Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 2-Amino-6-methyl-4,5,6,7-tetrahydro-1-benzothiophene-3-carbonitrile

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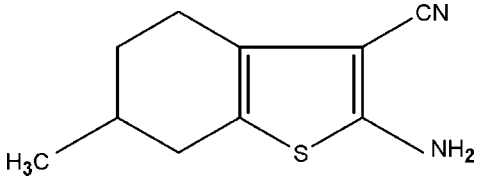
Received 25 January 2011; accepted 17 February 2011

Key indicators: single-crystal X-ray study; $T$ = 123 K; mean $\sigma$(C–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.040; $wR$ factor = 0.107; data-to-parameter ratio = 19.2.

In the title compound, $C_{10}H_{12}N_2S$, one of the C atoms of the cyclohexene ring (at position 6) and the methyl group attached to it are disordered over two sets of sites in a 0.650 (3):0.350 (3) ratio. The cyclohexene ring in both the major and minor occupancy conformers adopts a half-chair conformation. The thiophene ring is essentially planar (r.m.s. deviation = 0.05 Å). In the crystal, N—H···N hydrogen bonds involving the amino groups result in inversion dimers with $R_2^2(12)$ graph-set motif. Further N—H···N hydrogen bonds involving the amino and carbonitrile groups generate zigzag chains along the $a$ axis.

## Related literature

For preparation of the title compound, see: Shetty *et al.* (2009). For general background to benzothiophenes, see: Katritzky *et al.* (1996); Shishoo & Jain (1992). For related structures, see: Akkurt *et al.* (2008); Harrison *et al.* (2006); Vasu *et al.* (2004). For graph-set notation, see: Bernstein *et al.* (1995).




## Experimental

### Crystal data

$C_{10}H_{12}N_2S$
$M_r$ = 192.29
Monoclinic, $P2_1/c$
$a$ = 9.0415 (2) Å
$b$ = 8.3294 (2) Å
$c$ = 13.1283 (3) Å
$\beta$ = 90.169 (2)°
$V$ = 988.69 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.28 mm$^{-1}$
$T$ = 123 K
0.16 × 0.16 × 0.14 mm

### Data collection

Bruker SMART APEX CCD detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min}$ = 0.957, $T_{max}$ = 0.962
11284 measured reflections
2441 independent reflections
1878 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
$S$ = 1.07
2441 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$A$···N1$^{i}$ | 0.88 | 2.22 | 3.087 (2) | 170 |
| N2—H2$B$···N1$^{ii}$ | 0.88 | 2.41 | 3.247 (2) | 160 |

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

NSB is thankful to the University Grants Commission (UGC), India, for financial assistance and the Department of Science and Technology, (DST), India, for the data collection facility under the IRHPA–DST program.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2384).

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# supporting information

*Acta Cryst.* (2011). E**67**, o699 [doi:10.1107/S1600536811006076]

## 2-Amino-6-methyl-4,5,6,7-tetrahydro-1-benzothiophene-3-carbonitrile

**Mohamed Ziaulla, Afshan Banu, Noor Shahina Begum, Shridhar I. Panchamukhi and I. M. Khazi**

### S1. Comment

Benzothiophenes are important heterocycles either as biological active molecules or as luminescent components used in organic materials (Shishoo & Jain, 1992; Katritzky *et al.*, 1996). In this paper, we report the crystal structure of a benzothiophene derivative.

In the title compound (Fig. 1), the fused benzothiophene ring system is substituted with amino, methyl and carbonitrile groups. The carbon atoms C9 and C10 are disordered over two sites (C9A/C9B and C10A/C10B) with site occupancy factors 0.650 (3) and 0.350 (3) resulting in a major and a minor conformers. The cyclohexene ring in both conformers is in a half-chair conformation with C9A and C9B 0.547 (4) and 0.506 (6) Å, respectively, displaced on the opposite sides from the plane formed by the rest of the ring C-atoms (max. deviation being 0.063 (2) Å for C6). The thiophene ring is essentially planar. In several benzothiophene derivatives the cyclohexyl ring adopts half-chair conformation (Akkurt *et al.*, 2008; Harrison *et al.*, 2006; Vasu *et al.*, 2004). The crystal structure is stabilized by two types of N—H···N intermolecular interactions (Table 1); N2—H2A···N1 hydrogen bond forms centrosymmetric, head-to-head dimers about inversion centers corresponding to graph set $R^2_2(12)$ motif(Bernstein *et al.*, 1995) while N2—H2B···N1 hydrogen bonds generate chains of molecules in a zigzag pattern along the *a* axis (Fig. 2).

### S2. Experimental

The title compound was synthesized by following the procedure reported earlier (Shetty *et al.*, 2009).

### S3. Refinement

The H atoms were placed at calculated positions in the riding model approximation with N—H = 0.88 Å and C—H = 0.98, 0.99 and 1.00 Å for methylene, methyl and methyne type H-atoms, respectively; $U_{iso}$(H) = 1.2$U_{eq}$(N/non-methyl C) and 1.5$U_{eq}$(methyl C).

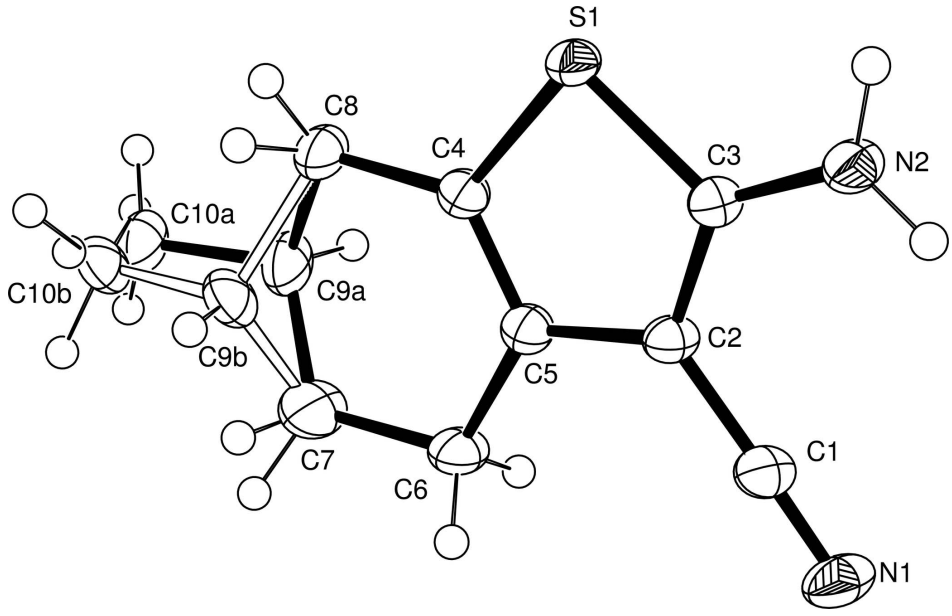


**Figure 1**

*ORTEP* (Farrugia, 1999) view of the title compound, showing 50% probability ellipsoids and the atom numbering scheme; C-atoms C9b and C10b represent the minor conformer.

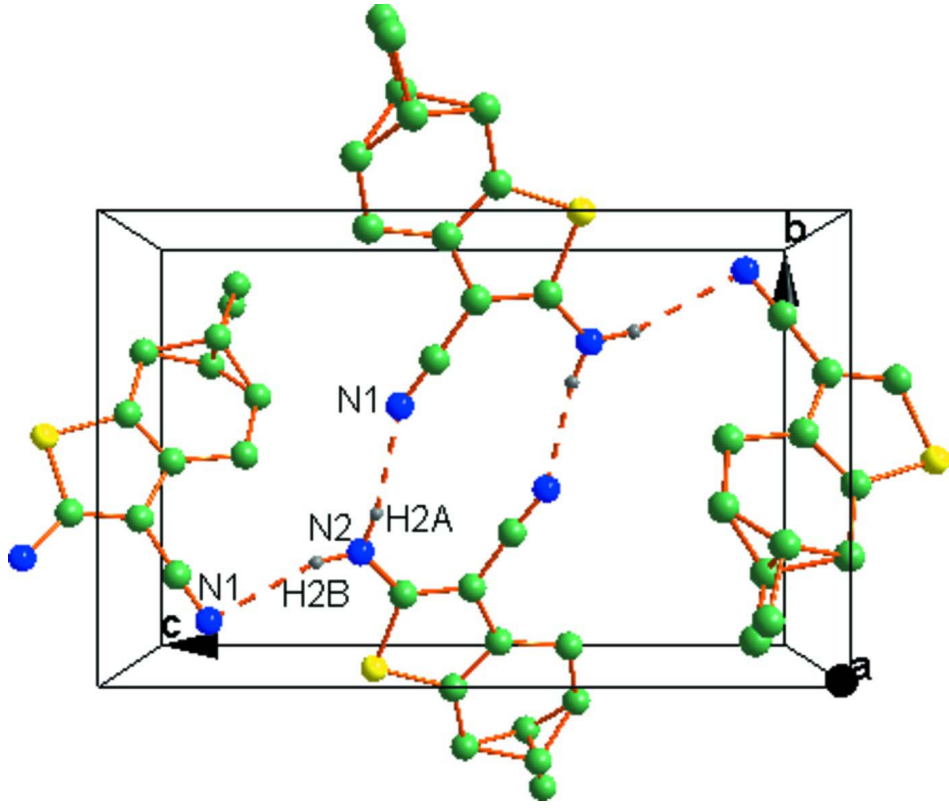


**Figure 2**

A unit cell packing of the title compound showing intermolecular interactions with dotted lines. H-atoms not involved in hydrogen bonding have been excluded.

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*Crystal data*

$C_{10}H_{12}N_2S$
$M_r = 192.29$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.0415$ (2) Å
$b = 8.3294$ (2) Å
$c = 13.1283$ (3) Å
$\beta = 90.169$ (2)°
$V = 988.69$ (4) Å$^3$
$Z = 4$
$F(000) = 408$
$D_x = 1.292$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2441 reflections
$\theta = 2.9$–$29.2°$
$\mu = 0.28$ mm$^{-1}$
$T = 123$ K
Block, yellow
0.16 × 0.16 × 0.14 mm

*Data collection*

Bruker SMART APEX CCD detector diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.957$, $T_{max} = 0.962$
11284 measured reflections
2441 independent reflections
1878 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 29.2°$, $\theta_{min} = 2.9°$
$h = -11 \rightarrow 12$
$k = -11 \rightarrow 10$
$l = -17 \rightarrow 16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.107$
$S = 1.07$
2441 reflections
127 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.2028P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å$^{-3}$
$\Delta\rho_{min} = -0.30$ e Å$^{-3}$

*Special details*

**Experimental**. The compound was synthesized by following the procedure given in NitinKumar *et al.*, (2009)
**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.
**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S1 | 0.68698 (5) | 0.02703 (5) | 0.34885 (3) | 0.02519 (15) | |
| N1 | 0.5603 (2) | −0.40456 (18) | 0.60740 (11) | 0.0378 (4) | |
| N2 | 0.54766 (17) | −0.25706 (17) | 0.33230 (10) | 0.0292 (4) | |

| | | | | | |
|---|---|---|---|---|---|
| H2A | 0.5129 | −0.3480 | 0.3565 | 0.035* | |
| H2B | 0.5362 | −0.2336 | 0.2674 | 0.035* | |
| C1 | 0.5991 (2) | −0.3000 (2) | 0.55684 (12) | 0.0263 (4) | |
| C2 | 0.64719 (19) | −0.16794 (19) | 0.49726 (12) | 0.0224 (4) | |
| C3 | 0.61829 (18) | −0.15372 (19) | 0.39455 (12) | 0.0217 (3) | |
| C4 | 0.75534 (19) | 0.0810 (2) | 0.46849 (12) | 0.0251 (4) | |
| C5 | 0.72696 (19) | −0.0328 (2) | 0.53858 (12) | 0.0236 (4) | |
| C6 | 0.7762 (2) | −0.0180 (2) | 0.64700 (13) | 0.0303 (4) | |
| H6A | 0.8312 | −0.1158 | 0.6670 | 0.036* | |
| H6B | 0.6885 | −0.0088 | 0.6916 | 0.036* | |
| C7 | 0.8734 (3) | 0.1266 (3) | 0.66129 (16) | 0.0514 (6) | |
| H7A | 0.8688 | 0.1572 | 0.7341 | 0.062* | 0.650 (3) |
| H7B | 0.9764 | 0.0931 | 0.6474 | 0.062* | 0.650 (3) |
| C8 | 0.8387 (2) | 0.2343 (2) | 0.48637 (13) | 0.0328 (4) | |
| H8A | 0.7892 | 0.3236 | 0.4500 | 0.039* | 0.650 (3) |
| H8B | 0.9405 | 0.2241 | 0.4596 | 0.039* | 0.650 (3) |
| C9A | 0.8440 (3) | 0.2705 (3) | 0.6012 (2) | 0.0296 (5) | 0.650 (3) |
| H9AA | 0.7434 | 0.3094 | 0.6209 | 0.036* | 0.650 (3) |
| C10A | 0.9518 (5) | 0.4053 (5) | 0.6248 (3) | 0.0429 (10) | 0.650 (3) |
| H10A | 0.9544 | 0.4241 | 0.6985 | 0.064* | 0.650 (3) |
| H10B | 0.9199 | 0.5034 | 0.5900 | 0.064* | 0.650 (3) |
| H10C | 1.0508 | 0.3753 | 0.6013 | 0.064* | 0.650 (3) |
| H7C | 0.8128 | 0.2072 | 0.6977 | 0.062* | 0.350 (3) |
| H7D | 0.9527 | 0.0937 | 0.7091 | 0.062* | 0.350 (3) |
| H8C | 0.7690 | 0.3238 | 0.4988 | 0.039* | 0.350 (3) |
| H8D | 0.8997 | 0.2611 | 0.4262 | 0.039* | 0.350 (3) |
| C9B | 0.9414 (6) | 0.2065 (6) | 0.5837 (4) | 0.0296 (5) | 0.350 (3) |
| H9BA | 1.0221 | 0.1334 | 0.5601 | 0.036* | 0.350 (3) |
| C10B | 1.0183 (9) | 0.3644 (11) | 0.6111 (7) | 0.0429 (10) | 0.350 (3) |
| H10D | 0.9449 | 0.4508 | 0.6140 | 0.064* | 0.350 (3) |
| H10E | 1.0924 | 0.3897 | 0.5592 | 0.064* | 0.350 (3) |
| H10F | 1.0669 | 0.3537 | 0.6775 | 0.064* | 0.350 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0327 (3) | 0.0257 (2) | 0.0171 (2) | −0.00706 (18) | −0.00294 (16) | 0.00388 (16) |
| N1 | 0.0684 (12) | 0.0241 (8) | 0.0207 (8) | −0.0089 (8) | −0.0066 (7) | 0.0026 (6) |
| N2 | 0.0453 (9) | 0.0247 (8) | 0.0175 (7) | −0.0093 (7) | −0.0035 (6) | 0.0003 (6) |
| C1 | 0.0395 (10) | 0.0207 (8) | 0.0187 (8) | −0.0005 (7) | −0.0035 (7) | −0.0027 (7) |
| C2 | 0.0287 (9) | 0.0203 (8) | 0.0183 (8) | 0.0000 (7) | −0.0001 (6) | 0.0010 (6) |
| C3 | 0.0241 (8) | 0.0207 (8) | 0.0203 (8) | −0.0003 (6) | 0.0011 (6) | 0.0004 (6) |
| C4 | 0.0296 (9) | 0.0270 (9) | 0.0185 (8) | −0.0053 (7) | −0.0033 (7) | 0.0010 (7) |
| C5 | 0.0263 (9) | 0.0243 (8) | 0.0202 (8) | −0.0017 (7) | −0.0011 (7) | 0.0004 (7) |
| C6 | 0.0427 (11) | 0.0294 (9) | 0.0189 (9) | −0.0041 (8) | −0.0057 (7) | 0.0030 (7) |
| C7 | 0.0744 (17) | 0.0471 (13) | 0.0327 (11) | −0.0244 (12) | −0.0248 (11) | 0.0067 (9) |
| C8 | 0.0418 (11) | 0.0330 (10) | 0.0237 (9) | −0.0159 (8) | −0.0040 (8) | 0.0028 (7) |
| C9A | 0.0321 (14) | 0.0302 (13) | 0.0265 (12) | −0.0074 (10) | −0.0027 (11) | −0.0028 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10A | 0.047 (3) | 0.052 (2) | 0.0294 (16) | −0.029 (2) | −0.0007 (19) | −0.0052 (15) |
| C7A | 0.0744 (17) | 0.0471 (13) | 0.0327 (11) | −0.0244 (12) | −0.0248 (11) | 0.0067 (9) |
| C8A | 0.0418 (11) | 0.0330 (10) | 0.0237 (9) | −0.0159 (8) | −0.0040 (8) | 0.0028 (7) |
| C9B | 0.0321 (14) | 0.0302 (13) | 0.0265 (12) | −0.0074 (10) | −0.0027 (11) | −0.0028 (10) |
| C10B | 0.047 (3) | 0.052 (2) | 0.0294 (16) | −0.029 (2) | −0.0007 (19) | −0.0052 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C3 | 1.7363 (16) | C7—H7A | 0.9900 |
| S1—C4 | 1.7451 (17) | C7—H7B | 0.9900 |
| N1—C1 | 1.150 (2) | C8—C9A | 1.538 (3) |
| N2—C3 | 1.347 (2) | C8—H8A | 0.9900 |
| N2—H2A | 0.8800 | C8—H8B | 0.9900 |
| N2—H2B | 0.8800 | C9A—C10A | 1.519 (5) |
| C1—C2 | 1.419 (2) | C9A—H9AA | 1.0000 |
| C2—C3 | 1.378 (2) | C10A—H10A | 0.9800 |
| C2—C5 | 1.442 (2) | C10A—H10B | 0.9800 |
| C4—C5 | 1.346 (2) | C10A—H10C | 0.9800 |
| C4—C8 | 1.501 (2) | C9B—C10B | 1.530 (10) |
| C5—C6 | 1.495 (2) | C9B—H9BA | 1.0000 |
| C6—C7 | 1.502 (3) | C10B—H10D | 0.9800 |
| C6—H6A | 0.9900 | C10B—H10E | 0.9800 |
| C6—H6B | 0.9900 | C10B—H10F | 0.9800 |
| C7—C9A | 1.459 (3) | | |
| | | | |
| C3—S1—C4 | 92.20 (8) | C9A—C7—H7A | 107.6 |
| C3—N2—H2A | 120.0 | C6—C7—H7A | 107.6 |
| C3—N2—H2B | 120.0 | C9A—C7—H7B | 107.6 |
| H2A—N2—H2B | 120.0 | C6—C7—H7B | 107.6 |
| N1—C1—C2 | 178.19 (17) | H7A—C7—H7B | 107.0 |
| C3—C2—C1 | 123.28 (15) | C4—C8—C9A | 109.52 (16) |
| C3—C2—C5 | 113.22 (14) | C4—C8—H8A | 109.8 |
| C1—C2—C5 | 123.47 (14) | C9A—C8—H8A | 109.8 |
| N2—C3—C2 | 128.84 (15) | C4—C8—H8B | 109.8 |
| N2—C3—S1 | 120.93 (12) | C9A—C8—H8B | 109.8 |
| C2—C3—S1 | 110.22 (12) | H8A—C8—H8B | 108.2 |
| C5—C4—C8 | 126.07 (15) | C7—C9A—C10A | 112.4 (3) |
| C5—C4—S1 | 111.48 (13) | C7—C9A—C8 | 112.0 (2) |
| C8—C4—S1 | 122.42 (12) | C10A—C9A—C8 | 111.3 (2) |
| C4—C5—C2 | 112.86 (15) | C7—C9A—H9AA | 106.9 |
| C4—C5—C6 | 122.40 (15) | C10A—C9A—H9AA | 106.9 |
| C2—C5—C6 | 124.73 (15) | C8—C9A—H9AA | 106.9 |
| C5—C6—C7 | 110.93 (15) | C10B—C9B—H9BA | 105.4 |
| C5—C6—H6A | 109.5 | C9B—C10B—H10D | 109.5 |
| C7—C6—H6A | 109.5 | C9B—C10B—H10E | 109.5 |
| C5—C6—H6B | 109.5 | H10D—C10B—H10E | 109.5 |
| C7—C6—H6B | 109.5 | C9B—C10B—H10F | 109.5 |
| H6A—C6—H6B | 108.0 | H10D—C10B—H10F | 109.5 |

| | | | |
|---|---|---|---|
| C9A—C7—C6 | 119.06 (19) | H10E—C10B—H10F | 109.5 |
| C1—C2—C3—N2 | −2.3 (3) | C1—C2—C5—C4 | −177.32 (17) |
| C5—C2—C3—N2 | 179.55 (17) | C3—C2—C5—C6 | −178.44 (17) |
| C1—C2—C3—S1 | 177.36 (14) | C1—C2—C5—C6 | 3.4 (3) |
| C5—C2—C3—S1 | −0.78 (19) | C4—C5—C6—C7 | −6.7 (3) |
| C4—S1—C3—N2 | −179.86 (15) | C2—C5—C6—C7 | 172.46 (19) |
| C4—S1—C3—C2 | 0.45 (13) | C5—C6—C7—C9A | 34.4 (3) |
| C3—S1—C4—C5 | 0.01 (14) | C5—C4—C8—C9A | −18.3 (3) |
| C3—S1—C4—C8 | 178.33 (16) | S1—C4—C8—C9A | 163.68 (16) |
| C8—C4—C5—C2 | −178.70 (17) | C6—C7—C9A—C10A | 179.9 (3) |
| S1—C4—C5—C2 | −0.5 (2) | C6—C7—C9A—C8 | −53.9 (3) |
| C8—C4—C5—C6 | 0.6 (3) | C4—C8—C9A—C7 | 42.0 (3) |
| S1—C4—C5—C6 | 178.83 (14) | C4—C8—C9A—C10A | 168.8 (3) |
| C3—C2—C5—C4 | 0.8 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···N1$^{i}$ | 0.88 | 2.22 | 3.087 (2) | 170 |
| N2—H2*B*···N1$^{ii}$ | 0.88 | 2.41 | 3.247 (2) | 160 |

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