organic compounds

3-[(4-Phenoxyphenyl)sulfanyl]-5-phenyl-1H-1,2,4-triazole

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The title compound, $C_{20}H_{15}N_3OS$, is V-shaped. In the 4-phenoxyphenyl group, the two rings are inclined to one another by 74.52 (13)°. These rings are inclined to the triazole ring by 72.20 (15) and 72.30 (15)°, respectively. The phenyl ring is inclined to the triazole ring by 10.85 (12)°. In the crystal, molecules are linked via N—H···N hydrogen bonds, forming chains propagating along [010]. These chains are linked via pairs of C—H···S hydrogen bonds, forming sheets lying parallel to the ac plane.

Related literature

For the synthesis, properties and various biological activities of functionalized 1,2,4-triazole derivatives, see: Holla et al. (2002, 2003); Walczak et al. (2004); Zitouni et al. (2005); Prasad et al. (2009); Wael et al. (2012); Almasirad et al. (2004); Amir & Shikha (2004); Kane et al. (1988); Akhtar et al. (2010). For the crystal structures of related N-free triazole derivatives, see for example: Qadeer et al. (2007); and for N-subsituted derivatives, see for example: Zhao et al. (2010); Wu et al. (2009). Working with sulfur-containing heterocycles may provide unexpected results and the title compound was obtained within an unprecedented series of results, see: Ben Othman et al. (2014).
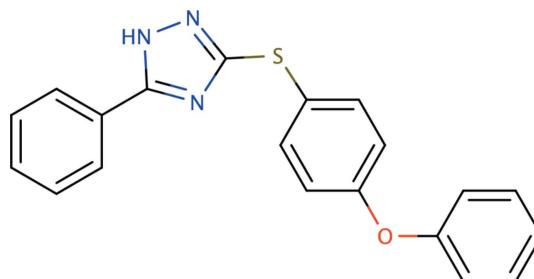
Experimental

Crystal data

$C_{20}H_{15}N_3OS$	V = 1700.5 (2) Å^3
M_r = 345.41	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 16.6112 (12) Å	μ = 0.20 mm^{-1}
b = 5.8445 (5) Å	T = 293 K
c = 17.5415 (10) Å	0.35 × 0.25 × 0.12 mm
β = 93.131 (5)°	

Data collection

Bruker–Nonius KappaCCD diffractometer	44120 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3099 independent reflections
T_{min} = 0.932, T_{max} = 0.976	2333 reflections with $I > 2\sigma(I)$
	R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.152	
S = 1.02	
3099 reflections	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
207 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N2^i	0.91 (3)	2.05 (3)	2.944 (3)	170 (2)
C16—H16···S1^{ii}	0.93	2.77	3.694 (2)	170

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

Data collection: COLLECT (Bruker–Nonius, 1998); cell refinement: DIRAX/LSQ (Duisenberg, 1992); data reduction: EVALCCD (Duisenberg et al., 2003); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2.

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2718).

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supplementary materials

3-[(4-Phenoxyphenyl)sulfanyl]-5-phenyl-1*H*-1,2,4-triazole

Raja Ben Othman, Mathieu Marchivie, Franck Suzenet and Sylvain Routier

1. Comment

From a medicinal chemistry point of view, it is of great interest to develop efficient methods for the synthesis and the functionalization of 1,2,4-triazoles, as they are known to possess a wide range of biological activities, such as, as anticancer (Holla *et al.*, 2002, 2003) antitubercular (Walczak *et al.*, 2004), antimicrobial (Zitouni *et al.*, 2005; Prasad *et al.*, 2009; Wael *et al.*, 2012), anticonvulsant (Almasirad *et al.*, 2004), anti-inflammatory, analgesic (Amir & Shikha, 2004), antidepressant (Kane *et al.*, 1988), and urease inhibitors (Akhtar *et al.*, 2010). Thus, the synthesis of 1,2,4-triazoles and the investigation of their chemical and biological behaviour have acquired more importance in recent decades for these reasons.

An efficient and convenient method was developed for the formation of substituted thiotriazoles via an organometallic addition and subsequent ring opening sequence of 3-substituted-[1,2,4]triazolo[3,4-][1,3,4]thiadiazole. This method is applicable to a wide range of substrates containing different functional groups and furnishes excellent yields of the corresponding unsubstituted 3 or 5-alkyl, aryl, alkynyl and alkenyl sulfanyl-1,2,4-triazole products.

Interestingly, working with sulfur-containing heterocycles may provide unexpected results and we report herein on the crystal structure of one derivative obtained within an unprecedented series of results (Ben Othman *et al.*, 2014).

The molecular structure of the title molecule is illustrated in Fig. 1. The molecule is V-shaped about atom S1. In the 4-phenoxyphenyl group the two rings (C9-C14 and C15-C20) are inclined to one another by 74.52 (13) °. These rings are inclined to the triazole ring (N1-N3/C7/C8) by 72.20 (15) and 72.30 (15) °, respectively. The phenyl ring (C1-C6) is inclined to the triazole ring by 10.85 (12) °.

In the crystal, molecules are linked via N-H⋯N hydrogen bonds forming chains propagating along [010]; see Table 1 and Fig. 2. These chains are linked via pairs of C-H⋯S hydrogen bonds forming sheets lying parallel to the ac plane (Table 1 and Fig. 2).

2. Experimental

For the synthesis of the title compound, see Fig. 3. In a 25 ml flask, phenyl ZnBr solution in THF (1.5 mmol, 0.5*M*) was added drop wise under argon at room temperature to a solution of 3-Phenyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazole (0.5 mmol) in THF (5 ml), and the mixture was stirred for 25 min (see Fig 3). At the end of the reaction, the mixture was quenched with 15 mL of an aqueous solution of saturated NH$_4$Cl, and extracted with CH$_2$Cl$_2$ (2 × 20 ml). The extract was dried over magnesium sulfate, filtered and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (eluent 6:4 petroleum ether/AcOEt). The title compound was obtained as a white solid in 80% yield. R_f = 0.60 (petroleum ether/EtOAc, 6:4); M.p. 318-320 K. HRMS (EI—MS): *m/z* calcd for C$_{20}$H$_{15}$N$_3$OS: 346.10086 [*M* + H]$^+$, found: 346.10112. Crystals of the title compound were obtained by vapor diffusion of petroleum ether into a solution of the title compound in a CH$_2$Cl$_2$/Et$_2$O/pentane mixture. Spectroscopic data for the title compound is available in the archived CIF.

3. Refinement

The NH H atom was located in a difference Fourier map and freely refined. The C-bound H atoms were included in calculated positions and treated as riding atoms: C-H = 0.93 Å with $U_{iso}(H) = 1.2U_{eq}(C)$.
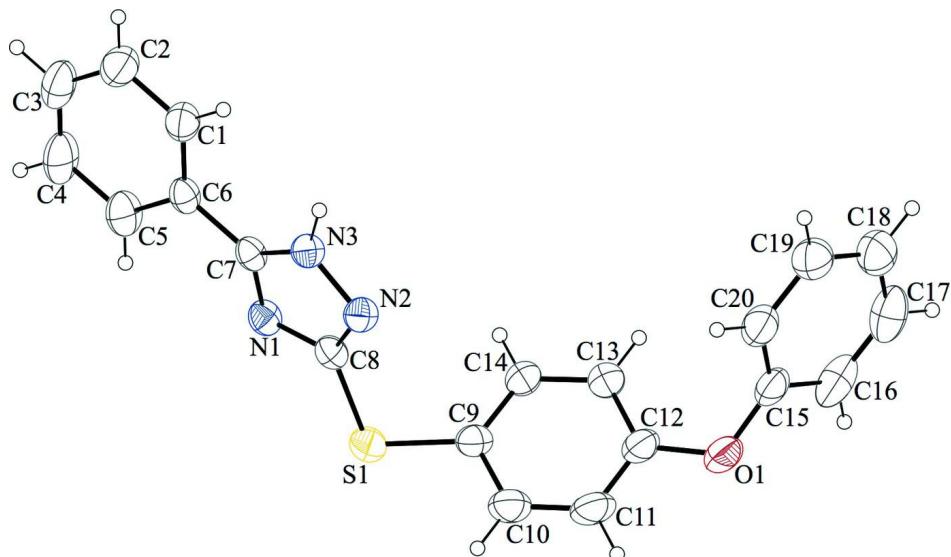
Figure 1

The molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.
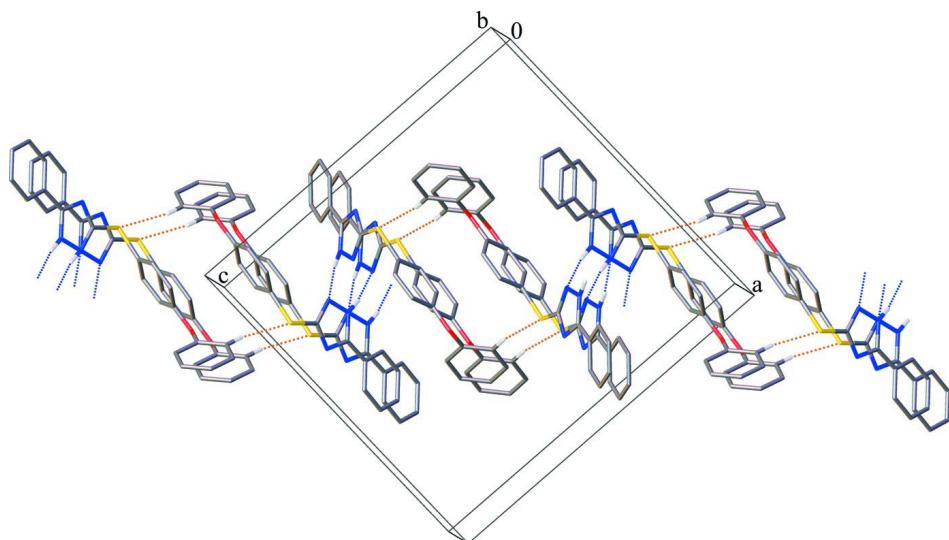
Figure 2

A perspective view along the b axis of the crystal pack of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for calrity).
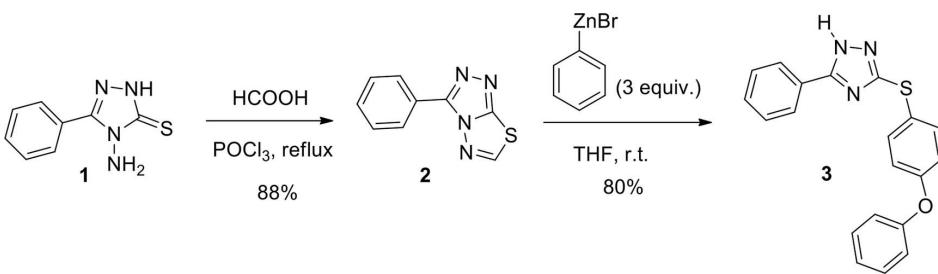
Figure 3

The synthetic route of the title compound.

3-[(4-Phenoxyphenyl)sulfanyl]-5-phenyl-1*H*-1,2,4-triazole

Crystal data

$C_{20}H_{15}N_3OS$	$Z = 4$
$M_r = 345.41$	$F(000) = 720$
Monoclinic, $P2_1/n$	$D_x = 1.349$ Mg m^{-3}
Hall symbol: -P 2yn	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 16.6112$ (12) Å	$\mu = 0.20$ mm^{-1}
$b = 5.8445$ (5) Å	$T = 293$ K
$c = 17.5415$ (10) Å	Block, colourless
$\beta = 93.131$ (5)°	$0.35 \times 0.25 \times 0.12$ mm
$V = 1700.5$ (2) Å^3	

Data collection

Bruker–Nonius KappaCCD diffractometer	44120 measured reflections
Radiation source: sealed X-ray tube	3099 independent reflections
Graphite monochromator	2333 reflections with $I > 2\sigma(I)$
profile data from φ scans and ω scans	$R_{int} = 0.034$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.4°$, $\theta_{min} = 3.3°$
	$h = -20 \rightarrow 20$
	$k = -7 \rightarrow 6$
$T_{min} = 0.932$, $T_{max} = 0.976$	$l = -21 \rightarrow 21$

Refinement

|	
Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.052$	Hydrogen site location: mixed
$wR(F^2) = 0.152$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.02$	
3099 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 1.0533P]$ where $P = (F_o^2 + 2F_c^2)/3$
207 parameters	
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.28$ e Å^{-3}
	$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Experimental. Spectroscopic data for the title compound: IR (ATR diamond): v (cm^{-1}) = 3082, 2927, 2864, 1581, 1482, 1324, 1242, 1006, 869, 786, 601, 725, 688; ^{1}H NMR (400 MHz, CDCl$_3$) δ (p.p.m.) = 12.92 (br. s, 1H), 7.90 (d, J = 6.9 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H), 7.43–7.28 (m, 5H), 7.15 (t, J = 7.3 Hz, 1H), 6.96 (d, J = 7.8 Hz, 2H), 6.84 (d, J = 8.4 Hz, 2H); ^{13}C NMR DEPT (101 MHz, CDCl$_3$): δ (p.p.m.) = 134.9 (2CH$_{Ar}$), 130.2 (CH$_{Ar}$), 129.9 (2CH$_{Ar}$), 128.8 (2CH$_{Ar}$), 126.5 (2CH$_{Ar}$), 124.1 (CH$_{Ar}$), 119.7 (2CH$_{Ar}$), 119 (2CH$_{Ar}$).

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}	Occ. (<1)
S1	0.74490 (4)	0.10572 (13)	0.07643 (5)	0.0708 (3)	
N3	0.69265 (12)	0.6140 (4)	0.19811 (11)	0.0507 (5)	
N2	0.74402 (12)	0.4385 (4)	0.18506 (11)	0.0540 (5)	
N1	0.64442 (12)	0.4567 (4)	0.09308 (11)	0.0539 (5)	
C7	0.63379 (13)	0.6216 (4)	0.14328 (13)	0.0474 (5)	
C6	0.56826 (8)	0.7865 (3)	0.13936 (9)	0.0518 (6)	
C1	0.56878 (10)	0.9796 (3)	0.18563 (10)	0.0661 (7)	
H1	0.6109	1.0031	0.2219	0.079*	
C2	0.50635 (12)	1.1377 (3)	0.17765 (12)	0.0811 (9)	
H2	0.5067	1.2669	0.2086	0.097*	
C3	0.44340 (10)	1.1026 (4)	0.12341 (13)	0.0885 (11)	
H3A	0.4016	1.2083	0.1181	0.106*	
C4	0.44288 (9)	0.9094 (4)	0.07715 (11)	0.0903 (11)	
H4	0.4008	0.8859	0.0409	0.108*	
C5	0.50531 (11)	0.7514 (3)	0.08512 (10)	0.0746 (8)	
H5	0.5050	0.6222	0.0542	0.090*	
C9	0.85042 (16)	0.1109 (4)	0.09473 (14)	0.0575 (6)	
C12	1.01532 (19)	0.0906 (5)	0.1147 (2)	0.0795 (9)	
C8	0.71159 (14)	0.3513 (4)	0.12111 (13)	0.0508 (6)	
C14	0.89725 (16)	0.2919 (5)	0.07283 (17)	0.0676 (7)	
H14	0.8728	0.4205	0.0506	0.081*	
C13	0.97963 (17)	0.2836 (5)	0.08361 (19)	0.0751 (8)	
H13	1.0110	0.4074	0.0700	0.090*	
C10	0.8872 (2)	−0.0810 (5)	0.12533 (18)	0.0750 (8)	
H10	0.8562	−0.2039	0.1403	0.090*	
C15	1.14533 (12)	0.2518 (3)	0.13117 (13)	0.0779 (9)	
C16	1.20579 (14)	0.2829 (4)	0.08041 (11)	0.0981 (13)	
H16	1.2114	0.1797	0.0407	0.118*	
C17	1.25792 (12)	0.4683 (5)	0.08903 (12)	0.0986 (12)	
H17	1.2984	0.4891	0.0551	0.118*	
C18	1.24959 (12)	0.6225 (4)	0.14841 (16)	0.0937 (11)	
H18	1.2845	0.7465	0.1542	0.112*	
C19	1.18913 (14)	0.5914 (3)	0.19916 (12)	0.0873 (10)	
H19	1.1836	0.6945	0.2389	0.105*	
C20	1.13700 (11)	0.4060 (4)	0.19055 (12)	0.0786 (9)	
H20	1.0966	0.3852	0.2245	0.094*	
C11	0.9694 (2)	−0.0913 (5)	0.1338 (2)	0.0885 (10)	
H11	0.9941	−0.2238	0.1527	0.106*	
H3	0.7060 (16)	0.717 (5)	0.2355 (16)	0.070 (8)*	
O1	1.09730 (15)	0.0639 (4)	0.1246 (2)	0.1279 (16)	0.997 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0633 (5)	0.0627 (5)	0.0859 (5)	−0.0058 (3)	−0.0005 (4)	−0.0210 (4)
N3	0.0525 (11)	0.0532 (12)	0.0451 (11)	0.0006 (9)	−0.0107 (9)	−0.0013 (9)
N2	0.0555 (12)	0.0555 (12)	0.0496 (11)	0.0026 (10)	−0.0097 (9)	0.0026 (9)
N1	0.0519 (12)	0.0602 (12)	0.0485 (11)	−0.0047 (10)	−0.0077 (9)	−0.0014 (10)
C7	0.0451 (12)	0.0534 (14)	0.0431 (12)	−0.0078 (10)	−0.0040 (10)	0.0065 (10)
C6	0.0446 (12)	0.0599 (15)	0.0501 (13)	−0.0055 (11)	−0.0035 (10)	0.0106 (11)
C1	0.0546 (15)	0.0708 (18)	0.0727 (17)	0.0024 (14)	0.0016 (13)	0.0024 (15)
C2	0.0718 (19)	0.076 (2)	0.098 (2)	0.0122 (16)	0.0194 (17)	0.0087 (17)
C3	0.0581 (18)	0.104 (3)	0.104 (2)	0.0201 (18)	0.0124 (17)	0.043 (2)
C4	0.0576 (17)	0.124 (3)	0.087 (2)	0.0107 (19)	−0.0181 (16)	0.026 (2)
C5	0.0611 (17)	0.094 (2)	0.0662 (17)	−0.0001 (16)	−0.0168 (13)	0.0056 (16)
C9	0.0646 (16)	0.0480 (14)	0.0596 (15)	0.0014 (12)	−0.0005 (12)	−0.0038 (11)
C12	0.0659 (18)	0.0560 (18)	0.114 (3)	0.0176 (14)	−0.0201 (17)	−0.0137 (16)
C8	0.0505 (13)	0.0522 (14)	0.0492 (13)	−0.0070 (11)	−0.0020 (10)	0.0019 (11)
C14	0.0598 (16)	0.0539 (16)	0.088 (2)	0.0077 (13)	−0.0042 (14)	0.0132 (14)
C13	0.0616 (17)	0.0563 (17)	0.107 (2)	0.0024 (14)	−0.0034 (16)	0.0073 (16)
C10	0.087 (2)	0.0506 (16)	0.086 (2)	0.0012 (14)	−0.0052 (16)	0.0067 (14)
C15	0.0549 (16)	0.070 (2)	0.106 (2)	0.0231 (15)	−0.0157 (16)	−0.0158 (17)
C16	0.082 (2)	0.137 (4)	0.074 (2)	0.050 (2)	−0.0119 (18)	−0.029 (2)
C17	0.069 (2)	0.148 (4)	0.079 (2)	0.026 (2)	0.0060 (17)	0.027 (2)
C18	0.076 (2)	0.094 (3)	0.110 (3)	0.0044 (19)	−0.005 (2)	0.017 (2)
C19	0.081 (2)	0.085 (2)	0.096 (2)	0.0046 (18)	0.0003 (18)	−0.0168 (19)
C20	0.0653 (18)	0.083 (2)	0.088 (2)	0.0146 (16)	0.0087 (16)	−0.0074 (17)
C11	0.098 (2)	0.0485 (17)	0.116 (3)	0.0183 (17)	−0.028 (2)	0.0039 (16)
O1	0.0700 (17)	0.0667 (17)	0.242 (4)	0.0247 (12)	−0.0404 (18)	−0.0344 (18)

Geometric parameters (Å, º)

S1—C9	1.765 (3)	C12—C13	1.374 (4)
S1—C8	1.740 (3)	C12—C11	1.361 (5)
N3—N2	1.362 (3)	C12—O1	1.372 (4)
N3—C7	1.334 (3)	C14—H14	0.9300
N3—H3	0.91 (3)	C14—C13	1.372 (4)
N2—C8	1.320 (3)	C13—H13	0.9300
N1—C7	1.324 (3)	C10—H10	0.9300
N1—C8	1.344 (3)	C10—C11	1.366 (5)
C7—C6	1.453 (3)	C15—C16	1.3900
C6—C1	1.3900	C15—C20	1.3900
C6—C5	1.3900	C15—O1	1.359 (3)
C1—H1	0.9300	C16—H16	0.9300
C1—C2	1.3900	C16—C17	1.3900
C2—H2	0.9300	C17—H17	0.9300
C2—C3	1.3900	C17—C18	1.3900
C3—H3A	0.9300	C18—H18	0.9300
C3—C4	1.3900	C18—C19	1.3900
C4—H4	0.9300	C19—H19	0.9300
C4—C5	1.3900	C19—C20	1.3900

C5—H5	0.9300	C20—H20	0.9300
C9—C14	1.380 (4)	C11—H11	0.9300
C9—C10	1.372 (4)		
C8—S1—C9	103.95 (12)	N2—C8—N1	115.2 (2)
N2—N3—H3	119.3 (18)	N1—C8—S1	119.43 (18)
C7—N3—N2	110.2 (2)	C9—C14—H14	119.7
C7—N3—H3	129.7 (18)	C13—C14—C9	120.5 (3)
C8—N2—N3	101.72 (19)	C13—C14—H14	119.7
C7—N1—C8	103.21 (19)	C12—C13—H13	120.4
N3—C7—C6	125.0 (2)	C14—C13—C12	119.3 (3)
N1—C7—N3	109.6 (2)	C14—C13—H13	120.4
N1—C7—C6	125.33 (19)	C9—C10—H10	120.0
C1—C6—C7	122.03 (14)	C11—C10—C9	120.0 (3)
C1—C6—C5	120.0	C11—C10—H10	120.0
C5—C6—C7	117.92 (14)	C16—C15—C20	120.0
C6—C1—H1	120.0	O1—C15—C16	119.5 (2)
C2—C1—C6	120.0	O1—C15—C20	120.4 (2)
C2—C1—H1	120.0	C15—C16—H16	120.0
C1—C2—H2	120.0	C15—C16—C17	120.0
C3—C2—C1	120.0	C17—C16—H16	120.0
C3—C2—H2	120.0	C16—C17—H17	120.0
C2—C3—H3A	120.0	C18—C17—C16	120.0
C2—C3—C4	120.0	C18—C17—H17	120.0
C4—C3—H3A	120.0	C17—C18—H18	120.0
C3—C4—H4	120.0	C19—C18—C17	120.0
C3—C4—C5	120.0	C19—C18—H18	120.0
C5—C4—H4	120.0	C18—C19—H19	120.0
C6—C5—H5	120.0	C18—C19—C20	120.0
C4—C5—C6	120.0	C20—C19—H19	120.0
C4—C5—H5	120.0	C15—C20—H20	120.0
C14—C9—S1	122.1 (2)	C19—C20—C15	120.0
C10—C9—S1	118.3 (2)	C19—C20—H20	120.0
C10—C9—C14	119.3 (3)	C12—C11—C10	120.6 (3)
C11—C12—C13	120.2 (3)	C12—C11—H11	119.7
C11—C12—O1	116.5 (3)	C10—C11—H11	119.7
O1—C12—C13	123.2 (3)	C15—O1—C12	119.5 (2)
N2—C8—S1	125.17 (19)		
S1—C9—C14—C13	−175.8 (2)	C9—C10—C11—C12	2.4 (5)
S1—C9—C10—C11	174.0 (3)	C8—S1—C9—C14	−57.9 (3)
N3—N2—C8—S1	−175.31 (18)	C8—S1—C9—C10	128.3 (2)
N3—N2—C8—N1	−0.1 (3)	C8—N1—C7—N3	0.4 (3)
N3—C7—C6—C1	12.0 (3)	C8—N1—C7—C6	−179.7 (2)
N3—C7—C6—C5	−170.67 (19)	C14—C9—C10—C11	0.0 (5)
N2—N3—C7—N1	−0.5 (3)	C13—C12—C11—C10	−2.8 (6)
N2—N3—C7—C6	179.66 (19)	C13—C12—O1—C15	24.8 (5)
N1—C7—C6—C1	−167.84 (18)	C10—C9—C14—C13	−2.0 (4)
N1—C7—C6—C5	9.5 (3)	C15—C16—C17—C18	0.0

C7—N3—N2—C8	0.4 (2)	C16—C15—C20—C19	0.0
C7—N1—C8—S1	175.31 (17)	C16—C15—O1—C12	−122.6 (3)
C7—N1—C8—N2	−0.2 (3)	C16—C17—C18—C19	0.0
C7—C6—C1—C2	177.32 (18)	C17—C18—C19—C20	0.0
C7—C6—C5—C4	−177.43 (17)	C18—C19—C20—C15	0.0
C6—C1—C2—C3	0.0	C20—C15—C16—C17	0.0
C1—C6—C5—C4	0.0	C20—C15—O1—C12	60.7 (4)
C1—C2—C3—C4	0.0	C11—C12—C13—C14	0.8 (5)
C2—C3—C4—C5	0.0	C11—C12—O1—C15	−158.7 (3)
C3—C4—C5—C6	0.0	O1—C12—C13—C14	177.2 (3)
C5—C6—C1—C2	0.0	O1—C12—C11—C10	−179.5 (3)
C9—S1—C8—N2	−34.3 (2)	O1—C15—C16—C17	−176.7 (2)
C9—S1—C8—N1	150.7 (2)	O1—C15—C20—C19	176.7 (2)
C9—C14—C13—C12	1.6 (5)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N2[i]	0.91 (3)	2.05 (3)	2.944 (3)	170 (2)
C16—H16···S1[ii]	0.93	2.77	3.694 (2)	170

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