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

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The title compound, C_{19}H_{17}N_{3}O_{3}S (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_{3}I gave compound (II), C_{20}H_{19}N_{3}O_{3}S. 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 benzothioiophe 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_{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 analags 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_{3}I, resulting in compound (II).
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 tri-methoxyphenyl 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)].

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.

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

| Bond/Angle | Value (14) |
|------------|------------|
| N1—N2     | 1.324 (2)  |
| N1—C9     | 1.343 (2)  |
| N2—N3     | 1.330 (2)  |
| C8—S1—C1  | 91.50 (8)  |
| N2—N1—C9  | 103.74 (13)|
| N2—N3—C10 | 103.74 (13)|
| C4—C3—C2  | 129.50 (16)|

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

| Bond/Angle | Value (15) |
|------------|------------|
| N1—N2     | 1.3266 (15)|
| N1—C9     | 1.3477 (16)|
| N2—N3     | 1.3279 (15)|
| C2—C1—C9  | 115.92 (10)|
| C4—C3—C2  | 129.94 (17)|
| C8—S1—C1  | 91.33 (8)  |
| C4—C3—C2  | 129.79 (17)|
| C8—S1—C1  | 95.8 (12)  |

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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 R21(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).

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

| D—H···A    | D—H | H···A | D···A | D—H···A |
|------------|------|-------|-------|---------|
| N2—H2N—O2 | 0.87 | 2.16  | 2.9381| 147.6   |
| N2—H2N—O3 | 0.87 | 2.20  | 2.8503| 130.8   |

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

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.

Table 4
Experimental details.

|                  | (I)     | (II)    |
|------------------|---------|---------|
| Chemical formula | C19H17N3O3S | C20H19N3O3S |
| Crystal system   | Monoclinic, P21/n | Triclinic, P1 |
| 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) |
| V (Å³)           | 1732.39 (8) | 1065.89 (4), 111.668 (5), 105.498 (4) |
| Z                | 4       | 2       |
| Radiation type   | Mo Kα  | Mo Kα  |
| μ (mm⁻¹)         | 0.21    | 0.21    |
| Crystal size (mm)| 0.30 × 0.30 × 0.05 | 0.22 × 0.20 × 0.15 |
| Data collection  | Nonius KappaCCD | Nonius KappaCCD |
| Absorption correction | Multi-scan (SADABS; Sheldrick, 2008a) | Multi-scan (SADABS; Sheldrick, 2008a) |
| Tmin, Tmax      | 0.816, 0.966 | 0.858, 0.962 |
| No. of measured, independent and observed | 28105, 3984, 3093 | 36591, 4097, 3572 |
| Rint            | 0.045   | 0.045   |
| (sin θ/λ)max (Å⁻¹) | 0.650 | 0.651 |
| Refinement       |        |        |
| R(F² > 2σ(F²))   | 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 |
| Δρmax, Δρmin (e Å⁻³) | 0.55, −0.29 | 0.31, −0.28 |

Computer programs: COLLECT (Nonius, 1998), SCALEPACK and DENZO-SMN (Otwonowski & Minor, 2006), SHELXS97, SHELXL2013, SHELXL2014 and XP in SHELXTL (Sheldrick, 2008b) and CIFTIX (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 (RCH₃) and 0.95 Å (Csp²H). Coordinates of the N-bound hydrogen were refined freely. Uiso(H) values were set to either 1.2Ueq or 1.5Ueq (RCH₃) 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.

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

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**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)-2H-1,2,3-triazole

**Crystal data**

- **Formula:** C_{19}H_{17}N_{3}O_{3}S
- **Mr:** 367.41
- **Space group:** P2_{1}/n
- **Cell parameters:**
  - **a:** 11.8983 (2) Å
  - **b:** 8.1860 (1) Å
  - **c:** 18.4582 (3) Å
  - **β:** 105.5046 (7)°
  - **Volume:** 1732.39 (5) Å³
- **Z:** 4

**Data collection**

- **Detector:** Nonius KappaCCD
- **Radiation source:** fine-focus sealed-tube
- **Detector resolution:** 9.1 pixels mm⁻¹
- **ω scans at fixed χ:** 55°
- **Absorption correction:** multi-scan (SADABS; Sheldrick, 2008a)
- **θmax:** 27.5°, θmin:** 1.8°
- **h:** -15→15
- **k:** -10→10
- **l:** -23→22
- **28105 measured reflections**
- **3984 independent reflections**
- **Rint:** 0.045
- **θmax:** 27.5°, θmin:** 1.8°

**Reference**

*Acta Cryst.* (2014). E70, 392-395 [doi:10.1107/S1600536814023095]
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 ($\AA^2$)

|    | x     | y     | z     | $U_{iso}$/Ueq |
|----|-------|-------|-------|---------------|
| 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) |
supporting information

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)  0.5777 (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 (Å\(^2\))

|   | U\(^{11}\) | U\(^{22}\) | U\(^{33}\) | U\(^{12}\) | U\(^{13}\) | U\(^{23}\) |
|---|----------|----------|----------|----------|----------|----------|
| S1| 0.0206 (2) | 0.0238 (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 (7) | 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) |

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|       |          |          |          |          |          |          |          |          |
|-------|----------|----------|----------|----------|----------|----------|----------|----------|
| 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, °)**

| Bond         | Distance   | Angle       | Distance   | Angle       | Distance   | Angle       |
|--------------|------------|-------------|------------|-------------|------------|-------------|
| S1—C8        | 1.7345 (17) | C6—C7       | 1.375 (3)  | C6—H6       | 0.9500     |
| S1—C1        | 1.7474 (17) | C6—C7       | 1.395 (2)  | C7—C8       | 1.416 (2)  |
| N1—N2        | 1.324 (2)   | C7—H7       | 0.9500     | C7—C8       | 1.416 (2)  |
| N1—C9        | 1.343 (2)   | C7—H7       | 0.9500     | C9—C10      | 1.471 (2)  |
| N2—N3        | 1.330 (2)   | C9—C10      | 1.395 (2)  | C9—C10      | 1.471 (2)  |
| N2—H2N       | 0.87 (2)    | C9—C10      | 1.395 (2)  | C10—C11     | 1.416 (2)  |
| N3—C10       | 1.345 (2)   | C10—C11     | 1.395 (2)  | C11—C16     | 1.471 (2)  |
| O1—C13       | 1.3660 (19) | C11—C12     | 1.404 (2)  | C12—C13     | 1.416 (2)  |
| O1—C17       | 1.4313 (19) | C12—C13     | 1.390 (2)  | C12—C13     | 1.416 (2)  |
| O2—C14       | 1.3829 (19) | C12—H12     | 0.9500     | C12—H12     | 0.9500     |
| O2—C18       | 1.439 (2)   | C13—C14     | 1.399 (2)  | C13—C14     | 1.399 (2)  |
| O3—C15       | 1.3712 (19) | C14—C15     | 1.394 (2)  | C14—C15     | 1.394 (2)  |
| O3—C19       | 1.4356 (19) | C15—C16     | 1.389 (2)  | C15—C16     | 1.389 (2)  |
| C1—C2        | 1.357 (2)   | C16—H16     | 0.9500     | C16—H16     | 0.9500     |
| C1—C9        | 1.460 (2)   | C17—H17A    | 0.9800     | C17—H17A    | 0.9800     |
| C2—C3        | 1.433 (2)   | C17—H17B    | 0.9800     | C17—H17B    | 0.9800     |
| C2—H2        | 0.9500     | C17—H17C    | 0.9800     | C17—H17C    | 0.9800     |
| C3—C4        | 1.402 (2)   | C18—H18A    | 0.9800     | C18—H18A    | 0.9800     |
| C3—C8        | 1.413 (2)   | C18—H18B    | 0.9800     | C18—H18B    | 0.9800     |
| C4—C5        | 1.381 (3)   | C18—H18C    | 0.9800     | C18—H18C    | 0.9800     |
| C4—H4        | 0.9500     | C19—H19A    | 0.9800     | C19—H19A    | 0.9800     |
| C5—C6        | 1.397 (3)   | C19—H19B    | 0.9800     | C19—H19B    | 0.9800     |
| C5—H5        | 0.9500     | C19—H19C    | 0.9800     | C19—H19C    | 0.9800     |

| Angle       | Distance   | Angle       | Distance   | Angle       | Distance   | Angle       |
|-------------|------------|-------------|------------|-------------|------------|-------------|
| C8—S1—C1    | 91.50 (8)  | C9—C10—C11 | 131.16 (14)| C9—C10—C11 | 131.16 (14)|
| N2—N1—C9    | 103.74 (13)| C16—C11—C12| 120.15 (15)| C16—C11—C12| 120.15 (15)|
| N1—N2—N3    | 116.21 (14)| C16—C11—C10| 119.01 (14)| C16—C11—C10| 119.01 (14)|
| N1—N2—H2N   | 120.6 (13) | C12—C11—C10| 120.77 (14)| C12—C11—C10| 120.77 (14)|
| N3—N2—H2N   | 123.2 (13) | C13—C12—C11| 119.68 (15)| C13—C12—C11| 119.68 (15)|
| N2—N3—C10   | 103.74 (13)| C13—C12—H12| 120.2      | C13—C12—H12| 120.2      |
| C13—O1—C17  | 117.13 (13)| C11—C12—H12| 120.2      | C11—C12—H12| 120.2      |
| C14—O2—C18  | 113.13 (13)| O1—C13—C12 | 124.98 (14)| O1—C13—C12 | 124.98 (14)|
| C15—O3—C19  | 116.85 (12)| O1—C13—C14 | 114.89 (14)| O1—C13—C14 | 114.89 (14)|
| C2—C1—C9    | 129.19 (15)| C12—C13—C14| 120.12 (15)| C12—C13—C14| 120.12 (15)|
| C2—C1—S1    | 112.12 (12)| O2—C14—C15 | 118.70 (14)| O2—C14—C15 | 118.70 (14)|
| C9—C1—S1    | 118.67 (12)| O2—C14—C13 | 121.49 (14)| O2—C14—C13 | 121.49 (14)|
| C1—C2—C3    | 113.49 (15)| C15—C14—C13| 119.72 (14)| C15—C14—C13| 119.72 (14)|
| C1—C2—H2    | 123.3      | O3—C15—C16 | 124.11 (15)| O3—C15—C16 | 124.11 (15)|
| C3—C2—H2    | 123.3      | O3—C15—C14 | 115.35 (14)| O3—C15—C14 | 115.35 (14)|
| C4—C3—C8    | 118.91 (16)| C16—C15—C14| 120.54 (14)| C16—C15—C14| 120.54 (14)|
| C4—C3—C2    | 129.50 (16)| C15—C16—C11| 119.66 (15)| C15—C16—C11| 119.66 (15)|
| Bond                        | Distance (Å) | Bond                        | Distance (Å) |
|-----------------------------|--------------|-----------------------------|--------------|
| 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        | 02—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)  |
Hydrogen-bond geometry (Å, °)

| D—H···A        | D—H  | H···A  | D···A    | D—H···A |
|----------------|-------|--------|----------|---------|
| N2—H2N···O2i   | 0.87 (2) | 2.16 (2) | 2.9381 (18) | 147.6 (18) |
| N2—H2N···O3i   | 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)-2H-1,2,3-triazole

Crystal data

C20H19N3O3S  
Z = 2  
F(000) = 400  
Dc = 1.421 Mg m−3  
Mo Kα radiation, λ = 0.71073 Å  
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  
θ and ω scans at fixed χ = 55°  
Absorption correction: multi-scan (SADABS; Sheldrick, 2008a)  
Tmin = 0.858, Tmax = 0.962  
3572 reflections with I > 2σ(I)  
36591 measured reflections  
4097 independent reflections  
h = −11→11  
k = −14→14  
l = −14→14

Refinement

Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.037  
wR(F²) = 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/[σ²(F²) + (0.0472P)² + 0.4023P]  
where P = (F² + 2Fc²)/3  
(Δσ)max = 0.001  
Δρmax = 0.31 e Å⁻³  
Δρmin = −0.28 e Å⁻³
**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       | Ueq     | 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) |
| S1  | 0.08551 (5)  | 0.39081 (4)  | 0.37446 (4)  | 0.01741 (12) |
| C2  | 0.2034 (2)   | 0.48053 (18) | 0.22483 (17) | 0.0176 (3) |
| H2  | 0.2776      | 0.5336      | 0.1986      | 0.021* |
| C3  | 0.0313 (3)   | 0.3687 (3)   | 0.1242 (2)   | 0.0164 (4) |
| C4  | -0.0638 (3)  | 0.31782 (16) | 0.0125 (2)   | 0.0203 (4) |
| H4  | -0.0517     | 0.3565      | 0.0215 (2)   | 0.024* |
| C5  | -0.2341 (3)  | 0.21078 (18) | -0.09952 (19)| 0.0210 (4) |
| H5  | -0.2990     | 0.1766      | 0.025*       | |
| C6  | -0.3125 (2)  | 0.1519 (2)   | -0.03024 (16)| 0.0195 (4) |
| H6  | -0.4295     | 0.0784      | 0.023*       | |
| C7  | -0.2218 (2)  | 0.19944 (19) | 0.11543 (18) | 0.0186 (4) |
| H7  | -0.2742     | 0.1591      | 0.1624       | 0.022* |
| C8  | -0.0508 (2)  | 0.3086 (2)   | 0.19147 (18) | 0.0161 (3) |
| C1' | 0.269 (8)    | 0.522 (7)    | 0.368 (2)    | 0.0145 (4) |
| S1' | 0.2341 (9)   | 0.5088 (7)   | 0.2013 (7)   | 0.0176 (3) |
| C2' | 0.134 (3)    | 0.418 (2)    | 0.3485 (19)  | 0.01741 (12) |
| H2' | 0.1347      | 0.3980      | 0.4251       | 0.021* |
| C3' | -0.013 (4)   | 0.335 (4)    | 0.207 (2)    | 0.0161 (3) |
| C4' | -0.173 (3)   | 0.221 (3)    | 0.150 (3)    | 0.0186 (4) |
| H4' | -0.1949     | 0.1828      | 0.2106       | 0.022* |
| C5' | -0.297 (4)   | 0.162 (3)    | 0.013 (3)    | 0.0195 (4) |
| H5' | -0.4184     | 0.1070      | -0.0167      | 0.023* |
| C6' | -0.248 (4)   | 0.183 (3)    | -0.086 (3)   | 0.0210 (4) |
| H6' | -0.3283     | 0.1303      | -0.1851      | 0.025* |
| C7' | -0.079 (4)   | 0.284 (3)    | -0.036 (3)   | 0.0203 (4) |
| H7' | -0.0314     | 0.2892      | -0.0985      | 0.024* |
### Supporting Information

| 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) |           |
| 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) |

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Geometric parameters (Å, °)

| Bond         | Distance (Å) | Angle (°) |
|--------------|--------------|-----------|
| 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     |
| Bond             | Length (Å) | Bond             | Length (Å) |
|------------------|------------|------------------|------------|
| 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 (3)  |
| 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)| C3—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      |
| Bond | Angle[^2] | Torsion[^2] |
|------|-----------|-------------|
| 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 | N2—C20—H20A | 109.5 |
| C6′—C5′—H5′ | 118 (2) | N2—C20—H20B | 109.5 |
| C7′—C6′—C5′ | 121.0 | H20A—C20—H20B | 109.5 |
| C7′—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 |

[^2]: Sup-11
| Bond                        | Value (°) |
|-----------------------------|-----------|
| C5′—C6′—C7′—C8′            | -12 (7)   |
| C6′—C7′—C8′—C3′            | 27 (8)    |
| C6′—C7′—C8′—S1′            | 178 (4)   |
| C4′—C3′—C8′—C7′            | -17 (8)   |
| C2′—C3′—C8′—C7′            | 160 (5)   |
| C4′—C3′—C8′—S1′            | -174 (4)  |
| C2′—C3′—C8′—S1′            | 3 (6)     |
| C1′—S1′—C8′—C7′            | -160 (6)  |
| C1′—S1′—C8′—C3′            | -6 (6)    |
| C2′—C1′—C9—N1              | -10 (12)  |
| S1′—C1′—C9—N1              | 174 (5)   |
| C2′—C1′—C9—C10             | -179 (6)  |
| C12—C13—C14—C15           | -3.22 (19) |
| C19—O3—C15—C16            | -2.60 (18) |
| C19—O3—C15—C14            | 175.84 (11) |
| O2—C14—C15—O3             | 7.32 (17)  |
| C13—C14—C15—O3            | -174.83 (11) |
| O2—C14—C15—C16            | -174.18 (11) |
| C13—C14—C15—C16           | 3.68 (19)  |
| O3—C15—C16—C11            | 177.06 (12) |
| C14—C15—C16—C11           | -1.30 (19)  |
| C12—C11—C16—C15           | -1.56 (19)  |
| C10—C11—C16—C15           | -178.49 (12) |