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## Structure Reports

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**(2Z)-2-Benzylidene-4-*n*-butyl-3,4-dihydro-2H-1,4-benzothiazin-3-one**Nada Kheira Sebbar,<sup>a</sup> Mohammed El Fal,<sup>a\*</sup> El Mokhtar Essassi,<sup>a</sup> Mohamed Saadi<sup>b</sup> and Lahcen El Ammari<sup>b</sup><sup>a</sup>Laboratoire de Chimie Organique Hétérocyclique URAC 21, Pôle de Compétence Pharmacochimie, Av. Ibn Battouta, BP 1014, Faculté des Sciences, Université Mohammed V-Agdal, Rabat, Morocco, and <sup>b</sup>Laboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Université Mohammed V-Agdal, Avenue Ibn Battouta, BP 1014, Rabat, Morocco

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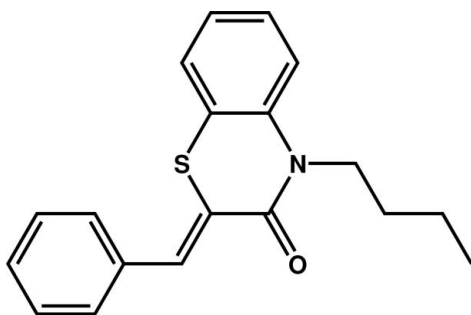
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.044;  $wR$  factor = 0.129; data-to-parameter ratio = 19.7.

In the title compound,  $\text{C}_{19}\text{H}_{19}\text{NOS}$ , the six-membered heterocyclic ring of the benzothiazine fragment exhibits a screw boat conformation. The plane of the fused benzene ring makes a dihedral angle of  $72.38$  ( $12$ )° with that of the terminal phenyl ring, and is nearly perpendicular to the mean plane formed by the atoms through the *n*-butyl chain, as indicated by the dihedral angle of  $88.1$  ( $2$ )°. In the crystal, molecules are linked by  $\text{C}-\text{H}\cdots\text{O}$  interactions to form supramolecular chains along  $[110]$ .

## Related literature

For the pharmaceutical and biochemical properties of benzothiazine and their derivatives, see: Malagu *et al.* (1998); Wammack *et al.* (2002); Rathore & Kumar (2006); Zia-ur-Rehman *et al.* (2009). For related structures, see: Sebbar *et al.* (2014); Saeed *et al.* (2010). For puckering calculations, see: Cremer & Pople (1975).



## Experimental

## Crystal data

$\text{C}_{19}\text{H}_{19}\text{NOS}$   
 $M_r = 309.41$   
 Triclinic,  $P\bar{1}$   
 $a = 8.7717$  (13) Å  
 $b = 8.8631$  (13) Å  
 $c = 12.3184$  (16) Å  
 $\alpha = 88.283$  (9)°  
 $\beta = 82.302$  (9)°  
 $\gamma = 60.895$  (8)°  
 $V = 828.5$  (2) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.20$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.37 \times 0.34 \times 0.28$  mm

## Data collection

Bruker X8 APEX diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2009)  
 $T_{\min} = 0.641$ ,  $T_{\max} = 0.746$   
 17374 measured reflections  
 3923 independent reflections  
 2912 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.129$   
 $S = 1.04$   
 3923 reflections  
 199 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.28$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.23$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C4}-\text{H4}\cdots\text{O1}^i$	0.93	2.50	3.407 (2)	165

Symmetry code: (i)  $x + 1, y - 1, z$ .

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT-Plus (Bruker, 2009); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: PLATON (Spek, 2009) and publCIF (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5313).

## References

- Bruker (2009). APEX2, SAINT-Plus and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Malagu, K., Boustie, J., David, M., Sauleau, J., Amoros, M., Girre, R. L. & Sauleau, A. (1998). *Pharm. Pharmacol. Commun.* **4**, 57–60.
- Rathore, B. S. & Kumar, M. (2006). *Bioorg. Med. Chem.* **14**, 5678–5682.
- Saeed, A., Mahmood, Z., Yang, S., Ahmad, S. & Salim, M. (2010). *Acta Cryst.* **E66**, o2289–o2290.
- Sebbar, N. K., Zerzouf, A., Essassi, E. M., Saadi, M. & El Ammari, L. (2014). *Acta Cryst.* **E70**, o160–o161.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Wammack, R., Remzi, M., Seitz, C., Djavan, B. & Marberger, M. (2002). *Eur. Urol.* **41**, 596–601.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Zia-ur-Rehman, M., Choudary, J. A., Elsegood, M. R. J., Siddiqui, H. L. & Khan, K. M. (2009). *Eur. J. Med. Chem.* **44**, 1311–1316.

## supporting information

*Acta Cryst.* (2014). E70, o686 [doi:10.1107/S160053681401054X]

**(2Z)-2-Benzylidene-4-*n*-butyl-3,4-dihydro-2H-1,4-benzothiazin-3-one**

**Nada Kheira Sebbar, Mohammed El Fal, El Mokhtar Essassi, Mohamed Saadi and Lahcen El Ammari**

**S1. Structural commentary**

Recently, a number of pharmacological tests revealed that benzothiazine derivatives present various biological activities. These derivatives are found potent analgesic (Wammack *et al.*, 2002); anti-viral (Malagu *et al.*, 1998; Rathore & Kumar, 2006) and anti-oxidant (Zia-ur-Rehman *et al.*, 2009). As a continuation of our research work devoted to the development of *N*-substituted benzothiazine with potential pharmacological activities, we have studied the action of 1-bromobutane towards 2-(benzylidene)-3,4-dihydro-2H-1,4-benzothiazin-3-one under phase transfer catalysis conditions using tetra *n*-butyl ammonium bromide as catalyst and potassium carbonate as base (Saeed *et al.*, 2010; Sebbar *et al.*, 2014) (Scheme 1).

The molecule of the title compound is build up from two fused six-membered rings linked to a phenyl ring and to a *n*-butyl chain as shown in Fig. 1. The benzothiazine fragment adopts a screw boat conformation as indicated by the puckering amplitude  $Q = 0.4701(14)$  Å, and spherical polar angle  $\theta = 70.21(19)^\circ$ , with  $\varphi = 333.4(2)^\circ$  (Cremer & Pople, 1975). The dihedral angle between the plane through the phenyl ring (C9 to C15) and the benzene ring (C1 to C6) is  $72.38(12)^\circ$ . The mean plane formed by the atoms belonging to the *n*-butyl chain (C16 to C19) is nearly perpendicular to the benzene ring as indicated by the dihedral angle between them of  $88.1(2)^\circ$ .

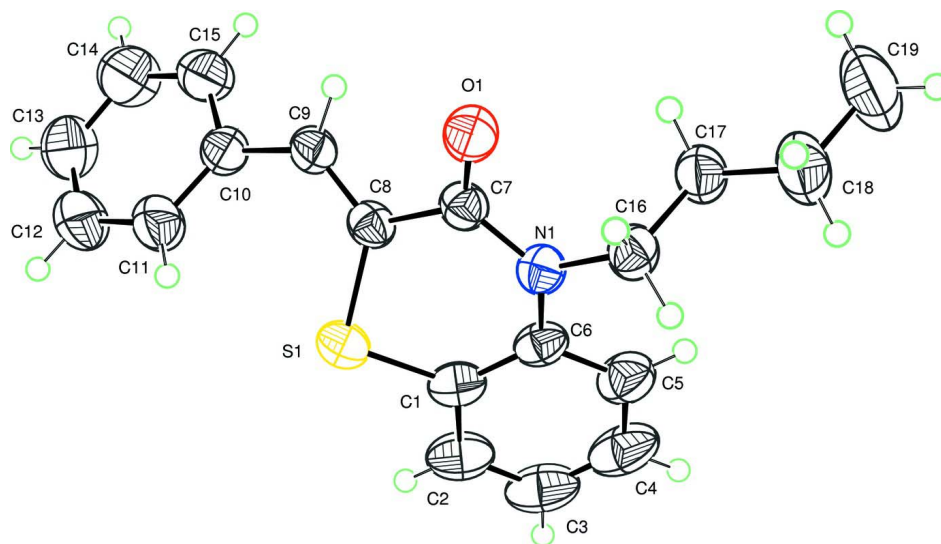
In the crystal, the molecules are linked by weak intermolecular C4–H4···O1 interactions, in a fashion to form chains along [1 1 0] (see Fig. 2 and Table 1).

**S2. Synthesis and crystallization**

To a solution of 2-(benzylidene)-3,4-dihydro-2H-1,4-benzothiazin-3-one (0.2 g, 0.7 mmol), potassium carbonate (0.4 g, 2.9 mmol) and tetra *n*-butyl ammonium bromide (0.024 g, 0.07 mmol) in DMF (15 ml) was added 1-bromobutane (0.20 ml, 1.89 mmol). Stirring was continued at room temperature for 24 h. The mixture was filtered and the solvent removed. The residue was washed with water. The organic compound was chromatographed on a column of silica gel with ethyl acetate-hexane (1/1) as eluent. Yellow crystals were isolated when the solvent was allowed to evaporate (yield = 48% and M.pt = 363 K).

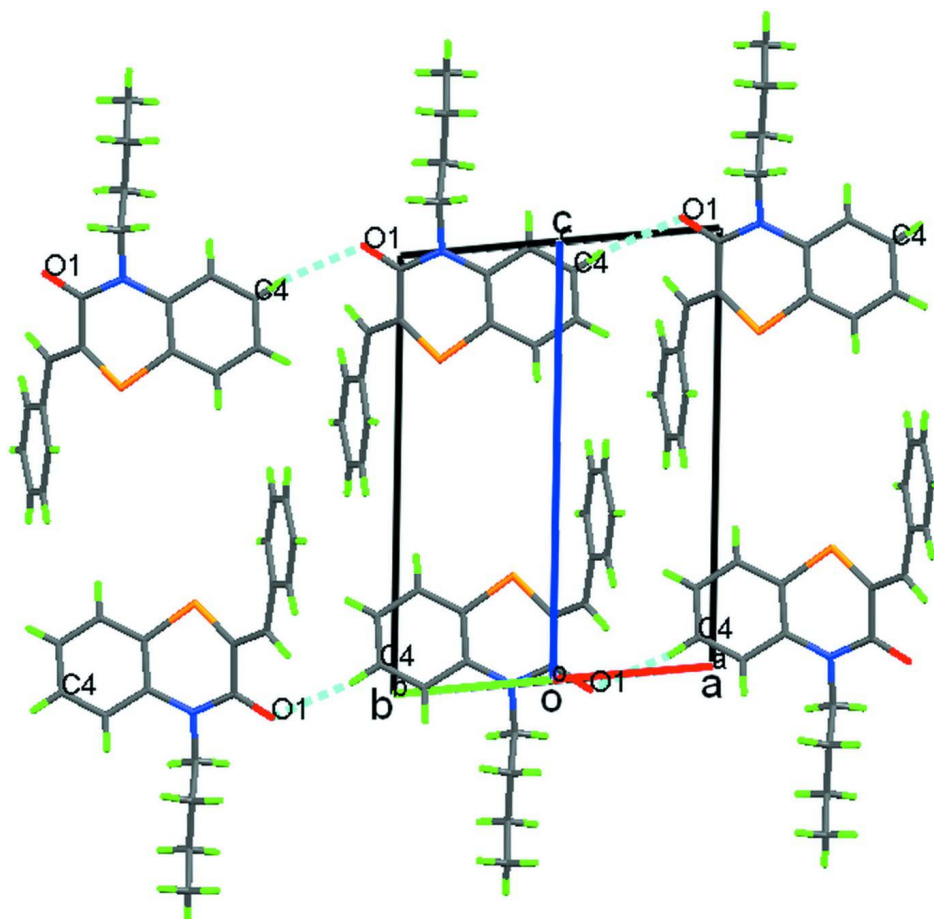
**S3. Refinement**

The H atoms were located in a difference map and treated as riding with C—H = 0.93 Å (aromatic), C—H = 0.97 Å (methylene) and C—H = 0.96 Å (methyl), and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$  (aromatic and methylene) and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}$  (methyl). The (0 0 1) reflection was omitted owing to poor agreement.



**Figure 1**

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

**Figure 2**

Structure projection along [1 1 0] of the title compound, showing molecules linked through C4–H4···O1 hydrogen bonds (dashed lines).

**(2Z)-2-Benzylidene-4-*n*-utyl-3,4-dihydro-2H-1,4-benzothiazin-3-one**

*Crystal data*

C<sub>19</sub>H<sub>19</sub>NOS

*M<sub>r</sub>* = 309.41

Triclinic, *P*1̄

Hall symbol: -P 1

*a* = 8.7717 (13) Å

*b* = 8.8631 (13) Å

*c* = 12.3184 (16) Å

$\alpha$  = 88.283 (9)°

$\beta$  = 82.302 (9)°

$\gamma$  = 60.895 (8)°

*V* = 828.5 (2) Å<sup>3</sup>

*Z* = 2

*F*(000) = 328

*D<sub>x</sub>* = 1.240 Mg m<sup>-3</sup>

Melting point: 363 K

Mo *K*α radiation,  $\lambda$  = 0.71073 Å

Cell parameters from 3923 reflections

$\theta$  = 2.6–27.9°

$\mu$  = 0.20 mm<sup>-1</sup>

*T* = 296 K

Block, yellow

0.37 × 0.34 × 0.28 mm

*Data collection*

Bruker X8 APEX  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator  
 $\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.641$ ,  $T_{\max} = 0.746$

17374 measured reflections

3923 independent reflections

2912 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 27.9^\circ$ ,  $\theta_{\min} = 2.6^\circ$

$h = -11 \rightarrow 11$

$k = -11 \rightarrow 11$

$l = -16 \rightarrow 16$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.129$

$S = 1.04$

3923 reflections

199 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-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.1635P]$

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

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

$\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

### 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.9210 (2)	0.3479 (2)	0.82655 (15)	0.0483 (4)
C2	1.0667 (3)	0.2113 (2)	0.76691 (19)	0.0648 (5)
H2	1.0839	0.2136	0.6909	0.078*
C3	1.1845 (3)	0.0746 (3)	0.8185 (2)	0.0758 (7)
H3	1.2794	-0.0181	0.7779	0.091*
C4	1.1622 (3)	0.0746 (3)	0.9299 (2)	0.0761 (7)
H4	1.2449	-0.0169	0.9653	0.091*
C5	1.0181 (3)	0.2089 (2)	0.99169 (18)	0.0622 (5)
H5	1.0048	0.2067	1.0678	0.075*
C6	0.8931 (2)	0.3472 (2)	0.93992 (14)	0.0447 (4)
C7	0.6448 (2)	0.6452 (2)	0.96790 (12)	0.0416 (3)
C8	0.69106 (19)	0.6857 (2)	0.85347 (12)	0.0408 (3)
C9	0.6528 (2)	0.8484 (2)	0.83178 (12)	0.0449 (4)
H9	0.5991	0.9273	0.8912	0.054*
C10	0.6840 (2)	0.9191 (2)	0.72724 (13)	0.0466 (4)
C11	0.6707 (3)	0.8635 (3)	0.62735 (15)	0.0647 (5)
H11	0.6374	0.7790	0.6247	0.078*
C12	0.7061 (3)	0.9318 (3)	0.53127 (16)	0.0779 (7)
H12	0.6960	0.8930	0.4646	0.093*

C13	0.7560 (3)	1.0563 (3)	0.53309 (18)	0.0795 (7)
H13	0.7860	1.0976	0.4680	0.095*
C14	0.7610 (4)	1.1182 (3)	0.6313 (2)	0.0900 (8)
H14	0.7893	1.2064	0.6335	0.108*
C15	0.7245 (3)	1.0520 (3)	0.72774 (17)	0.0720 (6)
H15	0.7271	1.0971	0.7942	0.086*
C16	0.6813 (2)	0.4450 (2)	1.11277 (13)	0.0498 (4)
H16A	0.7185	0.3226	1.1158	0.060*
H16B	0.5536	0.5075	1.1257	0.060*
C17	0.7506 (3)	0.4944 (3)	1.20408 (14)	0.0575 (5)
H17A	0.7010	0.6189	1.2084	0.069*
H17B	0.8776	0.4429	1.1879	0.069*
C18	0.7032 (4)	0.4335 (4)	1.31329 (17)	0.0835 (7)
H18A	0.5767	0.4786	1.3261	0.100*
H18B	0.7586	0.3084	1.3093	0.100*
C19	0.7580 (5)	0.4878 (5)	1.4086 (2)	0.1144 (11)
H19A	0.7221	0.4471	1.4750	0.172*
H19B	0.7032	0.6116	1.4136	0.172*
H19C	0.8837	0.4394	1.3983	0.172*
N1	0.73979 (17)	0.48044 (16)	1.00182 (10)	0.0425 (3)
O1	0.52331 (16)	0.75693 (15)	1.02912 (9)	0.0587 (3)
S1	0.77675 (6)	0.51831 (6)	0.75323 (3)	0.05429 (16)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0409 (8)	0.0423 (8)	0.0613 (10)	-0.0214 (7)	0.0011 (7)	-0.0073 (7)
C2	0.0519 (11)	0.0527 (10)	0.0814 (13)	-0.0224 (9)	0.0096 (10)	-0.0181 (10)
C3	0.0487 (11)	0.0488 (11)	0.114 (2)	-0.0139 (9)	0.0033 (12)	-0.0223 (12)
C4	0.0515 (11)	0.0422 (10)	0.127 (2)	-0.0129 (9)	-0.0271 (13)	0.0001 (11)
C5	0.0576 (11)	0.0471 (10)	0.0800 (13)	-0.0209 (9)	-0.0224 (10)	0.0038 (9)
C6	0.0390 (8)	0.0371 (8)	0.0612 (10)	-0.0204 (7)	-0.0090 (7)	-0.0005 (7)
C7	0.0403 (8)	0.0431 (8)	0.0394 (8)	-0.0188 (7)	-0.0045 (6)	-0.0009 (6)
C8	0.0358 (7)	0.0442 (8)	0.0376 (7)	-0.0160 (6)	-0.0026 (6)	-0.0032 (6)
C9	0.0464 (9)	0.0444 (8)	0.0371 (7)	-0.0174 (7)	-0.0028 (6)	-0.0013 (6)
C10	0.0470 (9)	0.0444 (8)	0.0422 (8)	-0.0184 (7)	-0.0021 (7)	0.0014 (7)
C11	0.0884 (14)	0.0673 (12)	0.0484 (10)	-0.0453 (11)	-0.0118 (10)	0.0071 (9)
C12	0.1082 (18)	0.0814 (15)	0.0432 (10)	-0.0467 (14)	-0.0055 (11)	0.0038 (10)
C13	0.1030 (18)	0.0853 (16)	0.0524 (12)	-0.0518 (14)	0.0037 (11)	0.0152 (11)
C14	0.139 (2)	0.0867 (16)	0.0709 (15)	-0.0779 (17)	-0.0069 (15)	0.0133 (12)
C15	0.1083 (17)	0.0646 (12)	0.0529 (11)	-0.0508 (12)	-0.0056 (11)	0.0009 (9)
C16	0.0588 (10)	0.0537 (10)	0.0468 (9)	-0.0350 (9)	-0.0085 (8)	0.0085 (7)
C17	0.0750 (13)	0.0627 (11)	0.0488 (9)	-0.0431 (10)	-0.0157 (9)	0.0101 (8)
C18	0.128 (2)	0.1015 (18)	0.0516 (11)	-0.0780 (17)	-0.0226 (12)	0.0191 (11)
C19	0.181 (3)	0.155 (3)	0.0538 (13)	-0.114 (3)	-0.0322 (17)	0.0187 (16)
N1	0.0422 (7)	0.0427 (7)	0.0425 (7)	-0.0207 (6)	-0.0057 (5)	0.0029 (5)
O1	0.0574 (7)	0.0498 (7)	0.0433 (6)	-0.0096 (6)	0.0075 (5)	-0.0014 (5)
S1	0.0622 (3)	0.0504 (3)	0.0421 (2)	-0.0221 (2)	-0.00101 (19)	-0.00837 (17)

*Geometric parameters (Å, °)*

C1—C6	1.386 (2)	C11—H11	0.9300
C1—C2	1.391 (2)	C12—C13	1.373 (4)
C1—S1	1.7505 (19)	C12—H12	0.9300
C2—C3	1.361 (3)	C13—C14	1.358 (3)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.360 (4)	C14—C15	1.377 (3)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.388 (3)	C15—H15	0.9300
C4—H4	0.9300	C16—N1	1.474 (2)
C5—C6	1.394 (2)	C16—C17	1.516 (2)
C5—H5	0.9300	C16—H16A	0.9700
C6—N1	1.421 (2)	C16—H16B	0.9700
C7—O1	1.2199 (18)	C17—C18	1.516 (3)
C7—N1	1.369 (2)	C17—H17A	0.9700
C7—C8	1.496 (2)	C17—H17B	0.9700
C8—C9	1.339 (2)	C18—C19	1.500 (3)
C8—S1	1.7514 (16)	C18—H18A	0.9700
C9—C10	1.466 (2)	C18—H18B	0.9700
C9—H9	0.9300	C19—H19A	0.9600
C10—C11	1.379 (3)	C19—H19B	0.9600
C10—C15	1.386 (3)	C19—H19C	0.9600
C11—C12	1.380 (3)		
C6—C1—C2	120.52 (18)	C14—C13—H13	120.5
C6—C1—S1	121.86 (12)	C12—C13—H13	120.5
C2—C1—S1	117.62 (16)	C13—C14—C15	120.8 (2)
C3—C2—C1	120.8 (2)	C13—C14—H14	119.6
C3—C2—H2	119.6	C15—C14—H14	119.6
C1—C2—H2	119.6	C14—C15—C10	121.0 (2)
C4—C3—C2	119.40 (19)	C14—C15—H15	119.5
C4—C3—H3	120.3	C10—C15—H15	119.5
C2—C3—H3	120.3	N1—C16—C17	114.51 (14)
C3—C4—C5	121.1 (2)	N1—C16—H16A	108.6
C3—C4—H4	119.5	C17—C16—H16A	108.6
C5—C4—H4	119.5	N1—C16—H16B	108.6
C4—C5—C6	120.2 (2)	C17—C16—H16B	108.6
C4—C5—H5	119.9	H16A—C16—H16B	107.6
C6—C5—H5	119.9	C16—C17—C18	110.99 (16)
C1—C6—C5	117.94 (16)	C16—C17—H17A	109.4
C1—C6—N1	121.31 (14)	C18—C17—H17A	109.4
C5—C6—N1	120.73 (16)	C16—C17—H17B	109.4
O1—C7—N1	120.87 (14)	C18—C17—H17B	109.4
O1—C7—C8	120.34 (14)	H17A—C17—H17B	108.0
N1—C7—C8	118.79 (13)	C19—C18—C17	113.8 (2)
C9—C8—C7	118.94 (14)	C19—C18—H18A	108.8
C9—C8—S1	123.81 (12)	C17—C18—H18A	108.8

C7—C8—S1	117.05 (12)	C19—C18—H18B	108.8
C8—C9—C10	128.90 (15)	C17—C18—H18B	108.8
C8—C9—H9	115.5	H18A—C18—H18B	107.7
C10—C9—H9	115.5	C18—C19—H19A	109.5
C11—C10—C15	117.53 (17)	C18—C19—H19B	109.5
C11—C10—C9	123.43 (17)	H19A—C19—H19B	109.5
C15—C10—C9	119.02 (16)	C18—C19—H19C	109.5
C10—C11—C12	120.8 (2)	H19A—C19—H19C	109.5
C10—C11—H11	119.6	H19B—C19—H19C	109.5
C12—C11—H11	119.6	C7—N1—C6	124.49 (13)
C13—C12—C11	120.6 (2)	C7—N1—C16	116.46 (13)
C13—C12—H12	119.7	C6—N1—C16	118.97 (13)
C11—C12—H12	119.7	C1—S1—C8	99.56 (8)
C14—C13—C12	119.0 (2)		

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C4—H4...O1 <sup>i</sup>	0.93	2.50	3.407 (2)	165

Symmetry code: (i)  $x+1, y-1, z$ .