

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## N-Ethyl-2-[1-(2-hydroxynaphthalen-1-yl)ethylidene]hydrazinecarbothioamide

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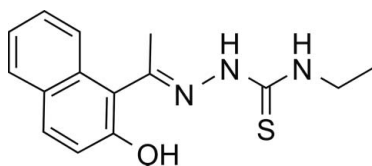
Received 23 May 2014; accepted 23 May 2014

Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.054;  $wR$  factor = 0.153; data-to-parameter ratio = 14.7.

In the title compound,  $\text{C}_{15}\text{H}_{17}\text{N}_3\text{OS}$ , the dihedral angle between the mean planes of the 2-hydroxynaphthyl ring system and the hydrazinecarbothioamide group is  $73.7(3)^\circ$ . In the crystal, weak  $\text{O}-\text{H}\cdots\text{S}$  and  $\text{C}-\text{H}\cdots\text{O}$  interactions and  $\pi-\pi$  stacking interactions involving one of the hydroxynaphthyl rings with a centroid-centroid distance of  $3.6648(14)$  Å are observed, forming infinite chains along  $[010]$ . In addition,  $\text{N}-\text{H}\cdots\text{S}$  interactions occur.

## Related literature

For the biological activity of thiosemicarbazones, see: Chellan *et al.* (2010). For binding motifs of thiosemicarbazones, see: Lobana *et al.* (2009). For thiosemicarbazones as ligands in catalysis, see: Xie *et al.* (2010). For related structures, see: Anderson *et al.* (2012, 2013a,b). For standard bond lengths, see: Allen *et al.* (1987).



## Experimental

## Crystal data

 $\text{C}_{15}\text{H}_{17}\text{N}_3\text{OS}$  $M_r = 287.38$ Triclinic,  $P\bar{1}$  $a = 8.8988(7)$  Å $b = 9.2993(8)$  Å $c = 9.4821(5)$  Å $\alpha = 92.525(6)^\circ$  $\beta = 113.034(7)^\circ$  $\gamma = 93.990(7)^\circ$  $V = 718.18(10)$  Å<sup>3</sup> $Z = 2$ Cu  $K\alpha$  radiation $\mu = 1.99$  mm<sup>-1</sup> $T = 173$  K $0.42 \times 0.22 \times 0.14$  mm

## Data collection

Agilent Eos Gemini diffractometer

Absorption correction: multi-scan

(CrysAlis PRO and CrysAlis

RED; Agilent, 2012)

 $T_{\min} = 0.429$ ,  $T_{\max} = 1.000$ 

4327 measured reflections

2710 independent reflections

2365 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.032$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$  $wR(F^2) = 0.153$  $S = 1.06$ 

2710 reflections

184 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.41$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.41$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{S1}^{\text{i}}$	0.84	2.40	3.2349 (17)	171
$\text{C14}-\text{H14A}\cdots\text{O1}^{\text{ii}}$	0.99	2.49	3.474 (4)	171
$\text{N2}-\text{H2}\cdots\text{S1}^{\text{iii}}$	0.88	2.79	3.548 (2)	145

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

Data collection: CrysAlis PRO (Agilent, 2012); cell refinement: CrysAlis PRO; data reduction: CrysAlis RED (Agilent, 2012); program(s) used to solve structure: SUPERFLIP (Palatinus & Chapuis, 2007; Palatinus & van der Lee, 2008; Palatinus *et al.*, 2012); program(s) used to refine structure: SHELXL2012 (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2.

JPJ acknowledges the NSF-MRI program (grant No. CHE-1039027) for funds to purchase the X-ray diffractometer.

Supporting information for this paper is available from the IUCr electronic archives (Reference: BT6981).

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## supporting information

*Acta Cryst.* (2014). E70, o732 [doi:10.1107/S1600536814012057]

**N-Ethyl-2-[1-(2-hydroxynaphthalen-1-yl)ethylidene]hydrazinecarbothioamide****Brian J. Anderson, Zachary A. Shalit and Jerry P. Jasinski****S1. Comment**

Thiosemicarbazones are a versatile class of ligands that have been studied for their biological activity (Chellan *et al.*, 2010), interesting binding motifs (Lobana *et al.*, 2009), and their use as ligands in catalysis (Xie *et al.*, 2010). We have previously reported the structure of three similar novel thiosemicarbazones (Anderson *et al.*, 2012; Anderson *et al.*, 2013a; Anderson *et al.*, 2013b). Here, we report the synthesis and crystal structure of a new novel thiosemicarbazone ligand starting with 2'-hydroxy-1'-acetonaphthone and 4-ethyl-thio-semicarbazide, C<sub>15</sub>H<sub>17</sub>N<sub>3</sub>OS.

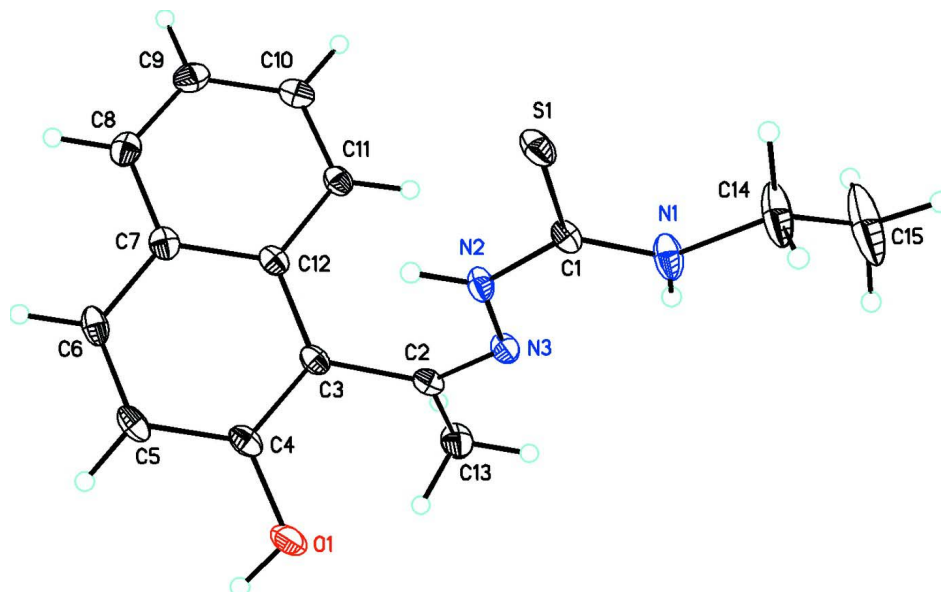
In the title compound, the dihedral angle between the mean planes of the 2-hydroxynaphthyl ring and the hydrazinecarbothioamide group (N3/N2/C1/S1/N1) is 73.7 (3)°. Bond lengths are in normal ranges (Allen *et al.*, 1987). In the crystal, weak O1—H1...S1 and C14—H14A...O1 intermolecular interactions and  $\pi$ - $\pi$  stacking interactions involving one of the hydroxynaphthyl rings are observed forming infinite polymeric chains along [010] (Cg2—Cg2 = 3.6648 (14)Å; 2-x, 1-y, -z; Cg2 = C7—C12). In addition, there are N-H...S interactions stabilizing the crystal structure.

**S2. Experimental**

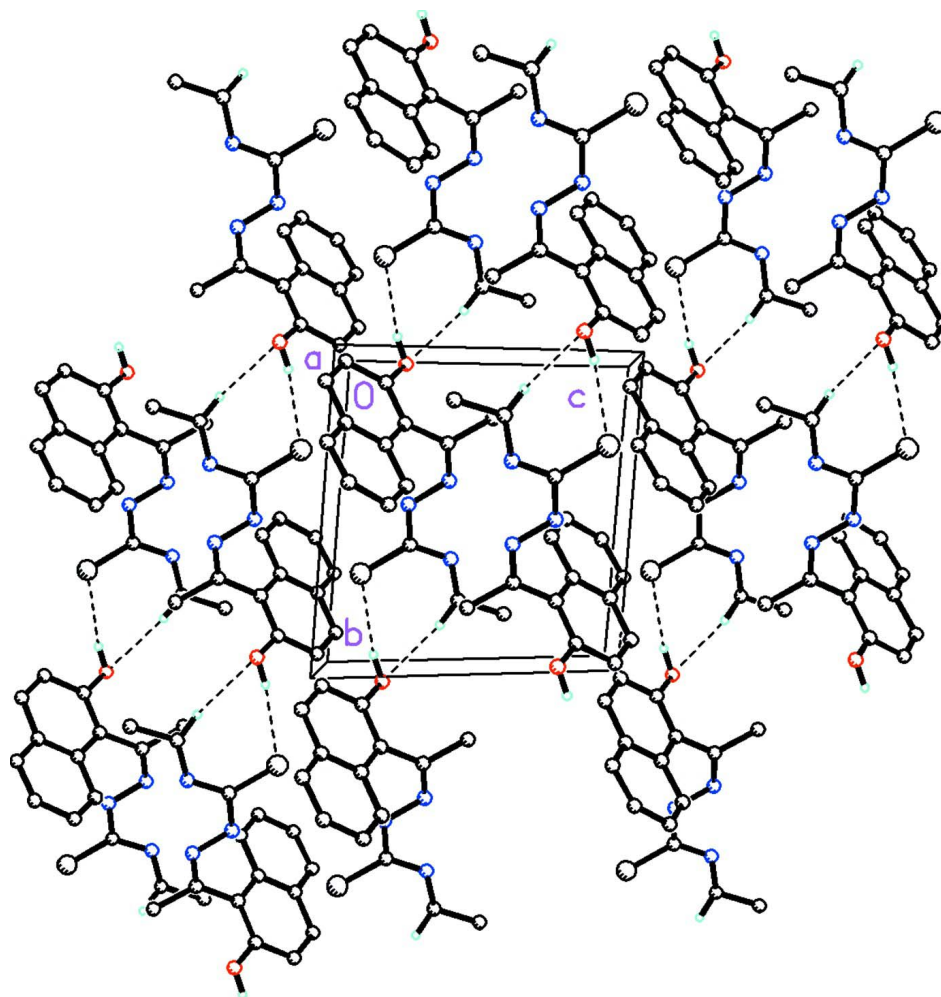
A 25 mL round bottom flask was charged with 0.2052 g (1.102 mmol) 2'-hydroxy-1'-acetonaphthone, and 0.1341 g (1.125 mmol) 4-ethyl-thio-semicarbazide and in 5 mL of 1:1 ratio H<sub>2</sub>O to ETOH. The resulting slurry was refluxed for 96 hours (Fig. 3). After reflux the opaque solution was transferred to a separatory funnel to which dichloromethane and water were added. The organic layer was separated and the aqueous layer was extracted twice with 5 mL DCM. The organic layers were combined, washed with brine and dried with magnesium sulfate, and the solvent was removed in vacuo to yield a colorless oil. The oil was dissolved in minimal 343° K acetonitrile and left to slowly cool to 273° K, after 24 days colorless crystals were observed. m.p. 444–446 K.

**S3. Refinement**

All of the H atoms were placed in their calculated positions and then refined using the riding model with Atom—H lengths of 0.95Å (CH), 0.99Å (CH<sub>2</sub>), 0.98Å (CH<sub>3</sub>), 0.88Å (NH) or 0.84Å (OH). Isotropic displacement parameters for these atoms were set to 1.2 (CH, CH<sub>2</sub>, NH) or 1.5 (CH<sub>3</sub>, OH) times  $U_{eq}$  of the parent atom. Idealised Me refined as rotating group. Idealised tetrahedral OH refined as rotating group.

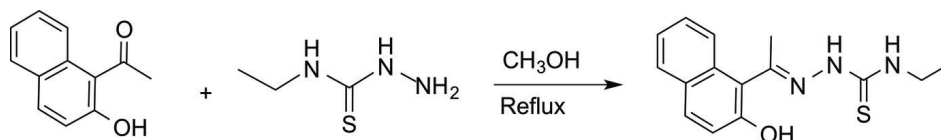
**Figure 1**

ORTEP drawing of  $C_{15}H_{17}N_3OS$ , showing the labeling scheme with 30% probability displacement ellipsoids.



**Figure 2**

Molecular packing for the title compound viewed along the *a* axis. Dashed lines indicate weak O1—H1...S1 and C14—H14A...O1 intermolecular interactions forming infinite polymeric chains along [010].



**Figure 3**

Reaction scheme.

### *N*-Ethyl-2-[1-(2-hydroxynaphthalen-1-yl)ethylidene]hydrazinecarbothioamide

#### Crystal data

$C_{15}H_{17}N_3OS$

$M_r = 287.38$

Triclinic,  $P\bar{1}$

$a = 8.8988$  (7) Å

$b = 9.2993$  (8) Å

$c = 9.4821$  (5) Å

$\alpha = 92.525$  (6)°

$\beta = 113.034$  (7)°

$\gamma = 93.990$  (7)°

$V = 718.18$  (10) Å<sup>3</sup>

$Z = 2$

$F(000) = 304$

$D_x = 1.329$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54184$  Å

Cell parameters from 1994 reflections  
 $\theta = 4.8\text{--}71.3^\circ$   
 $\mu = 1.99\text{ mm}^{-1}$

$T = 173\text{ K}$   
 Irregular, colourless  
 $0.42 \times 0.22 \times 0.14\text{ mm}$

*Data collection*

Agilent Eos Gemini  
 diffractometer  
 Radiation source: Enhance (Cu) X-ray Source  
 Graphite monochromator  
 Detector resolution:  $16.0416\text{ pixels mm}^{-1}$   
 $\omega$  scans  
 Absorption correction: multi-scan  
 (*CrysAlis PRO* and *CrysAlis RED*; Agilent,  
 2012)

$T_{\min} = 0.429$ ,  $T_{\max} = 1.000$   
 4327 measured reflections  
 2710 independent reflections  
 2365 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.032$   
 $\theta_{\max} = 71.4^\circ$ ,  $\theta_{\min} = 4.8^\circ$   
 $h = -10 \rightarrow 10$   
 $k = -10 \rightarrow 11$   
 $l = -11 \rightarrow 9$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.153$   
 $S = 1.06$   
 2710 reflections  
 184 parameters  
 0 restraints

Primary atom site location: structure-invariant  
 direct methods  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0898P)^2 + 0.2589P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.41\text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.41\text{ e \AA}^{-3}$

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.53047 (7)	0.69555 (5)	0.11487 (6)	0.0286 (2)
O1	0.6330 (2)	0.03677 (17)	0.2160 (2)	0.0360 (4)
H1	0.6056	-0.0495	0.1793	0.054*
N1	0.6310 (3)	0.6503 (2)	0.4127 (2)	0.0342 (5)
H1A	0.6824	0.5980	0.4893	0.041*
N2	0.6777 (2)	0.47722 (19)	0.2599 (2)	0.0242 (4)
H2	0.6733	0.4430	0.1702	0.029*
N3	0.7469 (2)	0.40209 (19)	0.3892 (2)	0.0234 (4)
C1	0.6168 (3)	0.6042 (2)	0.2735 (2)	0.0226 (5)
C2	0.8136 (3)	0.2868 (2)	0.3759 (3)	0.0230 (5)
C3	0.8262 (3)	0.2283 (2)	0.2325 (2)	0.0219 (4)
C4	0.7379 (3)	0.1000 (2)	0.1585 (3)	0.0251 (5)
C5	0.7515 (3)	0.0388 (2)	0.0254 (3)	0.0284 (5)
H5	0.6860	-0.0475	-0.0267	0.034*
C6	0.8583 (3)	0.1036 (2)	-0.0279 (3)	0.0278 (5)
H6	0.8656	0.0625	-0.1182	0.033*

C7	0.9594 (3)	0.2317 (2)	0.0491 (3)	0.0249 (5)
C8	1.0824 (3)	0.2928 (3)	0.0032 (3)	0.0294 (5)
H8	1.0948	0.2499	-0.0839	0.035*
C9	1.1834 (3)	0.4128 (3)	0.0832 (3)	0.0319 (5)
H9	1.2667	0.4516	0.0527	0.038*
C10	1.1634 (3)	0.4782 (2)	0.2102 (3)	0.0298 (5)
H10	1.2323	0.5623	0.2641	0.036*
C11	1.0455 (3)	0.4222 (2)	0.2576 (3)	0.0245 (5)
H11	1.0332	0.4684	0.3433	0.029*
C12	0.9418 (2)	0.2955 (2)	0.1797 (2)	0.0216 (4)
C13	0.8917 (3)	0.2093 (3)	0.5182 (3)	0.0344 (6)
H13A	1.0106	0.2152	0.5473	0.052*
H13B	0.8472	0.1076	0.4985	0.052*
H13C	0.8686	0.2543	0.6021	0.052*
C14	0.5673 (4)	0.7823 (4)	0.4484 (3)	0.0555 (9)
H14A	0.5943	0.8613	0.3931	0.067*
H14B	0.4462	0.7659	0.4092	0.067*
C15	0.6293 (6)	0.8273 (5)	0.6067 (4)	0.0845 (15)
H15A	0.5734	0.9099	0.6224	0.127*
H15B	0.7473	0.8558	0.6438	0.127*
H15C	0.6102	0.7475	0.6636	0.127*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0383 (4)	0.0169 (3)	0.0243 (3)	0.0057 (2)	0.0052 (2)	0.0014 (2)
O1	0.0362 (9)	0.0208 (8)	0.0520 (11)	-0.0063 (7)	0.0208 (8)	-0.0064 (7)
N1	0.0440 (12)	0.0314 (11)	0.0233 (10)	0.0204 (9)	0.0066 (9)	0.0003 (8)
N2	0.0329 (10)	0.0193 (9)	0.0190 (9)	0.0090 (7)	0.0078 (8)	0.0005 (7)
N3	0.0277 (9)	0.0196 (9)	0.0206 (9)	0.0026 (7)	0.0071 (7)	0.0008 (7)
C1	0.0210 (10)	0.0188 (10)	0.0236 (11)	0.0022 (8)	0.0044 (8)	-0.0017 (8)
C2	0.0221 (10)	0.0173 (10)	0.0273 (11)	0.0010 (8)	0.0075 (9)	0.0023 (8)
C3	0.0218 (10)	0.0158 (9)	0.0249 (11)	0.0050 (8)	0.0051 (8)	0.0021 (8)
C4	0.0215 (10)	0.0164 (10)	0.0331 (12)	0.0054 (8)	0.0054 (9)	0.0015 (8)
C5	0.0245 (11)	0.0184 (10)	0.0339 (12)	0.0052 (8)	0.0026 (9)	-0.0042 (9)
C6	0.0281 (11)	0.0261 (11)	0.0238 (11)	0.0115 (9)	0.0037 (9)	-0.0042 (9)
C7	0.0248 (11)	0.0235 (10)	0.0240 (11)	0.0100 (9)	0.0057 (9)	0.0048 (8)
C8	0.0339 (12)	0.0310 (12)	0.0253 (11)	0.0120 (10)	0.0119 (10)	0.0072 (9)
C9	0.0314 (12)	0.0323 (12)	0.0357 (13)	0.0065 (10)	0.0156 (10)	0.0124 (10)
C10	0.0274 (11)	0.0242 (11)	0.0339 (12)	0.0005 (9)	0.0078 (10)	0.0052 (9)
C11	0.0264 (11)	0.0194 (10)	0.0247 (11)	0.0040 (8)	0.0067 (9)	0.0019 (8)
C12	0.0205 (10)	0.0176 (10)	0.0222 (10)	0.0062 (8)	0.0029 (8)	0.0039 (8)
C13	0.0469 (14)	0.0273 (12)	0.0293 (13)	0.0120 (11)	0.0135 (11)	0.0078 (10)
C14	0.072 (2)	0.0528 (17)	0.0360 (15)	0.0411 (16)	0.0101 (14)	-0.0068 (13)
C15	0.107 (3)	0.074 (2)	0.051 (2)	0.062 (2)	0.002 (2)	-0.0201 (18)

*Geometric parameters (Å, °)*

S1—C1	1.695 (2)	C7—C8	1.420 (3)
O1—H1	0.8400	C7—C12	1.418 (3)
O1—C4	1.366 (3)	C8—H8	0.9500
N1—H1A	0.8800	C8—C9	1.372 (4)
N1—C1	1.324 (3)	C9—H9	0.9500
N1—C14	1.468 (3)	C9—C10	1.404 (4)
N2—H2	0.8800	C10—H10	0.9500
N2—N3	1.384 (3)	C10—C11	1.374 (3)
N2—C1	1.355 (3)	C11—H11	0.9500
N3—C2	1.286 (3)	C11—C12	1.423 (3)
C2—C3	1.491 (3)	C13—H13A	0.9800
C2—C13	1.499 (3)	C13—H13B	0.9800
C3—C4	1.378 (3)	C13—H13C	0.9800
C3—C12	1.428 (3)	C14—H14A	0.9900
C4—C5	1.413 (3)	C14—H14B	0.9900
C5—H5	0.9500	C14—C15	1.413 (4)
C5—C6	1.358 (3)	C15—H15A	0.9800
C6—H6	0.9500	C15—H15B	0.9800
C6—C7	1.422 (3)	C15—H15C	0.9800
C4—O1—H1	109.5	C9—C8—H8	119.6
C1—N1—H1A	117.7	C8—C9—H9	120.1
C1—N1—C14	124.6 (2)	C8—C9—C10	119.8 (2)
C14—N1—H1A	117.7	C10—C9—H9	120.1
N3—N2—H2	120.5	C9—C10—H10	119.5
C1—N2—H2	120.5	C11—C10—C9	120.9 (2)
C1—N2—N3	118.96 (18)	C11—C10—H10	119.5
C2—N3—N2	117.91 (18)	C10—C11—H11	119.7
N1—C1—S1	123.94 (17)	C10—C11—C12	120.6 (2)
N1—C1—N2	117.0 (2)	C12—C11—H11	119.7
N2—C1—S1	119.01 (16)	C7—C12—C3	119.48 (19)
N3—C2—C3	125.3 (2)	C7—C12—C11	118.3 (2)
N3—C2—C13	117.1 (2)	C11—C12—C3	122.1 (2)
C3—C2—C13	117.53 (19)	C2—C13—H13A	109.5
C4—C3—C2	119.75 (19)	C2—C13—H13B	109.5
C4—C3—C12	119.3 (2)	C2—C13—H13C	109.5
C12—C3—C2	120.65 (18)	H13A—C13—H13B	109.5
O1—C4—C3	117.4 (2)	H13A—C13—H13C	109.5
O1—C4—C5	121.37 (19)	H13B—C13—H13C	109.5
C3—C4—C5	121.2 (2)	N1—C14—H14A	108.7
C4—C5—H5	120.1	N1—C14—H14B	108.7
C6—C5—C4	119.9 (2)	H14A—C14—H14B	107.6
C6—C5—H5	120.1	C15—C14—N1	114.2 (3)
C5—C6—H6	119.4	C15—C14—H14A	108.7
C5—C6—C7	121.3 (2)	C15—C14—H14B	108.7
C7—C6—H6	119.4	C14—C15—H15A	109.5

C8—C7—C6	121.8 (2)	C14—C15—H15B	109.5
C12—C7—C6	118.7 (2)	C14—C15—H15C	109.5
C12—C7—C8	119.4 (2)	H15A—C15—H15B	109.5
C7—C8—H8	119.6	H15A—C15—H15C	109.5
C9—C8—C7	120.8 (2)	H15B—C15—H15C	109.5
O1—C4—C5—C6	-179.3 (2)	C5—C6—C7—C12	-2.9 (3)
N2—N3—C2—C3	1.4 (3)	C6—C7—C8—C9	-177.1 (2)
N2—N3—C2—C13	178.41 (19)	C6—C7—C12—C3	1.3 (3)
N3—N2—C1—S1	-179.44 (15)	C6—C7—C12—C11	178.85 (18)
N3—N2—C1—N1	1.5 (3)	C7—C8—C9—C10	-1.3 (3)
N3—C2—C3—C4	-111.9 (2)	C8—C7—C12—C3	-175.52 (19)
N3—C2—C3—C12	74.7 (3)	C8—C7—C12—C11	2.0 (3)
C1—N1—C14—C15	-165.9 (3)	C8—C9—C10—C11	1.2 (4)
C1—N2—N3—C2	-175.50 (18)	C9—C10—C11—C12	0.6 (3)
C2—C3—C4—O1	4.3 (3)	C10—C11—C12—C3	175.3 (2)
C2—C3—C4—C5	-177.72 (19)	C10—C11—C12—C7	-2.1 (3)
C2—C3—C12—C7	175.56 (18)	C12—C3—C4—O1	177.75 (18)
C2—C3—C12—C11	-1.9 (3)	C12—C3—C4—C5	-4.2 (3)
C3—C4—C5—C6	2.8 (3)	C12—C7—C8—C9	-0.3 (3)
C4—C3—C12—C7	2.1 (3)	C13—C2—C3—C4	71.2 (3)
C4—C3—C12—C11	-175.27 (19)	C13—C2—C3—C12	-102.2 (2)
C4—C5—C6—C7	0.9 (3)	C14—N1—C1—S1	3.2 (4)
C5—C6—C7—C8	173.9 (2)	C14—N1—C1—N2	-177.7 (3)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ S1 <sup>i</sup>	0.84	2.40	3.2349 (17)	171
C14—H14A $\cdots$ O1 <sup>ii</sup>	0.99	2.49	3.474 (4)	171
N2—H2 $\cdots$ S1 <sup>iii</sup>	0.88	2.79	3.548 (2)	145

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