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1-[5-(Dimethylamino)-1-naphthyl-sulfonyl]imidazolidine-2-thione

Yong Zhang* and Lei Teng

School of Chemical and Materials Engineering, Huangshi Institute of Technology, Huangshi 435003, People's Republic of China

Correspondence e-mail: zy0340907@yahoo.com.cn

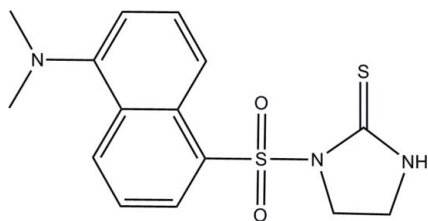
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.056; wR factor = 0.145; data-to-parameter ratio = 16.2.

In the title molecule, $\text{C}_{15}\text{H}_{17}\text{N}_3\text{O}_2\text{S}_2$, the dihedral angle between the naphthalene ring system and the imidazole ring is $89.63(2)^\circ$. The crystal structure is stabilized by weak intermolecular $\text{C}-\text{H}\cdots\pi$ and $\text{N}-\text{H}\cdots\pi$ interactions.

Related literature

For the applications of compounds containing a 5-(dimethylamino)naphthalene-1-sulfonyl group, see: Corradini *et al.* (1996, 1997); Christoforou *et al.* (2006). For a related structure, see: Zhang *et al.* (2009). For the synthetic procedure, see: Corradini *et al.* (1996).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{17}\text{N}_3\text{O}_2\text{S}_2$
 $M_r = 335.44$

 Monoclinic, $P2_1/n$
 $a = 15.364(4)$ Å

 $b = 6.9814(18)$ Å

 $c = 15.470(4)$ Å

 $\beta = 113.967(4)^\circ$
 $V = 1516.3(7)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.36$ mm⁻¹
 $T = 298$ K

 $0.33 \times 0.32 \times 0.28$ mm

Data collection

 Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1997)
 $T_{\min} = 0.955$, $T_{\max} = 0.965$

 8935 measured reflections
 3302 independent reflections
 2605 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.104$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.145$
 $S = 1.04$
 3302 reflections
 204 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.50$ e Å⁻³
 $\Delta\rho_{\min} = -0.35$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

 C_{g1} and C_{g2} are the centroids of the C1–C5/C10 and C5–C10 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C2-H2\cdots C_{g1}^i$	0.93	2.88	3.667 (3)	143
$N3-H3A\cdots C_{g2}^{ii}$	0.88 (3)	2.58 (3)	3.433 (3)	165 (2)

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

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5094).

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supporting information

Acta Cryst. (2010). E66, o2181 [https://doi.org/10.1107/S1600536810029788]

1-[5-(Dimethylamino)-1-naphthylsulfonyl]imidazolidine-2-thione**Yong Zhang and Lei Teng****S1. Comment**

The dansyl (5-(dimethylamino)naphthalene-1-sulfonyl) group has been widely used as a fluorophore due to its good fluorescent properties. Recently many dansyl derivatives have been reported (Corradini *et al.*, 1996,1997; Christoforou *et al.*, 2006). We show great interest in preparing fluorescent probes that are expected to bind to hydrophobic sites in proteins or membranes and have recently published a structure resulted to the title compound (Zhang *et al.*, 2009). With this in mind, the title compound, (I), was prepared and we report herein the crystal structure.

In the molecular structure (Fig. 1), the dihedral angle between the naphthalene ring and five-membered heterocyclic ring is 89.63 (2)°. The crystal structure is stabilized by weak intermolecular C—H \cdots π and N—H \cdots π interactions.

S2. Experimental

The intermediate *N*-(2-Aminoethyl)-5-(dimethylamino)naphthalene-1-sulfonamide was synthesized according to a literature procedure (Corradini *et al.*, 1996). Carbon bisulfide (0.76 g, 10 mmol) and sodium hydroxide(0.40 g, 10 mmol) were added into a stirred solution of the above intermediate (1.47 g, 5 mmol) in dry methanol (20 ml).The reaction mixture was allowed to stir for 24 hr at 293 K. The progress of the reaction was monitored by TLC, until the completion of reaction. The solvent was evaporated and the residue was purified by column chromatography (dichloromethane-ethyl acetate,1:8 v/v) to afford the title compound as a yellow solid. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in dichloromethane at room temperature.

S3. Refinement

All H atoms were placed in idealized positions [$C-H(\text{methyl})=0.96 \text{ \AA}$, 0.97 \AA (methylene) and 0.93 \AA (aromatic),with $U_{\text{iso}}(\text{H})= 1.5U_{\text{eq}}(\text{methyl C}) 1.2U_{\text{eq}}(\text{other C})$. N-bonded hydrogen atom was found from the difference map and refined with the restraint of $N-H = 0.88 (3) \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{N})$.

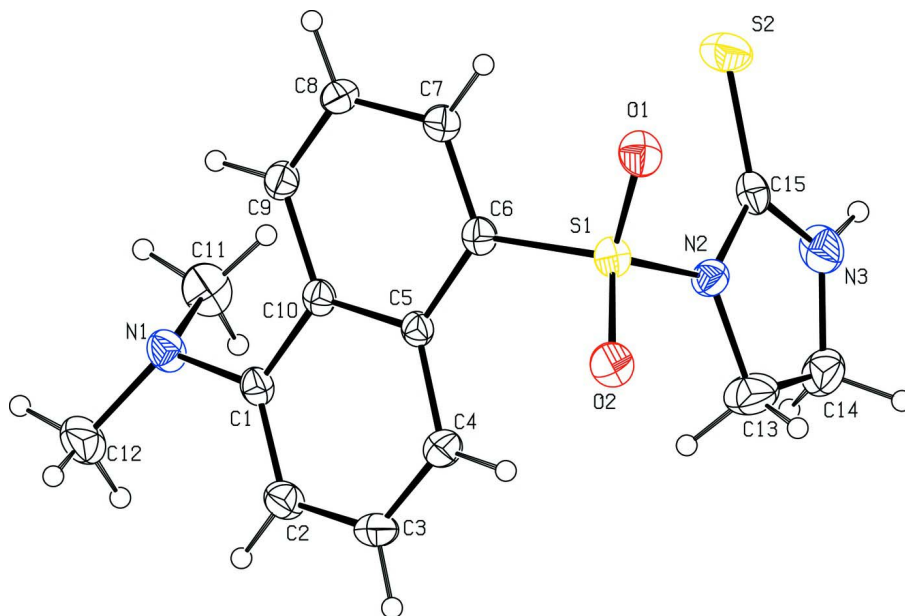


Figure 1

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

1-[5-(Dimethylamino)-1-naphthylsulfonyl]imidazolidine-2-thione

Crystal data

$C_{15}H_{17}N_3O_2S_2$

$M_r = 335.44$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1/n$

$a = 15.364\ (4)\ \text{\AA}$

$b = 6.9814\ (18)\ \text{\AA}$

$c = 15.470\ (4)\ \text{\AA}$

$\beta = 113.967\ (4)^\circ$

$V = 1516.3\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 700$

$D_x = 1.465\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2682 reflections

$\theta = 2.4\text{--}28.1^\circ$

$\mu = 0.36\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, yellow

$0.33 \times 0.32 \times 0.28\ \text{mm}$

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1997)

$T_{\min} = 0.955$, $T_{\max} = 0.965$

8935 measured reflections

3302 independent reflections

2605 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.104$

$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 2.4^\circ$

$h = -17 \rightarrow 19$

$k = -8 \rightarrow 8$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.056$

$wR(F^2) = 0.145$

$S = 1.04$

3302 reflections

204 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 atoms treated by a mixture of independent
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.071P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.50 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$$

Special details

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 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.61885 (15)	0.6070 (3)	0.72722 (15)	0.0306 (5)
C2	0.68621 (19)	0.7474 (3)	0.76019 (18)	0.0379 (6)
H2	0.6868	0.8466	0.7203	0.045*
C3	0.75453 (19)	0.7430 (3)	0.85392 (19)	0.0434 (6)
H3	0.8011	0.8375	0.8746	0.052*
C4	0.75429 (17)	0.6051 (3)	0.91479 (17)	0.0384 (6)
H4	0.7983	0.6099	0.9774	0.046*
C5	0.68768 (15)	0.4529 (3)	0.88431 (15)	0.0271 (5)
C6	0.68432 (16)	0.2951 (3)	0.94243 (16)	0.0299 (5)
C7	0.62594 (17)	0.1403 (3)	0.90561 (16)	0.0342 (5)
H7	0.6253	0.0398	0.9448	0.041*
C8	0.56701 (17)	0.1343 (3)	0.80839 (17)	0.0370 (6)
H8	0.5298	0.0267	0.7828	0.044*
C9	0.56426 (17)	0.2841 (3)	0.75203 (17)	0.0336 (5)
H9	0.5235	0.2792	0.6883	0.040*
C10	0.62191 (14)	0.4484 (3)	0.78765 (15)	0.0271 (5)
C11	0.45077 (18)	0.6337 (5)	0.6290 (2)	0.0536 (7)
H11A	0.4425	0.5578	0.6769	0.080*
H11B	0.4051	0.5953	0.5678	0.080*
H11C	0.4416	0.7665	0.6391	0.080*
C12	0.5635 (2)	0.7304 (4)	0.5667 (2)	0.0576 (8)
H12A	0.5604	0.8616	0.5837	0.086*
H12B	0.5157	0.7072	0.5043	0.086*
H12C	0.6253	0.7046	0.5677	0.086*
C13	0.7299 (2)	0.6395 (4)	1.1372 (2)	0.0589 (8)
H13A	0.7391	0.7055	1.0863	0.071*
H13B	0.7896	0.6394	1.1929	0.071*
C14	0.6522 (2)	0.7308 (4)	1.1575 (2)	0.0515 (7)
H14A	0.6764	0.7806	1.2216	0.062*
H14B	0.6223	0.8341	1.1135	0.062*

C15	0.60716 (16)	0.4138 (3)	1.11357 (15)	0.0336 (5)
N1	0.54715 (14)	0.6055 (3)	0.63401 (13)	0.0381 (5)
N2	0.69536 (13)	0.4434 (3)	1.10932 (13)	0.0328 (4)
N3	0.58699 (18)	0.5772 (3)	1.14554 (17)	0.0496 (6)
H3A	0.534 (2)	0.590 (4)	1.154 (2)	0.059*
O1	0.74468 (12)	0.1037 (2)	1.09977 (12)	0.0422 (4)
O2	0.84230 (11)	0.3748 (3)	1.09114 (11)	0.0414 (4)
S1	0.75069 (4)	0.29031 (8)	1.06638 (4)	0.03206 (19)
S2	0.53971 (5)	0.22082 (10)	1.08475 (6)	0.0524 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0297 (12)	0.0365 (13)	0.0286 (12)	0.0012 (10)	0.0150 (9)	0.0006 (9)
C2	0.0425 (14)	0.0352 (13)	0.0384 (13)	-0.0015 (10)	0.0190 (11)	0.0049 (10)
C3	0.0452 (16)	0.0396 (14)	0.0437 (15)	-0.0167 (11)	0.0165 (12)	-0.0017 (11)
C4	0.0372 (14)	0.0423 (14)	0.0316 (12)	-0.0092 (11)	0.0097 (10)	-0.0028 (10)
C5	0.0263 (11)	0.0309 (11)	0.0263 (11)	0.0000 (9)	0.0129 (8)	-0.0013 (9)
C6	0.0266 (11)	0.0343 (12)	0.0291 (11)	0.0026 (9)	0.0118 (9)	0.0001 (9)
C7	0.0402 (13)	0.0308 (12)	0.0339 (12)	-0.0022 (10)	0.0173 (10)	-0.0006 (10)
C8	0.0413 (14)	0.0339 (13)	0.0393 (13)	-0.0111 (11)	0.0200 (11)	-0.0103 (10)
C9	0.0303 (12)	0.0416 (13)	0.0295 (12)	-0.0041 (10)	0.0126 (9)	-0.0060 (10)
C10	0.0232 (10)	0.0328 (12)	0.0280 (11)	0.0021 (9)	0.0130 (9)	-0.0015 (9)
C11	0.0345 (15)	0.072 (2)	0.0477 (16)	0.0080 (14)	0.0092 (12)	0.0107 (14)
C12	0.0583 (19)	0.073 (2)	0.0356 (15)	-0.0115 (15)	0.0130 (13)	0.0146 (13)
C13	0.0640 (19)	0.0383 (15)	0.083 (2)	-0.0096 (14)	0.0386 (17)	-0.0153 (15)
C14	0.070 (2)	0.0387 (15)	0.0469 (16)	0.0002 (13)	0.0246 (15)	-0.0069 (12)
C15	0.0395 (13)	0.0371 (13)	0.0266 (11)	0.0071 (10)	0.0158 (10)	0.0067 (10)
N1	0.0322 (11)	0.0490 (12)	0.0302 (10)	0.0000 (9)	0.0099 (8)	0.0070 (9)
N2	0.0342 (11)	0.0307 (10)	0.0343 (11)	-0.0012 (8)	0.0145 (8)	-0.0036 (8)
N3	0.0595 (15)	0.0440 (13)	0.0595 (15)	0.0068 (11)	0.0389 (13)	-0.0025 (11)
O1	0.0509 (11)	0.0343 (9)	0.0399 (10)	0.0105 (8)	0.0169 (8)	0.0090 (7)
O2	0.0284 (9)	0.0521 (11)	0.0391 (10)	0.0026 (8)	0.0092 (7)	0.0035 (8)
S1	0.0314 (3)	0.0339 (3)	0.0303 (3)	0.0052 (2)	0.0119 (2)	0.0030 (2)
S2	0.0465 (4)	0.0484 (4)	0.0687 (5)	-0.0096 (3)	0.0300 (4)	0.0000 (3)

Geometric parameters (Å, °)

C1—C2	1.365 (3)	C11—H11B	0.9600
C1—N1	1.415 (3)	C11—H11C	0.9600
C1—C10	1.437 (3)	C12—N1	1.456 (3)
C2—C3	1.403 (4)	C12—H12A	0.9600
C2—H2	0.9300	C12—H12B	0.9600
C3—C4	1.348 (3)	C12—H12C	0.9600
C3—H3	0.9300	C13—N2	1.468 (3)
C4—C5	1.417 (3)	C13—C14	1.496 (4)
C4—H4	0.9300	C13—H13A	0.9700
C5—C10	1.425 (3)	C13—H13B	0.9700

C5—C6	1.436 (3)	C14—N3	1.428 (3)
C6—C7	1.372 (3)	C14—H14A	0.9700
C6—S1	1.769 (2)	C14—H14B	0.9700
C7—C8	1.406 (3)	C15—N3	1.329 (3)
C7—H7	0.9300	C15—N2	1.398 (3)
C8—C9	1.351 (3)	C15—S2	1.647 (2)
C8—H8	0.9300	N2—S1	1.6628 (19)
C9—C10	1.417 (3)	N3—H3A	0.88 (3)
C9—H9	0.9300	O1—S1	1.4182 (17)
C11—N1	1.464 (3)	O2—S1	1.4275 (17)
C11—H11A	0.9600		
C2—C1—N1	123.0 (2)	N1—C12—H12A	109.5
C2—C1—C10	119.4 (2)	N1—C12—H12B	109.5
N1—C1—C10	117.6 (2)	H12A—C12—H12B	109.5
C1—C2—C3	120.4 (2)	N1—C12—H12C	109.5
C1—C2—H2	119.8	H12A—C12—H12C	109.5
C3—C2—H2	119.8	H12B—C12—H12C	109.5
C4—C3—C2	121.7 (2)	N2—C13—C14	103.6 (2)
C4—C3—H3	119.2	N2—C13—H13A	111.0
C2—C3—H3	119.2	C14—C13—H13A	111.0
C3—C4—C5	120.7 (2)	N2—C13—H13B	111.0
C3—C4—H4	119.7	C14—C13—H13B	111.0
C5—C4—H4	119.7	H13A—C13—H13B	109.0
C4—C5—C10	118.31 (19)	N3—C14—C13	103.3 (2)
C4—C5—C6	124.9 (2)	N3—C14—H14A	111.1
C10—C5—C6	116.72 (19)	C13—C14—H14A	111.1
C7—C6—C5	121.9 (2)	N3—C14—H14B	111.1
C7—C6—S1	115.27 (17)	C13—C14—H14B	111.1
C5—C6—S1	122.80 (17)	H14A—C14—H14B	109.1
C6—C7—C8	119.7 (2)	N3—C15—N2	105.5 (2)
C6—C7—H7	120.1	N3—C15—S2	125.9 (2)
C8—C7—H7	120.1	N2—C15—S2	128.54 (17)
C9—C8—C7	120.3 (2)	C1—N1—C12	115.8 (2)
C9—C8—H8	119.8	C1—N1—C11	113.87 (19)
C7—C8—H8	119.8	C12—N1—C11	110.3 (2)
C8—C9—C10	121.7 (2)	C15—N2—C13	111.46 (19)
C8—C9—H9	119.2	C15—N2—S1	125.90 (16)
C10—C9—H9	119.2	C13—N2—S1	122.26 (17)
C9—C10—C5	119.38 (19)	C15—N3—C14	115.8 (2)
C9—C10—C1	121.37 (19)	C15—N3—H3A	121 (2)
C5—C10—C1	119.17 (19)	C14—N3—H3A	123.1 (19)
N1—C11—H11A	109.5	O1—S1—O2	119.00 (10)
N1—C11—H11B	109.5	O1—S1—N2	108.99 (10)
H11A—C11—H11B	109.5	O2—S1—N2	103.62 (10)
N1—C11—H11C	109.5	O1—S1—C6	108.55 (11)
H11A—C11—H11C	109.5	O2—S1—C6	110.75 (10)
H11B—C11—H11C	109.5	N2—S1—C6	104.94 (10)

N1—C1—C2—C3	178.9 (2)	C2—C1—N1—C12	16.4 (3)
C10—C1—C2—C3	-3.7 (4)	C10—C1—N1—C12	-161.2 (2)
C1—C2—C3—C4	-2.1 (4)	C2—C1—N1—C11	-113.0 (3)
C2—C3—C4—C5	3.6 (4)	C10—C1—N1—C11	69.5 (3)
C3—C4—C5—C10	0.7 (3)	N3—C15—N2—C13	-2.2 (3)
C3—C4—C5—C6	177.1 (2)	S2—C15—N2—C13	177.2 (2)
C4—C5—C6—C7	-171.7 (2)	N3—C15—N2—S1	-175.13 (17)
C10—C5—C6—C7	4.8 (3)	S2—C15—N2—S1	4.3 (3)
C4—C5—C6—S1	11.1 (3)	C14—C13—N2—C15	-1.1 (3)
C10—C5—C6—S1	-172.47 (15)	C14—C13—N2—S1	172.14 (18)
C5—C6—C7—C8	-0.3 (3)	N2—C15—N3—C14	5.0 (3)
S1—C6—C7—C8	177.21 (17)	S2—C15—N3—C14	-174.4 (2)
C6—C7—C8—C9	-3.2 (3)	C13—C14—N3—C15	-5.7 (3)
C7—C8—C9—C10	1.9 (4)	C15—N2—S1—O1	-44.0 (2)
C8—C9—C10—C5	2.9 (3)	C13—N2—S1—O1	143.8 (2)
C8—C9—C10—C1	179.8 (2)	C15—N2—S1—O2	-171.64 (17)
C4—C5—C10—C9	170.7 (2)	C13—N2—S1—O2	16.1 (2)
C6—C5—C10—C9	-6.0 (3)	C15—N2—S1—C6	72.1 (2)
C4—C5—C10—C1	-6.3 (3)	C13—N2—S1—C6	-100.1 (2)
C6—C5—C10—C1	177.01 (18)	C7—C6—S1—O1	12.6 (2)
C2—C1—C10—C9	-169.1 (2)	C5—C6—S1—O1	-169.92 (17)
N1—C1—C10—C9	8.5 (3)	C7—C6—S1—O2	145.02 (18)
C2—C1—C10—C5	7.8 (3)	C5—C6—S1—O2	-37.5 (2)
N1—C1—C10—C5	-174.59 (19)	C7—C6—S1—N2	-103.77 (18)
N2—C13—C14—N3	3.7 (3)	C5—C6—S1—N2	73.67 (19)

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 and Cg2 are the centroids of the C1—C5/C10 and C5—C10 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 \cdots Cg1 ⁱ	0.93	2.88	3.667 (3)	143
N3—H3A \cdots Cg2 ⁱⁱ	0.88 (3)	2.58 (3)	3.433 (3)	165 (2)

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