O3—N6—C15	118.2 (2)	N4—C12—H12	120.3
C2—C1—C5	129.17 (19)	C4—C12—H12	120.3
C2—C1—S1	111.39 (15)	N5—C13—C14	118.84 (18)

C5—C1—S1	119.43 (16)	N5—C13—C18	121.25 (18)
C1—C2—C3	113.04 (19)	C14—C13—C18	119.9 (2)
C1—C2—H2	123.5	C15—C14—C13	118.01 (19)
C3—C2—H2	123.5	C15—C14—H14	121.0
C4—C3—C2	112.95 (19)	C13—C14—H14	121.0
C4—C3—H3	123.5	C14—C15—C16	123.8 (2)
C2—C3—H3	123.5	C14—C15—N6	118.30 (19)
C3—C4—C12	128.36 (19)	C16—C15—N6	117.9 (2)
C3—C4—S1	111.45 (15)	C15—C16—C17	117.0 (2)
C12—C4—S1	120.17 (16)	C15—C16—H16	121.5
N1—C5—C1	118.30 (19)	C17—C16—H16	121.5
N1—C5—H5	120.9	C18—C17—C16	121.3 (2)
C1—C5—H5	120.9	C18—C17—H17	119.4
N2—C6—C11	121.80 (19)	C16—C17—H17	119.4
N2—C6—C7	118.27 (19)	C17—C18—C13	119.98 (19)
C11—C6—C7	119.9 (2)	C17—C18—H18	120.0
C8—C7—C6	117.9 (2)	C13—C18—H18	120.0
C8—C7—H7	121.1		

C5—N1—N2—C6	179.76 (19)	N3—C8—C9—C10	179.9 (2)
C12—N4—N5—C13	−170.79 (19)	C8—C9—C10—C11	1.1 (3)
C4—S1—C1—C2	0.37 (17)	C9—C10—C11—C6	−0.8 (3)
C4—S1—C1—C5	179.95 (17)	N2—C6—C11—C10	−179.8 (2)
C5—C1—C2—C3	−179.9 (2)	C7—C6—C11—C10	−0.6 (3)
S1—C1—C2—C3	−0.4 (2)	N5—N4—C12—C4	176.44 (18)
C1—C2—C3—C4	0.2 (3)	C3—C4—C12—N4	173.0 (2)
C2—C3—C4—C12	178.6 (2)	S1—C4—C12—N4	−8.6 (3)
C2—C3—C4—S1	0.1 (2)	N4—N5—C13—C14	165.61 (18)
C1—S1—C4—C3	−0.26 (17)	N4—N5—C13—C18	−14.5 (3)
C1—S1—C4—C12	−178.90 (17)	N5—C13—C14—C15	178.8 (2)
N2—N1—C5—C1	−178.75 (17)	C18—C13—C14—C15	−1.1 (3)
C2—C1—C5—N1	−180.0 (2)	C13—C14—C15—C16	0.2 (3)
S1—C1—C5—N1	0.5 (3)	C13—C14—C15—N6	179.7 (2)
N1—N2—C6—C11	−12.2 (3)	O4—N6—C15—C14	−15.1 (3)
N1—N2—C6—C7	168.57 (18)	O3—N6—C15—C14	164.4 (2)
N2—C6—C7—C8	−179.20 (19)	O4—N6—C15—C16	164.5 (2)
C11—C6—C7—C8	1.5 (3)	O3—N6—C15—C16	−16.0 (3)
C6—C7—C8—C9	−1.2 (3)	C14—C15—C16—C17	0.8 (4)
C6—C7—C8—N3	178.75 (18)	N6—C15—C16—C17	−178.7 (2)
O1—N3—C8—C9	19.0 (3)	C15—C16—C17—C18	−1.0 (4)
O2—N3—C8—C9	−161.5 (2)	C16—C17—C18—C13	0.1 (3)
O1—N3—C8—C7	−161.0 (2)	N5—C13—C18—C17	−178.9 (2)
O2—N3—C8—C7	18.6 (3)	C14—C13—C18—C17	0.9 (3)
C7—C8—C9—C10	−0.1 (3)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the S1,C1–C4 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2n···O2^{i}	0.89 (2)	2.27 (2)	3.103 (2)	156 (2)
C2—H2···O3^{ii}	0.95	2.46	3.278 (3)	145
C18—H18···O4^{iii}	0.95	2.48	3.241 (3)	137
C12—H12···*Cg*1^{iv}	0.95	2.58	3.323 (2)	135

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) *x*−1, *y*, *z*+1; (iii) *x*, *y*, *z*+1; (iv) *x*, −*y*+3/2, *z*−1/2.