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Comparison of crystal structures of 4-(benzo[*b*]-thiophen-2-yl)-5-(3,4,5-trimethoxyphenyl)-2*H*-1,2,3-triazole and 4-(benzo[*b*]thiophen-2-yl)-2-methyl-5-(3,4,5-trimethoxyphenyl)-2*H*-1,2,3-triazole

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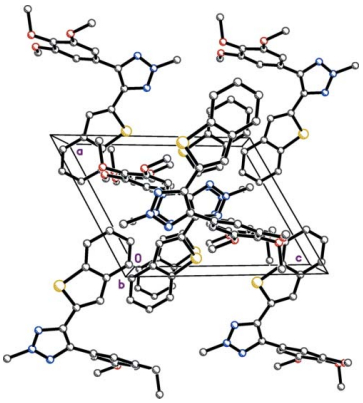
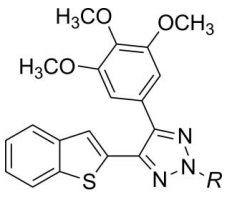
The title compound, $C_{19}H_{17}N_3O_3S$ (I), was prepared by a [3 + 2]cycloaddition azide condensation reaction using sodium azide and L-proline as a Lewis base catalyst. *N*-Methylation of compound (I) using CH_3I gave compound (II), $C_{20}H_{19}N_3O_3S$. The benzothiophene ring systems in (I) and (II) are almost planar, with r.m.s deviations from the mean plane = 0.0205 (14) in (I) and 0.016 (2) Å in (II). In (I) and (II), the triazole rings make dihedral angles of 32.68 (5) and 10.43 (8)°, respectively, with the mean planes of the benzothiophene ring systems. The trimethoxy phenyl rings make dihedral angles with the benzothiophene rings of 38.48 (4) in (I) and 60.43 (5)° in (II). In the crystal of (I), the molecules are linked into chains by N—H···O hydrogen bonds with $R_1^2(5)$ ring motifs. After the *N*-methylation of structure (I), no hydrogen-bonding interactions were observed for structure (II). The crystal structure of (II) has a minor component of disorder that corresponds to a 180° flip of the benzothiophene ring system [occupancy ratio 0.9363 (14):0.0637 (14)].

1. Chemical context

In continuation of our work on the development of benzothiophene cyano combretastatin A-4 analogs as anti-cancer agents (Penthala *et al.*, 2013), we have synthesized a series of novel CA-4 analogs by constructing a triazole ring structure (I) by chemical modification of the cyano group on the stilbene unit of cyano-CA-4 analogs utilizing a [3 + 2]cycloaddition azide condensation reaction with sodium azide in the presence of L-proline Lewis base as catalyst. This chemical modification is essential to restrict the tendency toward *cis–trans* isomerization of the cyano-stilbene moiety in cyano-CA-4 analogs (Penthala *et al.*, 2013). To further check the position of the hydrogen atom in the triazole ring system in (I), an *N*-methylation reaction was carried out on (I) using CH_3I, resulting in compound (II).





(I) *R* = H
(II) *R* = CH_3

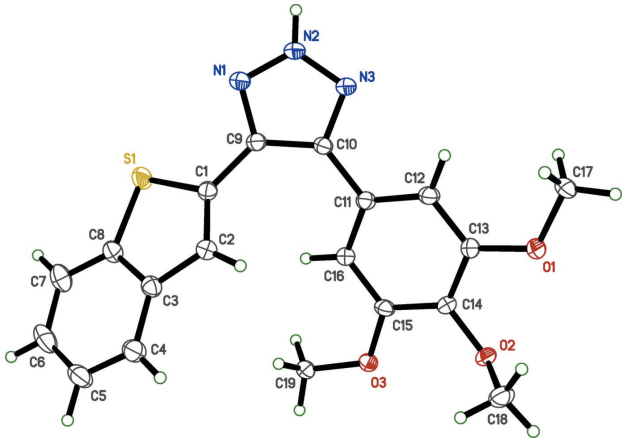


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

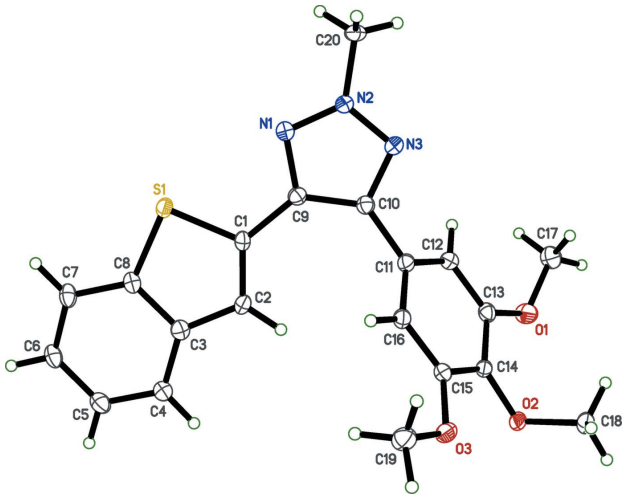


Figure 2
The molecular structure of (II), with displacement ellipsoids drawn at the 50% probability level.

2. Structural commentary

In order to obtain detailed information on the structural conformations of (I) and (II) for analysis of structure–activity relationships (SAR), including the position of the hydrogen atom in the triazole ring system of (I) and the position of methylation on the triazole ring system in (II), we determined the X-ray crystal structures of (I) and (II); see Figs. 1 and 2, respectively.

Selected geometric parameters are given in Tables 1 and 2 for (I) and (II), respectively. The benzothiophene rings are almost planar with r.m.s deviations from the mean plane of 0.0205 (14) in (I) and 0.016 (2) Å in (II), with bond distances and angles comparable with those reported for other benzothiophene derivatives (Sonar *et al.*, 2007) and triazole analogs (Madadi *et al.*, 2014). The triazole rings make dihedral angles of 32.68 (5)° and 10.43 (8)°, respectively, in (I) and (II) with the mean plane of the benzothiophene ring systems. The trimethoxyphenyl rings make dihedral angles of 38.48 (4) in (I) and 60.43 (5)° in (II) with the benzothiophene ring systems. In both compounds (I) and (II), deviations from ideal geometry are observed in the bond angles C1—S1—C8, N2—N1—C9, N2—N3—C10, which are compressed, and C1—C9—C10, C9—C10—C11, C2—C3—C4, which are expanded (see Tables 1 and 2). After *N*-methylation, no significant difference is observed for the N1—N2—N3 bond angle [116.2 (1) and 115.9 (1)°, respectively, for (I) and (II)]. The crystal structure of (II) has a minor component of disorder that corresponds to a 180° flip of the benzothiophene ring system [occupancy ratio 0.9363 (14):0.0637 (14)].

Table 1
Selected geometric parameters (Å, °) for (I).

N1—N2	1.324 (2)	N2—H2*N*	0.87 (2)
N1—C9	1.343 (2)	N3—C10	1.345 (2)
N2—N3	1.330 (2)		
C8—S1—C1	91.50 (8)	C10—C9—C1	131.64 (14)
N2—N1—C9	103.74 (13)	C9—C10—C11	131.16 (14)
N2—N3—C10	103.74 (13)	O1—C13—C14	114.89 (14)
C4—C3—C2	129.50 (16)		

Table 2
Selected geometric parameters (Å, °) for (II).

N1—N2	1.3266 (15)	N2—C20	1.4527 (16)
N1—C9	1.3477 (16)	N3—C10	1.3450 (16)
N2—N3	1.3279 (15)		
N1—N2—N3	115.92 (10)	C4′—C3′—C2′	132 (2)
C2—C1—C9	129.94 (17)	C7′—C8′—S1′	129 (2)
C8—S1—C1	91.33 (8)	C1′—C9—C10	127.3 (11)
C4—C3—C2	129.79 (17)	C10—C9—C1	132.41 (13)
C9—C1′—S1′	128.0 (18)	C9—C10—C11	132.90 (12)
C8′—S1′—C1′	95.8 (12)		

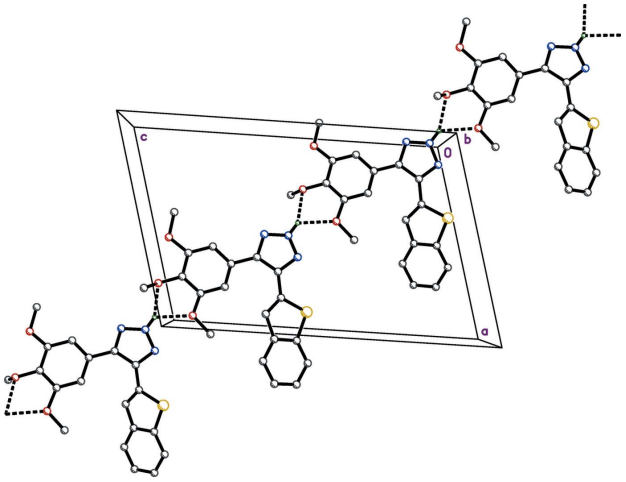


Figure 3
Hydrogen bonding in the crystal structure of (I), viewed along the *b* axis. Dashed lines represent hydrogen bonds, which join molecules into chains along the [101] direction.

Table 3
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O2^{i}	0.87 (2)	2.16 (2)	2.9381 (18)	147.6 (18)
N2—H2N···O3^{i}	0.87 (2)	2.20 (2)	2.8503 (18)	130.8 (17)

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

3. Supramolecular features

Hydrogen bonding and the mode of packing of (I) is illustrated in Fig. 3, and the mode of packing of (II) is illustrated in Fig. 4. In the structure of (I), the molecules are linked by intermolecular hydrogen bonds (N2—H2N···O2 and N2—H2N···O3), forming $R_1^2(5)$ ring motifs (Table 3), which propagate as chains along the [101] direction. Contacts between adjacent chains form two-dimensional pleated-sheet networks in the *ac* plane. No significant hydrogen-bonding interactions were found in the structure of (II).

4. Database survey

A search of the 2014 release of the Cambridge Structural Database on unit-cell dimensions for (I) and (II) revealed four triazole structures (HOZZAY, UPEWAO, SAFZEG & VUSNEC), although none bore any particular relation to compounds (I) or (II). A search on the triazole ring fragment with either H or methyl attached to the middle N atom revealed 48 and 17 hits, respectively, none of which contained either benzothiophene or trimethoxybenzene functional groups.

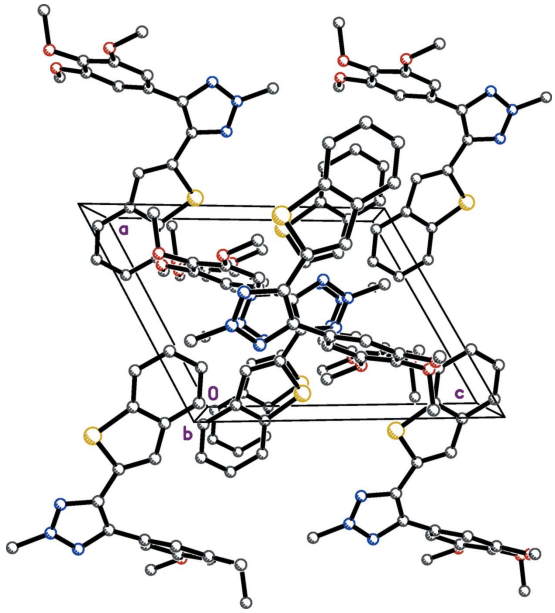


Figure 4
Crystal packing of (II), as viewed along the *b* axis.

Table 4
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{19}H_{17}N_3O_3S$	$C_{20}H_{19}N_3O_3S$
M_r	367.41	381.44
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$
Temperature (K)	90	90
a, b, c (Å)	11.8983 (2), 8.1860 (1), 18.4582 (3)	8.8579 (1), 11.0761 (1), 11.2626 (1)
α, β, γ (°)	90, 105.5046 (7), 90	106.859 (4), 111.668 (5), 105.498 (4)
V (Å^3)	1732.39 (5)	891.51 (4)
Z	4	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.21	0.21
Crystal size (mm)	0.30 × 0.30 × 0.05	0.22 × 0.20 × 0.15
Data collection		
Diffractometer	Nonius KappaCCD	Nonius KappaCCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2008*a*)	Multi-scan (*SADABS*; Sheldrick, 2008*a*)
T_{min}, T_{max}	0.816, 0.966	0.858, 0.962
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28105, 3984, 3093	36591, 4097, 3572
R_{int}	0.045	0.045
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650	0.651
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.124, 1.07	0.037, 0.096, 1.08
No. of reflections	3984	4097
No. of parameters	241	276
No. of restraints	0	161
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.29	0.31, −0.28

Computer programs: *COLLECT* (Nonius, 1998), *SCALEPACK* and *DENZO-SMN* (Otwinowski & Minor, 2006), *SHELXS97*, *SHELXL2013*, *SHELXL2014* and *XP* in *SHELXTL* (Sheldrick, 2008*b*) and *CIFFIX* (Parkin, 2013).

5. Synthesis and crystallization

The title compounds were prepared according to a previously reported procedure (Penthala *et al.*, 2014). Recrystallization from methanol afforded (I) and (II) as yellow and pale-yellow crystalline products, respectively, which were suitable for X-ray analysis.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 4. H atoms were found in difference Fourier maps. Carbon-bound hydrogens were subsequently placed at idealized positions with constrained distances of 0.98 (RCH_3) and 0.95 Å (Csp^2H). Coordinates of the N-bound hydrogen were refined freely. U_{iso}(H) values were set to either $1.2U_{eq}$ or $1.5U_{eq}$ (RCH_3) of the attached atom.

Refinement progress was checked using *PLATON* (Spek, 2009) and by an *R*-tensor (Parkin, 2000). To ensure satisfactory refinement of disordered groups in the structure, a combination of constraints and restraints was employed. The constraints (*SHELXL* command EADP) were used to fix overlapping fragments. Restraints were used to maintain the integrity of ill-defined or disordered groups (*SHELXL* commands SAME and RIGU).

In structure (II), there was a small amount of a second conformation for the benzothiophene ring systems, with major and minor component fractions of 93.63 (14) and 6.37 (14)%, respectively.

Acknowledgements

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supporting information

Acta Cryst. (2014). E**70**, 392-395 [doi:10.1107/S1600536814023095]

Comparison of crystal structures of 4-(benzo[*b*]thiophen-2-yl)-5-(3,4,5-trimethoxyphenyl)-2*H*-1,2,3-triazole and 4-(benzo[*b*]thiophen-2-yl)-2-methyl-5-(3,4,5-trimethoxyphenyl)-2*H*-1,2,3-triazole

Narsimha Reddy Penthala, Nikhil Reddy Madadi, Shobanbabu Bommagani, Sean Parkin and Peter A. Crooks

Computing details

For both compounds, data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 2006); data reduction: *DENZO-SMN* (Otwinowski & Minor, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008b). Program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008b) for (I); *SHELXL2014* (Sheldrick, 2008b) for (II). For both compounds, molecular graphics: *XP in SHELXTL* (Sheldrick, 2008b). Software used to prepare material for publication: *SHELXL2013* (Sheldrick, 2008b) and *CIFFIX* (Parkin, 2013) for (I); *SHELXL2014* (Sheldrick, 2008b) and *CIFFIX* (Parkin, 2013) for (II).

(I) 4-(Benzo[*b*]thiophen-2-yl)-5-(3,4,5-trimethoxyphenyl)-2*H*-1,2,3-triazole

Crystal data

$C_{19}H_{17}N_3O_3S$
$M_r = 367.41$
Monoclinic, $P2_1/n$
$a = 11.8983$ (2) Å
$b = 8.1860$ (1) Å
$c = 18.4582$ (3) Å
$\beta = 105.5046$ (7)°
$V = 1732.39$ (5) Å^3
$Z = 4$
$F(000) = 768$
$D_x = 1.409$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4236 reflections
$\theta = 1.0$–$27.5°$
$\mu = 0.21$ mm^{-1}
$T = 90$ K
Plate, yellow
0.30 × 0.30 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed-tube
Detector resolution: 9.1 pixels mm^{-1}
φ and ω scans at fixed $\chi = 55°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008a)
$T_{min} = 0.816$, $T_{max} = 0.966$
28105 measured reflections
3984 independent reflections
3093 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.8°$
$h = -15 \rightarrow 15$
$k = -10 \rightarrow 10$
$l = -23 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.124$
$S = 1.07$
3984 reflections
241 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.6092P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Special details

Experimental. The crystal was mounted with polyisobutene oil on the tip of a fine glass fibre, fastened in a copper mounting pin with electrical solder. It was placed directly into the cold stream of a liquid nitrogen based cryostat, according to published methods (Hope, H. (1994). *Prog. Inorg. Chem.* **41**, 1–19; Parkin, S. & Hope, H. (1998). *J. Appl. Cryst.* **31**, 945–953.).
Diffraction data were collected with the crystal at 90 K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

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.

Refinement. Refinement progress was checked using *PLATON* (Spek, 2009) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.39424 (4)	0.67530 (6)	0.05935 (2)	0.02500 (14)
N1	0.13369 (12)	0.64131 (18)	0.05222 (8)	0.0209 (3)
N2	0.03228 (12)	0.67054 (18)	0.06649 (8)	0.0214 (3)
H2N	−0.0306 (18)	0.686 (2)	0.0298 (12)	0.026*
N3	0.03376 (12)	0.68041 (18)	0.13868 (8)	0.0201 (3)
O1	0.07856 (10)	0.60706 (15)	0.42702 (6)	0.0223 (3)
O2	0.30089 (10)	0.69738 (14)	0.49128 (6)	0.0196 (3)
O3	0.44759 (9)	0.76268 (15)	0.40977 (6)	0.0208 (3)
C1	0.33215 (14)	0.5986 (2)	0.12815 (9)	0.0186 (3)
C2	0.41132 (14)	0.5191 (2)	0.18353 (9)	0.0195 (3)
H2	0.3920	0.4676	0.2247	0.023*
C3	0.52685 (14)	0.5204 (2)	0.17387 (9)	0.0200 (4)
C4	0.63017 (15)	0.4549 (2)	0.22005 (10)	0.0268 (4)
H4	0.6291	0.3961	0.2642	0.032*
C5	0.73349 (15)	0.4767 (2)	0.20067 (11)	0.0307 (4)
H5	0.8037	0.4322	0.2317	0.037*
C6	0.73606 (16)	0.5633 (3)	0.13592 (12)	0.0331 (5)
H6	0.8084	0.5787	0.1242	0.040*
C7	0.63591 (16)	0.6268 (2)	0.08883 (12)	0.0299 (4)
H7	0.6380	0.6841	0.0445	0.036*
C8	0.53112 (14)	0.6047 (2)	0.10787 (10)	0.0217 (4)

C9	0.20875 (14)	0.6295 (2)	0.12074 (9)	0.0173 (3)
C10	0.14626 (13)	0.6547 (2)	0.17521 (9)	0.0169 (3)
C11	0.18559 (14)	0.6599 (2)	0.25781 (9)	0.0171 (3)
C12	0.10917 (14)	0.6200 (2)	0.30137 (9)	0.0185 (3)
H12	0.0323	0.5837	0.2778	0.022*
C13	0.14657 (14)	0.6340 (2)	0.37917 (9)	0.0181 (3)
C14	0.26154 (14)	0.6801 (2)	0.41410 (9)	0.0171 (3)
C15	0.33703 (13)	0.7182 (2)	0.37040 (9)	0.0176 (3)
C16	0.29866 (14)	0.7127 (2)	0.29248 (9)	0.0172 (3)
H16	0.3492	0.7448	0.2629	0.021*
C17	−0.04088 (14)	0.5647 (3)	0.39398 (10)	0.0260 (4)
H17A	−0.0797	0.6537	0.3612	0.039*
H17B	−0.0800	0.5468	0.4338	0.039*
H17C	−0.0447	0.4646	0.3643	0.039*
C18	0.32569 (16)	0.5438 (2)	0.53021 (10)	0.0259 (4)
H18A	0.2566	0.4735	0.5159	0.039*
H18B	0.3464	0.5630	0.5846	0.039*
H18C	0.3909	0.4904	0.5167	0.039*
C19	0.53268 (14)	0.7777 (2)	0.36776 (9)	0.0212 (4)
H19A	0.5353	0.6761	0.3402	0.032*
H19B	0.6095	0.7987	0.4023	0.032*
H19C	0.5113	0.8685	0.3321	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0206 (2)	0.0338 (3)	0.0226 (2)	0.00439 (18)	0.00931 (17)	0.00745 (18)
N1	0.0158 (7)	0.0285 (8)	0.0174 (7)	−0.0002 (6)	0.0024 (5)	0.0010 (6)
N2	0.0157 (7)	0.0307 (8)	0.0159 (7)	0.0012 (6)	0.0006 (6)	0.0012 (6)
N3	0.0156 (7)	0.0280 (8)	0.0155 (7)	−0.0002 (6)	0.0018 (5)	0.0006 (6)
O1	0.0161 (6)	0.0335 (7)	0.0177 (6)	−0.0042 (5)	0.0052 (5)	0.0008 (5)
O2	0.0195 (6)	0.0246 (6)	0.0131 (5)	−0.0010 (5)	0.0018 (4)	0.0004 (5)
O3	0.0130 (5)	0.0316 (7)	0.0172 (6)	−0.0039 (5)	0.0028 (4)	−0.0021 (5)
C1	0.0164 (8)	0.0221 (9)	0.0172 (8)	−0.0025 (6)	0.0043 (6)	−0.0024 (6)
C2	0.0180 (8)	0.0209 (9)	0.0192 (8)	0.0007 (7)	0.0043 (6)	0.0000 (6)
C3	0.0174 (8)	0.0200 (9)	0.0228 (8)	−0.0011 (6)	0.0054 (6)	−0.0037 (7)
C4	0.0222 (9)	0.0298 (10)	0.0265 (9)	0.0032 (7)	0.0033 (7)	−0.0050 (8)
C5	0.0172 (8)	0.0348 (11)	0.0370 (10)	0.0046 (8)	0.0021 (7)	−0.0094 (9)
C6	0.0187 (9)	0.0350 (11)	0.0487 (12)	−0.0004 (8)	0.0143 (8)	−0.0058 (9)
C7	0.0246 (9)	0.0310 (10)	0.0389 (11)	−0.0005 (8)	0.0169 (8)	0.0014 (8)
C8	0.0189 (8)	0.0234 (9)	0.0239 (9)	0.0007 (7)	0.0076 (7)	0.0002 (7)
C9	0.0157 (8)	0.0197 (8)	0.0156 (8)	−0.0001 (6)	0.0025 (6)	0.0009 (6)
C10	0.0134 (7)	0.0201 (8)	0.0164 (8)	−0.0004 (6)	0.0025 (6)	0.0011 (6)
C11	0.0160 (8)	0.0181 (8)	0.0165 (8)	0.0012 (6)	0.0030 (6)	−0.0001 (6)
C12	0.0155 (8)	0.0211 (9)	0.0174 (8)	−0.0014 (6)	0.0016 (6)	0.0001 (6)
C13	0.0168 (8)	0.0211 (9)	0.0170 (8)	−0.0007 (6)	0.0055 (6)	0.0014 (6)
C14	0.0176 (8)	0.0197 (8)	0.0132 (7)	0.0008 (6)	0.0028 (6)	0.0011 (6)
C15	0.0129 (7)	0.0196 (8)	0.0183 (8)	−0.0004 (6)	0.0007 (6)	−0.0021 (6)

C16	0.0159 (8)	0.0196 (8)	0.0160 (8)	0.0006 (6)	0.0037 (6)	0.0004 (6)
C17	0.0139 (8)	0.0400 (11)	0.0235 (9)	−0.0043 (7)	0.0041 (7)	0.0044 (8)
C18	0.0266 (9)	0.0292 (10)	0.0197 (8)	−0.0005 (7)	0.0024 (7)	0.0069 (7)
C19	0.0143 (8)	0.0301 (9)	0.0193 (8)	−0.0014 (7)	0.0047 (6)	0.0006 (7)

Geometric parameters (Å, º)

S1—C8	1.7345 (17)	C6—C7	1.375 (3)
S1—C1	1.7474 (17)	C6—H6	0.9500
N1—N2	1.324 (2)	C7—C8	1.395 (2)
N1—C9	1.343 (2)	C7—H7	0.9500
N2—N3	1.330 (2)	C9—C10	1.416 (2)
N2—H2N	0.87 (2)	C10—C11	1.471 (2)
N3—C10	1.345 (2)	C11—C16	1.395 (2)
O1—C13	1.3660 (19)	C11—C12	1.404 (2)
O1—C17	1.4313 (19)	C12—C13	1.390 (2)
O2—C14	1.3829 (19)	C12—H12	0.9500
O2—C18	1.439 (2)	C13—C14	1.399 (2)
O3—C15	1.3712 (19)	C14—C15	1.394 (2)
O3—C19	1.4356 (19)	C15—C16	1.389 (2)
C1—C2	1.357 (2)	C16—H16	0.9500
C1—C9	1.460 (2)	C17—H17A	0.9800
C2—C3	1.433 (2)	C17—H17B	0.9800
C2—H2	0.9500	C17—H17C	0.9800
C3—C4	1.402 (2)	C18—H18A	0.9800
C3—C8	1.413 (2)	C18—H18B	0.9800
C4—C5	1.381 (3)	C18—H18C	0.9800
C4—H4	0.9500	C19—H19A	0.9800
C5—C6	1.397 (3)	C19—H19B	0.9800
C5—H5	0.9500	C19—H19C	0.9800
C8—S1—C1	91.50 (8)	C9—C10—C11	131.16 (14)
N2—N1—C9	103.74 (13)	C16—C11—C12	120.15 (15)
N1—N2—N3	116.21 (14)	C16—C11—C10	119.01 (14)
N1—N2—H2N	120.6 (13)	C12—C11—C10	120.77 (14)
N3—N2—H2N	123.2 (13)	C13—C12—C11	119.68 (15)
N2—N3—C10	103.74 (13)	C13—C12—H12	120.2
C13—O1—C17	117.13 (13)	C11—C12—H12	120.2
C14—O2—C18	113.13 (13)	O1—C13—C12	124.98 (14)
C15—O3—C19	116.85 (12)	O1—C13—C14	114.89 (14)
C2—C1—C9	129.19 (15)	C12—C13—C14	120.12 (15)
C2—C1—S1	112.12 (12)	O2—C14—C15	118.70 (14)
C9—C1—S1	118.67 (12)	O2—C14—C13	121.49 (14)
C1—C2—C3	113.49 (15)	C15—C14—C13	119.72 (14)
C1—C2—H2	123.3	O3—C15—C16	124.11 (15)
C3—C2—H2	123.3	O3—C15—C14	115.35 (14)
C4—C3—C8	118.91 (16)	C16—C15—C14	120.54 (14)
C4—C3—C2	129.50 (16)	C15—C16—C11	119.66 (15)

C8—C3—C2	111.58 (15)	C15—C16—H16	120.2
C5—C4—C3	119.33 (18)	C11—C16—H16	120.2
C5—C4—H4	120.3	O1—C17—H17A	109.5
C3—C4—H4	120.3	O1—C17—H17B	109.5
C4—C5—C6	120.79 (17)	H17A—C17—H17B	109.5
C4—C5—H5	119.6	O1—C17—H17C	109.5
C6—C5—H5	119.6	H17A—C17—H17C	109.5
C7—C6—C5	121.26 (17)	H17B—C17—H17C	109.5
C7—C6—H6	119.4	O2—C18—H18A	109.5
C5—C6—H6	119.4	O2—C18—H18B	109.5
C6—C7—C8	118.28 (18)	H18A—C18—H18B	109.5
C6—C7—H7	120.9	O2—C18—H18C	109.5
C8—C7—H7	120.9	H18A—C18—H18C	109.5
C7—C8—C3	121.41 (16)	H18B—C18—H18C	109.5
C7—C8—S1	127.30 (15)	O3—C19—H19A	109.5
C3—C8—S1	111.27 (12)	O3—C19—H19B	109.5
N1—C9—C10	108.37 (14)	H19A—C19—H19B	109.5
N1—C9—C1	119.98 (15)	O3—C19—H19C	109.5
C10—C9—C1	131.64 (14)	H19A—C19—H19C	109.5
N3—C10—C9	107.94 (14)	H19B—C19—H19C	109.5
N3—C10—C11	120.88 (14)		

C9—N1—N2—N3	0.50 (19)	C1—C9—C10—N3	179.11 (17)
N1—N2—N3—C10	−0.27 (19)	N1—C9—C10—C11	−177.91 (16)
C8—S1—C1—C2	−1.94 (14)	C1—C9—C10—C11	0.8 (3)
C8—S1—C1—C9	176.59 (14)	N3—C10—C11—C16	−148.68 (16)
C9—C1—C2—C3	−176.73 (16)	C9—C10—C11—C16	29.4 (3)
S1—C1—C2—C3	1.60 (19)	N3—C10—C11—C12	28.3 (2)
C1—C2—C3—C4	178.36 (18)	C9—C10—C11—C12	−153.60 (18)
C1—C2—C3—C8	−0.3 (2)	C16—C11—C12—C13	0.3 (2)
C8—C3—C4—C5	1.1 (3)	C10—C11—C12—C13	−176.69 (15)
C2—C3—C4—C5	−177.44 (17)	C17—O1—C13—C12	−1.4 (2)
C3—C4—C5—C6	0.2 (3)	C17—O1—C13—C14	178.07 (15)
C4—C5—C6—C7	−1.2 (3)	C11—C12—C13—O1	176.42 (15)
C5—C6—C7—C8	1.0 (3)	C11—C12—C13—C14	−3.1 (2)
C6—C7—C8—C3	0.3 (3)	C18—O2—C14—C15	−105.70 (17)
C6—C7—C8—S1	178.72 (15)	C18—O2—C14—C13	77.81 (19)
C4—C3—C8—C7	−1.4 (3)	O1—C13—C14—O2	−0.5 (2)
C2—C3—C8—C7	177.41 (16)	C12—C13—C14—O2	179.04 (15)
C4—C3—C8—S1	−179.98 (13)	O1—C13—C14—C15	−176.96 (14)
C2—C3—C8—S1	−1.20 (19)	C12—C13—C14—C15	2.6 (2)
C1—S1—C8—C7	−176.74 (18)	C19—O3—C15—C16	−10.7 (2)
C1—S1—C8—C3	1.76 (14)	C19—O3—C15—C14	170.44 (14)
N2—N1—C9—C10	−0.50 (18)	O2—C14—C15—O3	3.1 (2)
N2—N1—C9—C1	−179.41 (15)	C13—C14—C15—O3	179.67 (15)
C2—C1—C9—N1	−150.00 (18)	O2—C14—C15—C16	−175.80 (15)
S1—C1—C9—N1	31.8 (2)	C13—C14—C15—C16	0.8 (2)
C2—C1—C9—C10	31.4 (3)	O3—C15—C16—C11	177.62 (15)

S1—C1—C9—C10	−146.86 (16)	C14—C15—C16—C11	−3.6 (2)
N2—N3—C10—C9	−0.08 (18)	C12—C11—C16—C15	3.1 (2)
N2—N3—C10—C11	178.42 (15)	C10—C11—C16—C15	−179.95 (15)
N1—C9—C10—N3	0.37 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*N*···O2^{i}	0.87 (2)	2.16 (2)	2.9381 (18)	147.6 (18)
N2—H2*N*···O3^{i}	0.87 (2)	2.20 (2)	2.8503 (18)	130.8 (17)

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

(II) 4-(Benzo[*b*]thiophen-2-yl)-2-methyl-5-(3,4,5-trimethoxyphenyl)-2*H*-1,2,3-triazole

Crystal data

$C_{20}H_{19}N_3O_3S$
M_r = 381.44
Triclinic, $P\overline{1}$
a = 8.8579 (1) Å
b = 11.0761 (1) Å
c = 11.2626 (1) Å
α = 106.859 (4)°
β = 111.668 (5)°
γ = 105.498 (4)°
V = 891.51 (4) Å^3
Z = 2
$F(000)$ = 400
D_x = 1.421 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4076 reflections
θ = 1.0–27.5°
μ = 0.21 mm^{-1}
T = 90 K
Cut block, pale yellow
0.22 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed-tube
Detector resolution: 9.1 pixels mm^{-1}
φ and ω scans at fixed χ = 55°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008a)
T_{min} = 0.858, T_{max} = 0.962
36591 measured reflections
4097 independent reflections
3572 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 27.6°, θ_{min} = 2.1°
h = −11→11
k = −14→14
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.096
S = 1.08
4097 reflections
276 parameters
161 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.4023P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Special details

Experimental. The crystal was mounted with polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid nitrogen based cryostat, according to published methods (Hope, 1994; Parkin & Hope, 1998).
Diffraction data were collected with the crystal at 90 K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

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.

Refinement. Refinement progress was checked using *PLATON* (Spek, 2009) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.41837 (14)	0.62237 (11)	0.61716 (11)	0.0160 (2)	
N2	0.56418 (15)	0.73837 (11)	0.71234 (11)	0.0158 (2)	
N3	0.65185 (15)	0.80574 (11)	0.66113 (11)	0.0163 (2)	
O1	0.75768 (14)	1.08896 (10)	0.37490 (10)	0.0234 (2)	
O2	0.77188 (12)	0.90655 (10)	0.16639 (9)	0.0178 (2)	
O3	0.72458 (13)	0.64975 (10)	0.14267 (10)	0.0197 (2)	
C1	0.2506 (5)	0.5036 (4)	0.36036 (19)	0.0145 (4)	0.9363 (14)
S1	0.08551 (5)	0.39081 (4)	0.37446 (4)	0.01741 (12)	0.9363 (14)
C2	0.2034 (2)	0.48053 (18)	0.22483 (17)	0.0176 (3)	0.9363 (14)
H2	0.2776	0.5336	0.1986	0.021*	0.9363 (14)
C3	0.0313 (3)	0.3687 (3)	0.1242 (2)	0.0164 (4)	0.9363 (14)
C4	−0.0638 (3)	0.31782 (16)	−0.0245 (2)	0.0203 (4)	0.9363 (14)
H4	−0.0117	0.3565	−0.0725	0.024*	0.9363 (14)
C5	−0.2341 (3)	0.21078 (18)	−0.09952 (19)	0.0210 (4)	0.9363 (14)
H5	−0.2990	0.1766	−0.1996	0.025*	0.9363 (14)
C6	−0.3125 (2)	0.1519 (2)	−0.03024 (16)	0.0195 (4)	0.9363 (14)
H6	−0.4295	0.0784	−0.0841	0.023*	0.9363 (14)
C7	−0.2218 (2)	0.19944 (19)	0.11543 (18)	0.0186 (4)	0.9363 (14)
H7	−0.2742	0.1591	0.1624	0.022*	0.9363 (14)
C8	−0.0508 (2)	0.3086 (2)	0.19147 (18)	0.0161 (3)	0.9363 (14)
C1′	0.269 (8)	0.522 (7)	0.368 (2)	0.0145 (4)	0.0637 (14)
S1′	0.2341 (9)	0.5088 (7)	0.2013 (7)	0.0176 (3)	0.0637 (14)
C2′	0.134 (3)	0.418 (2)	0.3485 (19)	0.01741 (12)	0.0637 (14)
H2′	0.1347	0.3980	0.4251	0.021*	0.0637 (14)
C3′	−0.013 (4)	0.335 (4)	0.207 (2)	0.0161 (3)	0.0637 (14)
C4′	−0.173 (3)	0.221 (3)	0.150 (3)	0.0186 (4)	0.0637 (14)
H4′	−0.1949	0.1828	0.2106	0.022*	0.0637 (14)
C5′	−0.297 (4)	0.162 (3)	0.013 (3)	0.0195 (4)	0.0637 (14)
H5′	−0.4184	0.1070	−0.0167	0.023*	0.0637 (14)
C6′	−0.248 (4)	0.183 (3)	−0.086 (3)	0.0210 (4)	0.0637 (14)
H6′	−0.3283	0.1303	−0.1851	0.025*	0.0637 (14)
C7′	−0.079 (4)	0.284 (3)	−0.036 (3)	0.0203 (4)	0.0637 (14)
H7′	−0.0314	0.2892	−0.0985	0.024*	0.0637 (14)

C8′	0.020 (4)	0.377 (5)	0.108 (2)	0.0164 (4)	0.0637 (14)
C9	0.40890 (17)	0.61277 (13)	0.49233 (13)	0.0147 (2)	
C10	0.55623 (17)	0.72729 (13)	0.52005 (13)	0.0146 (2)	
C11	0.61431 (17)	0.77268 (13)	0.42743 (13)	0.0152 (3)	
C12	0.65731 (17)	0.91063 (13)	0.44855 (13)	0.0166 (3)	
H12	0.6472	0.9730	0.5204	0.020*	
C13	0.71519 (17)	0.95607 (13)	0.36342 (14)	0.0168 (3)	
C14	0.72907 (16)	0.86471 (13)	0.25757 (13)	0.0152 (3)	
C15	0.69315 (17)	0.72810 (13)	0.24151 (13)	0.0157 (3)	
C16	0.63395 (17)	0.68128 (13)	0.32534 (13)	0.0158 (3)	
H16	0.6073	0.5881	0.3131	0.019*	
C17	0.8251 (2)	1.19553 (14)	0.51199 (15)	0.0231 (3)	
H17A	0.7287	1.1871	0.5366	0.035*	
H17B	0.8707	1.2866	0.5122	0.035*	
H17C	0.9222	1.1864	0.5817	0.035*	
C18	0.96108 (18)	0.98269 (14)	0.22249 (14)	0.0202 (3)	
H18A	1.0093	1.0624	0.3134	0.030*	
H18B	0.9813	1.0158	0.1555	0.030*	
H18C	1.0214	0.9217	0.2368	0.030*	
C19	0.7008 (2)	0.51278 (14)	0.13104 (16)	0.0231 (3)	
H19A	0.7781	0.5191	0.2233	0.035*	
H19B	0.7324	0.4680	0.0608	0.035*	
H19C	0.5754	0.4577	0.1010	0.035*	
C20	0.63205 (18)	0.78558 (14)	0.86407 (13)	0.0185 (3)	
H20A	0.7016	0.7356	0.8977	0.028*	
H20B	0.5314	0.7673	0.8838	0.028*	
H20C	0.7089	0.8856	0.9131	0.028*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0151 (5)	0.0152 (5)	0.0154 (5)	0.0047 (4)	0.0069 (4)	0.0059 (4)
N2	0.0165 (5)	0.0154 (5)	0.0138 (5)	0.0042 (4)	0.0078 (4)	0.0059 (4)
N3	0.0167 (5)	0.0171 (5)	0.0156 (5)	0.0054 (4)	0.0087 (4)	0.0080 (4)
O1	0.0339 (6)	0.0148 (5)	0.0214 (5)	0.0074 (4)	0.0139 (4)	0.0095 (4)
O2	0.0152 (5)	0.0207 (5)	0.0139 (4)	0.0021 (4)	0.0061 (4)	0.0093 (4)
O3	0.0235 (5)	0.0176 (5)	0.0206 (5)	0.0070 (4)	0.0147 (4)	0.0077 (4)
C1	0.0134 (11)	0.0112 (15)	0.0170 (6)	0.0034 (6)	0.0072 (6)	0.0057 (6)
S1	0.01419 (19)	0.01807 (19)	0.01687 (18)	0.00243 (14)	0.00642 (14)	0.00913 (14)
C2	0.0158 (7)	0.0180 (8)	0.0178 (7)	0.0032 (6)	0.0091 (6)	0.0086 (6)
C3	0.0152 (7)	0.0161 (7)	0.0178 (7)	0.0070 (6)	0.0078 (6)	0.0069 (6)
C4	0.0197 (8)	0.0183 (10)	0.0186 (7)	0.0045 (8)	0.0098 (6)	0.0051 (7)
C5	0.0192 (7)	0.0194 (9)	0.0169 (7)	0.0056 (7)	0.0054 (6)	0.0051 (6)
C6	0.0139 (7)	0.0159 (7)	0.0197 (9)	0.0032 (5)	0.0030 (7)	0.0059 (8)
C7	0.0123 (8)	0.0167 (8)	0.0219 (10)	0.0039 (7)	0.0043 (7)	0.0096 (8)
C8	0.0138 (10)	0.0135 (11)	0.0179 (7)	0.0048 (7)	0.0057 (6)	0.0062 (7)
C1′	0.0134 (11)	0.0112 (15)	0.0170 (6)	0.0034 (6)	0.0072 (6)	0.0057 (6)
S1′	0.0158 (7)	0.0180 (8)	0.0178 (7)	0.0032 (6)	0.0091 (6)	0.0086 (6)

C2′	0.01419 (19)	0.01807 (19)	0.01687 (18)	0.00243 (14)	0.00642 (14)	0.00913 (14)
C3′	0.0138 (10)	0.0135 (11)	0.0179 (7)	0.0048 (7)	0.0057 (6)	0.0062 (7)
C4′	0.0123 (8)	0.0167 (8)	0.0219 (10)	0.0039 (7)	0.0043 (7)	0.0096 (8)
C5′	0.0139 (7)	0.0159 (7)	0.0197 (9)	0.0032 (5)	0.0030 (7)	0.0059 (8)
C6′	0.0192 (7)	0.0194 (9)	0.0169 (7)	0.0056 (7)	0.0054 (6)	0.0051 (6)
C7′	0.0197 (8)	0.0183 (10)	0.0186 (7)	0.0045 (8)	0.0098 (6)	0.0051 (7)
C8′	0.0152 (7)	0.0161 (7)	0.0178 (7)	0.0070 (6)	0.0078 (6)	0.0069 (6)
C9	0.0155 (6)	0.0149 (6)	0.0143 (6)	0.0065 (5)	0.0072 (5)	0.0073 (5)
C10	0.0139 (6)	0.0148 (6)	0.0145 (6)	0.0059 (5)	0.0067 (5)	0.0062 (5)
C11	0.0129 (6)	0.0168 (6)	0.0139 (6)	0.0042 (5)	0.0055 (5)	0.0074 (5)
C12	0.0168 (6)	0.0161 (6)	0.0143 (6)	0.0053 (5)	0.0070 (5)	0.0057 (5)
C13	0.0161 (6)	0.0145 (6)	0.0161 (6)	0.0038 (5)	0.0055 (5)	0.0075 (5)
C14	0.0122 (6)	0.0179 (6)	0.0123 (6)	0.0027 (5)	0.0045 (5)	0.0076 (5)
C15	0.0126 (6)	0.0171 (6)	0.0129 (6)	0.0039 (5)	0.0049 (5)	0.0048 (5)
C16	0.0144 (6)	0.0149 (6)	0.0162 (6)	0.0040 (5)	0.0068 (5)	0.0070 (5)
C17	0.0271 (7)	0.0144 (6)	0.0252 (7)	0.0079 (6)	0.0121 (6)	0.0066 (6)
C18	0.0161 (6)	0.0214 (7)	0.0201 (7)	0.0034 (5)	0.0086 (5)	0.0094 (6)
C19	0.0295 (8)	0.0184 (7)	0.0257 (7)	0.0106 (6)	0.0178 (6)	0.0085 (6)
C20	0.0225 (7)	0.0193 (6)	0.0126 (6)	0.0070 (5)	0.0089 (5)	0.0063 (5)

Geometric parameters (Å, º)

N1—N2	1.3266 (15)	C3′—C4′	1.383 (17)
N1—C9	1.3477 (16)	C3′—C8′	1.413 (17)
N2—N3	1.3279 (15)	C4′—C5′	1.346 (17)
N2—C20	1.4527 (16)	C4′—H4′	0.9500
N3—C10	1.3450 (16)	C5′—C6′	1.393 (18)
O1—C13	1.3720 (15)	C5′—H5′	0.9500
O1—C17	1.4210 (17)	C6′—C7′	1.385 (18)
O2—C14	1.3721 (14)	C6′—H6′	0.9500
O2—C18	1.4398 (16)	C7′—C8′	1.405 (18)
O3—C15	1.3666 (15)	C7′—H7′	0.9500
O3—C19	1.4340 (16)	C9—C10	1.4122 (17)
C1—C2	1.347 (3)	C10—C11	1.4723 (17)
C1—C9	1.466 (2)	C11—C16	1.3943 (18)
C1—S1	1.742 (2)	C11—C12	1.3956 (18)
S1—C8	1.7380 (17)	C12—C13	1.3930 (18)
C2—C3	1.429 (2)	C12—H12	0.9500
C2—H2	0.9500	C13—C14	1.3911 (18)
C3—C8	1.409 (2)	C14—C15	1.4018 (18)
C3—C4	1.410 (2)	C15—C16	1.3926 (17)
C4—C5	1.384 (2)	C16—H16	0.9500
C4—H4	0.9500	C17—H17A	0.9800
C5—C6	1.404 (2)	C17—H17B	0.9800
C5—H5	0.9500	C17—H17C	0.9800
C6—C7	1.383 (2)	C18—H18A	0.9800
C6—H6	0.9500	C18—H18B	0.9800
C7—C8	1.397 (2)	C18—H18C	0.9800

C7—H7	0.9500	C19—H19A	0.9800
C1′—C2′	1.318 (19)	C19—H19B	0.9800
C1′—C9	1.32 (2)	C19—H19C	0.9800
C1′—S1′	1.74 (2)	C20—H20A	0.9800
S1′—C8′	1.731 (18)	C20—H20B	0.9800
C2′—C3′	1.439 (17)	C20—H20C	0.9800
C2′—H2′	0.9500		

N2—N1—C9	103.78 (10)	C8′—C7′—H7′	120.8
N1—N2—N3	115.92 (10)	C7′—C8′—C3′	119 (2)
N1—N2—C20	122.69 (11)	C7′—C8′—S1′	129 (2)
N3—N2—C20	121.27 (11)	C3′—C8′—S1′	106.9 (14)
N2—N3—C10	104.04 (10)	C1′—C9—N1	123.7 (13)
C13—O1—C17	116.24 (10)	C1′—C9—C10	127.3 (11)
C14—O2—C18	113.82 (10)	N1—C9—C10	108.26 (11)
C15—O3—C19	116.41 (10)	N1—C9—C1	118.94 (13)
C2—C1—C9	129.94 (17)	C10—C9—C1	132.41 (13)
C2—C1—S1	112.33 (14)	N3—C10—C9	108.00 (11)
C9—C1—S1	117.55 (13)	N3—C10—C11	119.08 (11)
C8—S1—C1	91.33 (8)	C9—C10—C11	132.90 (12)
C1—C2—C3	113.73 (19)	C16—C11—C12	120.79 (11)
C1—C2—H2	123.1	C16—C11—C10	120.52 (11)
C3—C2—H2	123.1	C12—C11—C10	118.61 (11)
C8—C3—C4	118.66 (16)	C13—C12—C11	119.45 (12)
C8—C3—C2	111.50 (15)	C13—C12—H12	120.3
C4—C3—C2	129.79 (17)	C11—C12—H12	120.3
C5—C4—C3	119.15 (16)	O1—C13—C14	116.02 (11)
C5—C4—H4	120.4	O1—C13—C12	123.56 (12)
C3—C4—H4	120.4	C14—C13—C12	120.40 (12)
C4—C5—C6	121.14 (16)	O2—C14—C13	120.25 (11)
C4—C5—H5	119.4	O2—C14—C15	120.12 (11)
C6—C5—H5	119.4	C13—C14—C15	119.60 (11)
C7—C6—C5	120.88 (16)	O3—C15—C16	124.29 (12)
C7—C6—H6	119.6	O3—C15—C14	115.29 (11)
C5—C6—H6	119.6	C16—C15—C14	120.40 (12)
C6—C7—C8	118.04 (16)	C15—C16—C11	119.24 (12)
C6—C7—H7	121.0	C15—C16—H16	120.4
C8—C7—H7	121.0	C11—C16—H16	120.4
C7—C8—C3	122.13 (15)	O1—C17—H17A	109.5
C7—C8—S1	126.75 (13)	O1—C17—H17B	109.5
C3—C8—S1	111.11 (11)	H17A—C17—H17B	109.5
C2′—C1′—C9	124.6 (18)	O1—C17—H17C	109.5
C2′—C1′—S1′	107.3 (15)	H17A—C17—H17C	109.5
C9—C1′—S1′	128.0 (18)	H17B—C17—H17C	109.5
C8′—S1′—C1′	95.8 (12)	O2—C18—H18A	109.5
C1′—C2′—C3′	117.3 (18)	O2—C18—H18B	109.5
C1′—C2′—H2′	121.4	H18A—C18—H18B	109.5
C3′—C2′—H2′	121.4	O2—C18—H18C	109.5

C4′—C3′—C8′	115.8 (17)	H18A—C18—H18C	109.5
C4′—C3′—C2′	132 (2)	H18B—C18—H18C	109.5
C8′—C3′—C2′	111.8 (16)	O3—C19—H19A	109.5
C5′—C4′—C3′	122 (2)	O3—C19—H19B	109.5
C5′—C4′—H4′	118.9	H19A—C19—H19B	109.5
C3′—C4′—H4′	118.9	O3—C19—H19C	109.5
C4′—C5′—C6′	120 (2)	H19A—C19—H19C	109.5
C4′—C5′—H5′	120.2	H19B—C19—H19C	109.5
C6′—C5′—H5′	120.2	N2—C20—H20A	109.5
C7′—C6′—C5′	118 (2)	N2—C20—H20B	109.5
C7′—C6′—H6′	121.0	H20A—C20—H20B	109.5
C5′—C6′—H6′	121.0	N2—C20—H20C	109.5
C6′—C7′—C8′	118 (2)	H20A—C20—H20C	109.5
C6′—C7′—H7′	120.8	H20B—C20—H20C	109.5

C9—N1—N2—N3	−0.21 (14)	S1′—C1′—C9—C10	6 (12)
C9—N1—N2—C20	−176.39 (11)	S1′—C1′—C9—C1	−157 (59)
N1—N2—N3—C10	−0.26 (14)	N2—N1—C9—C1′	−170 (6)
C20—N2—N3—C10	175.99 (11)	N2—N1—C9—C10	0.57 (13)
C2—C1—S1—C8	0.6 (4)	N2—N1—C9—C1	−173.1 (3)
C9—C1—S1—C8	176.2 (4)	S1—C1—C9—C1′	−154 (51)
C9—C1—C2—C3	−175.7 (5)	C2—C1—C9—N1	173.7 (5)
S1—C1—C2—C3	−0.7 (5)	S1—C1—C9—N1	−1.1 (5)
C1—C2—C3—C8	0.5 (4)	C2—C1—C9—C10	1.8 (8)
C1—C2—C3—C4	177.8 (4)	S1—C1—C9—C10	−172.95 (18)
C8—C3—C4—C5	0.0 (4)	N2—N3—C10—C9	0.60 (13)
C2—C3—C4—C5	−177.2 (3)	N2—N3—C10—C11	179.11 (11)
C3—C4—C5—C6	−0.4 (3)	C1′—C9—C10—N3	170 (6)
C4—C5—C6—C7	0.1 (3)	N1—C9—C10—N3	−0.76 (14)
C5—C6—C7—C8	0.6 (3)	C1—C9—C10—N3	171.7 (4)
C6—C7—C8—C3	−1.1 (4)	C1′—C9—C10—C11	−9 (6)
C6—C7—C8—S1	177.16 (19)	N1—C9—C10—C11	−178.99 (13)
C4—C3—C8—C7	0.8 (4)	C1—C9—C10—C11	−6.5 (4)
C2—C3—C8—C7	178.4 (2)	N3—C10—C11—C16	129.29 (13)
C4—C3—C8—S1	−177.7 (2)	C9—C10—C11—C16	−52.6 (2)
C2—C3—C8—S1	−0.1 (3)	N3—C10—C11—C12	−47.72 (17)
C1—S1—C8—C7	−178.7 (3)	C9—C10—C11—C12	130.36 (15)
C1—S1—C8—C3	−0.3 (3)	C16—C11—C12—C13	2.02 (19)
C2′—C1′—S1′—C8′	9 (7)	C10—C11—C12—C13	179.01 (12)
C9—C1′—S1′—C8′	−176 (9)	C17—O1—C13—C14	−151.61 (12)
C9—C1′—C2′—C3′	175 (7)	C17—O1—C13—C12	29.78 (18)
S1′—C1′—C2′—C3′	−8 (8)	C11—C12—C13—O1	178.95 (12)
C1′—C2′—C3′—C4′	−180 (7)	C11—C12—C13—C14	0.40 (19)
C1′—C2′—C3′—C8′	4 (8)	C18—O2—C14—C13	82.87 (15)
C8′—C3′—C4′—C5′	−7 (7)	C18—O2—C14—C15	−99.29 (14)
C2′—C3′—C4′—C5′	177 (4)	O1—C13—C14—O2	−4.02 (18)
C3′—C4′—C5′—C6′	21 (6)	C12—C13—C14—O2	174.63 (11)
C4′—C5′—C6′—C7′	−11 (6)	O1—C13—C14—C15	178.13 (11)

C5′—C6′—C7′—C8′	−12 (7)	C12—C13—C14—C15	−3.22 (19)
C6′—C7′—C8′—C3′	27 (8)	C19—O3—C15—C16	−2.60 (18)
C6′—C7′—C8′—S1′	178 (4)	C19—O3—C15—C14	175.84 (11)
C4′—C3′—C8′—C7′	−17 (8)	O2—C14—C15—O3	7.32 (17)
C2′—C3′—C8′—C7′	160 (5)	C13—C14—C15—O3	−174.83 (11)
C4′—C3′—C8′—S1′	−174 (4)	O2—C14—C15—C16	−174.18 (11)
C2′—C3′—C8′—S1′	3 (6)	C13—C14—C15—C16	3.68 (19)
C1′—S1′—C8′—C7′	−160 (6)	O3—C15—C16—C11	177.06 (12)
C1′—S1′—C8′—C3′	−6 (6)	C14—C15—C16—C11	−1.30 (19)
C2′—C1′—C9—N1	−10 (12)	C12—C11—C16—C15	−1.56 (19)
S1′—C1′—C9—N1	174 (5)	C10—C11—C16—C15	−178.49 (12)
C2′—C1′—C9—C10	−179 (6)		