Acta Cryst E
CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure and Hirshfeld surface analysis of 2-methyl-3-nitro-*N*-[(*E*)-(5-nitrothiophen-2-yl)-methylidene]aniline

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Received 28 December 2020
Accepted 13 January 2021

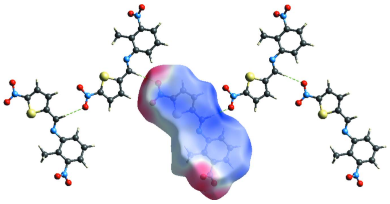
Edited by A. V. Yatsenko, Moscow State University, Russia

Keywords: crystal structure; thiophene; 5-nitrothiophen-2-yl; Schiff base; Hirshfeld surface analysis; hydrogen bonding.

CCDC reference: 2055920

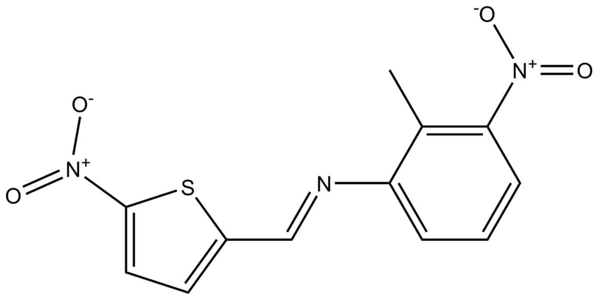
Supporting information: this article has supporting information at journals.iucr.org/e

The title compound, $C_{12}H_9N_3O_4S$, synthesized by condensation of 5-nitrothiophene-2-carbaldehyde and 2-methyl-3-nitroaniline, crystallizes in the orthorhombic space group $P2_12_12_1$. In the molecule, the aromatic benzene and thiophene rings are twisted with respect to each other, making a dihedral angle of 23.16 (7)°. In the crystal, molecules are linked by intermolecular C—H⋯O hydrogen bonds into chains extending along the *c*-axis direction. Weak π–π stacking interactions along the *a*-axis direction provide additional stabilization of the crystal structure. The roles of the various intermolecular interactions were clarified by Hirshfeld surface analysis, which reveals that the crystal packing is dominated by O⋯H (39%) and H⋯H (21.3%) contacts. The crystal studied was refined as a two-component inversion twin.



1. Chemical context

Bioactivity is an important topic, which includes many areas such as the synthesis of new drugs, creams, agricultural products and so on. In this respect, Schiff bases are organic molecules suitable for bioactivity applications because of the imine bond that increases the lipophilic character of the molecule. The imine bond provides a synthetic route to structural chirality, changes the electronic properties and leads to solubility in different media (Tarafder *et al.*, 2008). Schiff bases can include heterocycles or amino acid residues and can be easily obtained by the condensation of primary amines with aldehydes or ketones without by-products, thus giving the pure product for biological treatments (Yu *et al.*, 2009; Lobana *et al.*, 2009). Many natural products contain thiophene groups, which lead to pharmacological properties. Thiophene-containing molecules are used in medicinal chemistry for therapeutic applications (Mishra *et al.*, 2011). 5-Nitrothiophene-2-carboxaldehyde derivatives exhibit antibacterial properties (Foroumadi *et al.*, 2003). This highly reactive molecule has been used in chemosensor applications (Ye *et al.*, 2019). In the present study, a new Schiff base, 2-methyl-3-nitro-*N*-[(*E*)-(5-nitrothiophen-2-yl)methylidene]aniline (I), was obtained in crystalline form from the reaction of 5-nitrothiophene-2-carbaldehyde with 2-methyl-3-nitroaniline.



2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The molecule adopts the *E* configuration with respect to the C═N bond and the benzene and thiophene rings form a dihedral angle of 23.16 (7)°. The deviation from planarity can be attributed to packing forces. The nitro group attached to the thiophene ring is strongly conjugated with the π-system of this ring, as evident from the short N2—C7 distance (see Table 1). As a result, this nitro group is almost coplanar with the thiophene ring. The nitro group attached to the benzene ring is twisted by 48.4 (2)° with respect to this ring, and thus the π-conjugation is much weaker in this case. The length of the C8═N2 bond is 1.277 (4) Å, which is consistent with those in the related structures 4-(naphthalen-2-yl)-*N*-[(*Z*)-4-propoxybenzylidene]-1,3-thiazol-2-amine [1.284 (3) Å; Sheakh Mohamad *et al.*, 2020] and (*E*)-2,4-di-tert-butyl-6-[(3-chloro-4-methylphenylimino)methyl]phenol [1.278 (4) Å; Kansiz *et al.*, 2018]. The C9—S1 and C12—S1 bonds in the thiophene ring are slightly shorter than a standard Csp^2—S single bond (1.76 Å; Allen *et al.*, 1987) as a result of the π-conjugation with the double bonds. At the same time, these S—C bonds are longer than those in the structure of 6-[(*E*)-2-(thiophen-2-yl)ethenyl]-4,5-dihydropyridazin-3(2*H*)-one [1.691 (3) Å; Daoui *et al.*, 2019].

Table 1
Selected bond lengths (Å).

S1—C12	1.714 (4)	N1—O2	1.218 (5)
S1—C9	1.718 (4)	N1—C3	1.474 (5)
N2—C8	1.277 (4)	N3—O3	1.216 (6)
N2—C7	1.411 (4)	N3—O4	1.230 (6)
N1—O1	1.211 (5)	N3—C12	1.423 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O4^{i}	0.93 (4)	2.56 (4)	3.492 (5)	176 (3)

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

3. Supramolecular features

In the crystal structure, molecules are connected by weak intermolecular C8—H8···O4^{i} hydrogen bonds into chains stretched along the *c*-axis direction (Table 2; Fig. 2). As a result, the molecules form stacks extended along the *a*-axis direction. The shortest intercentroid separation of 3.603 (2) Å within the stack indicates π–π stacking interactions between the benzene and thiophene rings, which are, however, very weak, since intermolecular contacts shorter than the sum of van der Waals radii are absent from these stacks.

4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.41, update of November 2019; Groom *et al.*, 2016) for (*E*)-*N*-[(5-nitrothiophen-2-yl)methylene]aniline gave 15 hits including 4-methyl-*N*-[(5-nitrothiophen-2-yl)methylidene]-aniline (EXIWIS; Cai *et al.*, 2011), *N*-(2-chlorophenyl)-1-(5-nitrothiophen-2-yl)methanimine (FIBKUZ; Tari *et al.*, 2018) and 1-(5-nitro-2-thienyl)-*N*-(2-phenoxyphenyl)methanimine (TONBAB; Tanak *et al.*, 2014). In FIBKUZ and TONBAB, intermolecular C—H···O hydrogen bonds are important features in the crystal packing, as in the structure of the title compound. In EXIWIS, the C═N bond length [1.277 (2) Å] is the same as in the title compound and longer than in both FIBKUZ [1.265 (6) Å] and in TONBAB [1.261 (4) Å]. The N—O bond lengths in the nitro groups in the title compound are the same within standard deviations as the corresponding

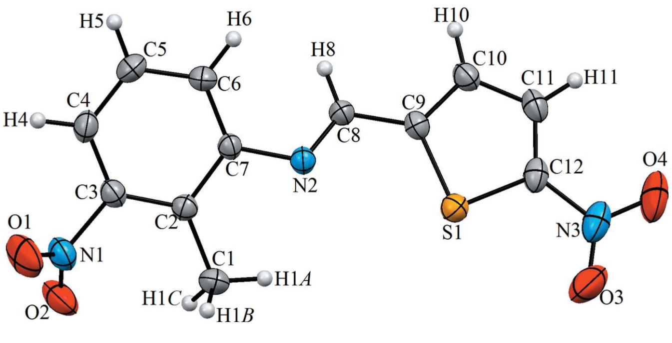


Figure 1
The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

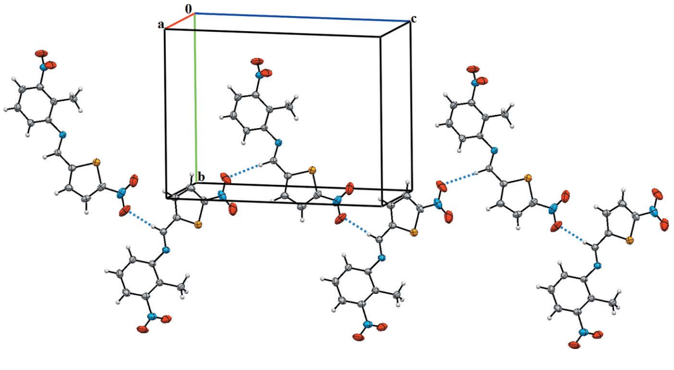


Figure 2
A view of the crystal packing of the title compound parallel to the *bc* plane. C—H···O interactions are indicated by dotted lines.

bond lengths in all of the reference structures. The C—S bond lengths in EXIWIS, FIBKUZ and TONBAB range from 1.694 (3) to 1.730 (2) Å. The corresponding bond lengths in the title compound fall within these limits.

5. Hirshfeld surface analysis

The Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) was carried out using the *CrystalExplorer17.5* (Turner *et al.*, 2017). The Hirshfeld surface and the associated two-dimensional fingerprint plots were used to quantify the various intermolecular interactions in the title compound. The Hirshfeld surfaces mapped over $d_{\rm norm}$ and electrostatic potential are illustrated in Fig. 3. In Fig. 3*a*, the red spots correspond to the O···H contacts. The electrostatic potential (Fig. 3*b*) shows donor (red) and acceptor (blue) regions. The percentage contribution of various interactions is shown in the fingerprint plot (Fig. 4). The most important interactions for determining the morphology of the crystal are H···H, O···H and S···H contacts, their individual contributions being 39%, 21.3% and 5.9%, respectively. C···N/N···C (5.8%) and C···H/H···C (5.4%) contacts are also observed. The Hirshfeld surface analysis confirms the importance of H-atom contacts in establishing the crystal packing.

6. Synthesis and crystallization

The title compound was prepared by refluxing a solution containing 5-nitrothiophene-2-carbaldehyde (0,07 mmol) and 2-methyl-3-nitroaniline (0,07 mmol) in ethanol (40 ml) for 5 h under stirring. The obtained yellow crystalline material was washed with ethanol and dried at room temperature (yield: 78%, m.p. 433 K). Crystals were grown from a solution in ethanol.

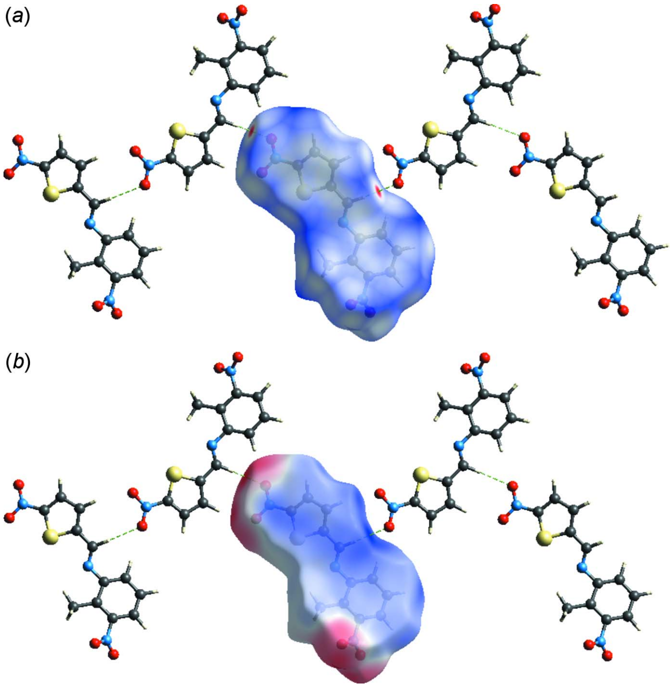


Figure 3
Hirshfeld surfaces of the title compound mapped over (*a*) d_{norm} and (*b*) electrostatic potential.

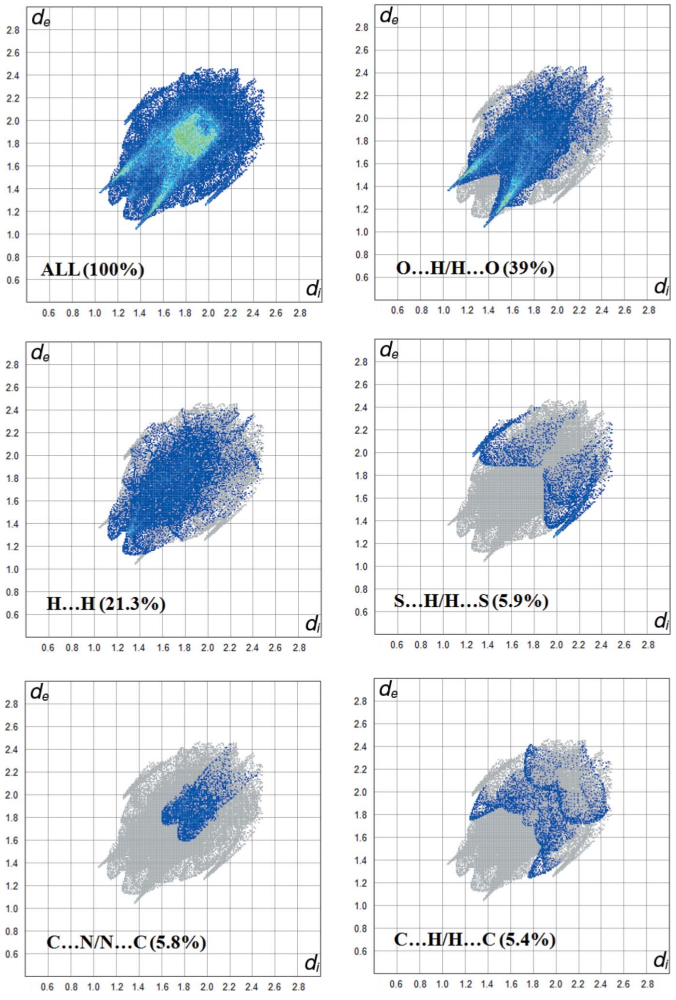


Figure 4
Two-dimensional fingerprint plots for the title compound, with the relative contributions of the atom pairs to the Hirshfeld surface.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The C-bound H atoms were placed in idealized positions and refined using a riding model with C—H = 0.93–0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C-methyl) and 1.2$U_{\rm eq}$(C) for other C-bound H atoms. The structure was refined as a two-component inversion twin.

Funding information

This study was supported by Ondokuz Mayıs University under Project No. PYO·FEN.1906.19.001.

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Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_9N_3O_4S$
M_r	291.28
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	293
a, b, c (Å)	7.1335 (4), 11.7297 (6), 15.4593 (7)
V (Å^3)	1293.54 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.27
Crystal size (mm)	0.75 × 0.39 × 0.14
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.839, 0.966
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6707, 3930, 2258
R_{int}	0.049
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.714
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.053, 0.129, 0.91
No. of reflections	3930
No. of parameters	186
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.19
Absolute structure	Refined as an inversion twin.
Absolute structure parameter	0.59 (15)

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXT2017/1* (Sheldrick, 2015*a*), *SHELXL2017/1* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020) and *WinGX* (Farrugia, 2012).

supporting information

Acta Cryst. (2021). E**77**, 138-141   [https://doi.org/10.1107/S2056989021000529]

Crystal structure and Hirshfeld surface analysis of 2-methyl-3-nitro-*N*-[(*E*)-(5-nitrothiophen-2-yl)methylidene]aniline

Sevgi Kansiz, Necmi Dege, Seyhan Ozturk, Nesuhi Akdemir, Erdoğan Tarcan, Ali Arslanhan and Eiad Saif

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*(Stoe & Cie, 2002); data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXT2017/1* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2017/1* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

2-Methyl-3-nitro-*N*-[(*E*)-(5-nitrothiophen-2-yl)methylidene]aniline

Crystal data

$C_{12}H_9N_3O_4S$
$M_r = 291.28$
Orthorhombic, $P2_12_12_1$
$a = 7.1335\ (4)$ Å
$b = 11.7297\ (6)$ Å
$c = 15.4593\ (7)$ Å
$V = 1293.54\ (11)$ Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.496$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6261 reflections
$\theta = 2.2–30.9°$
$\mu = 0.27$ mm^{-1}
$T = 293$ K
Stick, yellow
0.75 × 0.39 × 0.14 mm

Data collection

Stoe IPDS 2 diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)
$T_{\min} = 0.839$, $T_{\max} = 0.966$
6707 measured reflections
3930 independent reflections
2258 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.049$
$\theta_{\max} = 30.5°$, $\theta_{\min} = 2.2°$
$h = -8 \rightarrow 10$
$k = -16 \rightarrow 12$
$l = -22 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.129$
$S = 0.91$
3930 reflections
186 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.063P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Absolute structure: Refined as an inversion twin.

Absolute structure parameter: 0.59 (15)

Special details

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

Refinement. Refined as a two-component inversion twin

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.63801 (15)	0.83820 (8)	0.62698 (6)	0.0504 (3)
N2	0.6540 (5)	0.6808 (2)	0.47460 (18)	0.0437 (7)
N1	0.7061 (6)	0.2794 (3)	0.3994 (3)	0.0637 (10)
O2	0.8371 (6)	0.2537 (3)	0.4460 (2)	0.0886 (11)
O1	0.5937 (6)	0.2119 (3)	0.3708 (3)	0.0988 (13)
C8	0.7015 (6)	0.7831 (3)	0.4567 (2)	0.0432 (8)
C7	0.6589 (5)	0.5963 (3)	0.4097 (2)	0.0397 (7)
C9	0.6899 (5)	0.8708 (3)	0.5213 (2)	0.0431 (8)
C6	0.6287 (6)	0.6186 (3)	0.3229 (2)	0.0487 (9)
H6	0.610040	0.693392	0.304891	0.058*
C3	0.6828 (5)	0.4000 (3)	0.3749 (3)	0.0474 (8)
N3	0.6315 (7)	1.0145 (4)	0.7400 (3)	0.0756 (12)
C2	0.6860 (5)	0.4834 (3)	0.4389 (2)	0.0423 (8)
C4	0.6524 (7)	0.4210 (3)	0.2888 (2)	0.0549 (9)
H4	0.649811	0.361601	0.249035	0.066*
O3	0.5983 (7)	0.9420 (5)	0.7941 (2)	0.1054 (15)
C12	0.6570 (6)	0.9799 (3)	0.6526 (2)	0.0537 (10)
C11	0.7002 (6)	1.0482 (3)	0.5853 (3)	0.0575 (11)
H11	0.714508	1.126873	0.589073	0.069*
O4	0.6437 (7)	1.1168 (4)	0.7564 (3)	0.1149 (15)
C10	0.7204 (6)	0.9856 (3)	0.5095 (3)	0.0520 (10)
H10	0.751275	1.017854	0.456431	0.062*
C1	0.7131 (7)	0.4585 (4)	0.5333 (3)	0.0559 (10)
H1A	0.710488	0.528510	0.565450	0.084*
H1B	0.614362	0.409413	0.553194	0.084*
H1C	0.831809	0.421538	0.541751	0.084*
C5	0.6258 (7)	0.5321 (4)	0.2626 (2)	0.0573 (10)
H5	0.606041	0.548561	0.204434	0.069*
H8	0.747 (5)	0.807 (3)	0.403 (2)	0.034 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0561 (5)	0.0487 (5)	0.0465 (4)	−0.0019 (5)	−0.0005 (5)	−0.0003 (4)
N2	0.0474 (16)	0.0390 (15)	0.0448 (15)	−0.0002 (14)	0.0051 (14)	−0.0033 (12)
N1	0.075 (3)	0.0406 (18)	0.075 (3)	0.0010 (19)	0.002 (2)	−0.0039 (17)

O2	0.110 (3)	0.0465 (17)	0.109 (3)	0.012 (2)	−0.021 (3)	0.0103 (18)
O1	0.120 (3)	0.0470 (17)	0.130 (3)	−0.0226 (19)	−0.015 (3)	−0.009 (2)
C8	0.052 (2)	0.0367 (18)	0.0407 (18)	0.0013 (15)	0.0051 (16)	0.0002 (14)
C7	0.0433 (19)	0.0334 (15)	0.0424 (17)	−0.0017 (16)	0.0050 (16)	−0.0029 (13)
C9	0.042 (2)	0.0383 (17)	0.0490 (19)	0.0033 (14)	−0.0003 (15)	−0.0014 (15)
C6	0.062 (2)	0.0445 (18)	0.0400 (18)	0.007 (2)	−0.0010 (19)	0.0059 (14)
C3	0.050 (2)	0.0368 (17)	0.055 (2)	0.0010 (15)	0.0040 (19)	0.0005 (17)
N3	0.070 (2)	0.093 (3)	0.063 (2)	0.006 (3)	−0.006 (2)	−0.032 (2)
C2	0.045 (2)	0.0399 (18)	0.0423 (18)	−0.0028 (15)	0.0037 (15)	−0.0006 (15)
C4	0.064 (3)	0.050 (2)	0.050 (2)	0.002 (2)	0.005 (2)	−0.0148 (17)
O3	0.131 (4)	0.134 (4)	0.052 (2)	0.012 (3)	0.006 (2)	−0.010 (2)
C12	0.050 (2)	0.056 (2)	0.055 (2)	0.004 (2)	−0.0073 (19)	−0.0194 (18)
C11	0.064 (3)	0.038 (2)	0.070 (3)	0.0038 (18)	−0.004 (2)	−0.0101 (19)
O4	0.141 (4)	0.101 (3)	0.102 (3)	0.004 (3)	−0.007 (3)	−0.063 (3)
C10	0.061 (2)	0.0366 (19)	0.058 (2)	−0.0001 (17)	−0.0024 (19)	0.0013 (17)
C1	0.073 (3)	0.046 (2)	0.048 (2)	0.000 (2)	0.002 (2)	0.0085 (17)
C5	0.073 (3)	0.063 (2)	0.0361 (18)	0.002 (3)	−0.001 (2)	−0.0028 (17)

Geometric parameters (Å, º)

S1—C12	1.714 (4)	C3—C2	1.392 (5)
S1—C9	1.718 (4)	N3—O3	1.216 (6)
N2—C8	1.277 (4)	N3—O4	1.230 (6)
N2—C7	1.411 (4)	N3—C12	1.423 (5)
N1—O1	1.211 (5)	C2—C1	1.501 (5)
N1—O2	1.218 (5)	C4—C5	1.377 (6)
N1—C3	1.474 (5)	C4—H4	0.9300
C8—C9	1.435 (5)	C12—C11	1.349 (6)
C8—H8	0.93 (4)	C11—C10	1.391 (6)
C7—C6	1.384 (5)	C11—H11	0.9300
C7—C2	1.412 (5)	C10—H10	0.9300
C9—C10	1.376 (5)	C1—H1A	0.9600
C6—C5	1.378 (5)	C1—H1B	0.9600
C6—H6	0.9300	C1—H1C	0.9600
C3—C4	1.371 (5)	C5—H5	0.9300

C12—S1—C9	89.25 (18)	C3—C2—C1	123.8 (3)
C8—N2—C7	120.0 (3)	C7—C2—C1	120.8 (3)
O1—N1—O2	124.3 (4)	C3—C4—C5	118.6 (3)
O1—N1—C3	117.3 (4)	C3—C4—H4	120.7
O2—N1—C3	118.4 (4)	C5—C4—H4	120.7
N2—C8—C9	120.5 (3)	C11—C12—N3	126.4 (4)
N2—C8—H8	124 (2)	C11—C12—S1	114.6 (3)
C9—C8—H8	115 (2)	N3—C12—S1	119.0 (3)
C6—C7—N2	123.5 (3)	C12—C11—C10	111.1 (4)
C6—C7—C2	120.6 (3)	C12—C11—H11	124.5
N2—C7—C2	115.8 (3)	C10—C11—H11	124.5
C10—C9—C8	126.9 (4)	C9—C10—C11	112.9 (4)

C10—C9—S1	112.2 (3)	C9—C10—H10	123.5
C8—C9—S1	120.9 (3)	C11—C10—H10	123.6
C5—C6—C7	121.3 (3)	C2—C1—H1A	109.5
C5—C6—H6	119.4	C2—C1—H1B	109.5
C7—C6—H6	119.4	H1A—C1—H1B	109.5
C4—C3—C2	124.5 (3)	C2—C1—H1C	109.5
C4—C3—N1	116.1 (3)	H1A—C1—H1C	109.5
C2—C3—N1	119.4 (4)	H1B—C1—H1C	109.5
O3—N3—O4	123.7 (5)	C4—C5—C6	119.7 (4)
O3—N3—C12	118.6 (4)	C4—C5—H5	120.2
O4—N3—C12	117.7 (5)	C6—C5—H5	120.2
C3—C2—C7	115.4 (3)		

C7—N2—C8—C9	177.5 (3)	N2—C7—C2—C3	177.4 (3)
C8—N2—C7—C6	−30.4 (6)	C6—C7—C2—C1	−178.2 (4)
C8—N2—C7—C2	153.2 (4)	N2—C7—C2—C1	−1.7 (6)
N2—C8—C9—C10	−173.9 (4)	C2—C3—C4—C5	1.0 (7)
N2—C8—C9—S1	7.5 (5)	N1—C3—C4—C5	178.7 (4)
C12—S1—C9—C10	0.4 (3)	O3—N3—C12—C11	−177.8 (5)
C12—S1—C9—C8	179.2 (3)	O4—N3—C12—C11	2.5 (8)
N2—C7—C6—C5	−176.7 (4)	O3—N3—C12—S1	0.6 (7)
C2—C7—C6—C5	−0.5 (7)	O4—N3—C12—S1	−179.2 (4)
O1—N1—C3—C4	−47.0 (6)	C9—S1—C12—C11	0.0 (4)
O2—N1—C3—C4	132.2 (4)	C9—S1—C12—N3	−178.6 (4)
O1—N1—C3—C2	130.8 (4)	N3—C12—C11—C10	178.1 (4)
O2—N1—C3—C2	−50.0 (6)	S1—C12—C11—C10	−0.3 (5)
C4—C3—C2—C7	−1.2 (6)	C8—C9—C10—C11	−179.3 (4)
N1—C3—C2—C7	−178.8 (3)	S1—C9—C10—C11	−0.7 (4)
C4—C3—C2—C1	177.9 (4)	C12—C11—C10—C9	0.6 (5)
N1—C3—C2—C1	0.3 (6)	C3—C4—C5—C6	−0.5 (7)
C6—C7—C2—C3	0.9 (6)	C7—C6—C5—C4	0.3 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O4$^{\rm i}$	0.93 (4)	2.56 (4)	3.492 (5)	176 (3)

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