

# Crystal structure of 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one 1-oxide

Hemant P. Yennawar,<sup>a</sup> Ryan Fox,<sup>b</sup> Quentin J. Moyer,<sup>b</sup> Ziwei Yang<sup>b</sup> and Lee J. Silverberg<sup>b\*</sup>

<sup>a</sup>Department of Biochemistry and Molecular Biology, Pennsylvania State University, University Park, PA 16802, USA, and

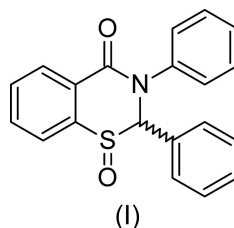
<sup>b</sup>Pennsylvania State University, Schuylkill Campus, 200 University Drive, Schuylkill Haven, PA 17972, USA.

\*Correspondence e-mail: ljs43@psu.edu

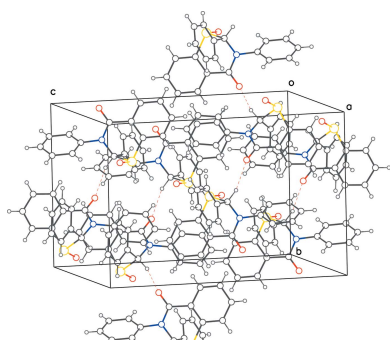
In the racemic title compound, C<sub>20</sub>H<sub>15</sub>NO<sub>2</sub>S, the planes of the two phenyl substituents form dihedral angles of 48.97 (15) and 69.26 (15)<sup>o</sup> with that of the fused benzene ring of the parent benzothiazine ring, while the heterocyclic thiazine ring exhibits a screw-boat pucker. The O atom on the S atom of the ring is pseudo-axial on the thiazine ring and *trans* to the 2-phenyl group. In the crystal, molecules are arranged in layers in the *ac* plane, the layers being linked across *b* through intermolecular C—H···O hydrogen-bonding interactions.

## 1. Chemical context

The 2,3-dihydro-4*H*-1,3-benzothiazin-4-one scaffold has shown a wide range of bioactivity, including antitumor (Li *et al.*, 2012; Wang *et al.*, 2015; Kamel *et al.*, 2010; Nofal *et al.*, 2014), antimicrobial (Popiolek *et al.*, 2016; Mandour *et al.*, 2007), antimalarial (Mei *et al.*, 2013), HIV–RT inhibition (Jeng *et al.*, 2015; Hou *et al.*, 2016) and cyclooxygenase COX-2 enzyme inhibition (Zarghi *et al.*, 2009). The *S*-oxides of these compounds have been little studied (a search found fewer than 50), despite the evidence of enhanced activity in the similar 2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-ones (Surrey *et al.*, 1958; Surrey, 1963*a,b*) and 1,3-thiazolidin-4-ones (Gududuru *et al.*, 2004). Also of potential interest is the triphenyltin chloride adduct, which may have enhanced antifungal activity (Eng *et al.*, 1996).



Recently, we reported the crystal structures of 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazine-4-one 1-oxide (Yennawar, Yang & Silverberg, 2016) and the 1:1 adduct of triphenyltin chloride and 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one (Yennawar, Fox & Silverberg, 2016). Attempts to prepare the triphenyltin chloride adduct of 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one instead produced the sulfoxide 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one 1-oxide on two separate occasions. The sulfoxide was also intentionally prepared by oxidation of 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one with Oxone<sup>®</sup>. It has not yet been determined how the sulfoxide formed in the tin reaction, but



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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C1-H1\cdots O2^i$	0.98	2.31	3.240 (3)	157

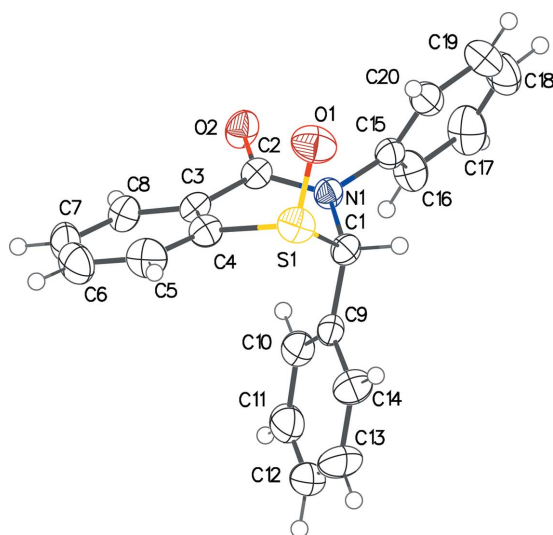
 Symmetry code: (i)  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$ .

Bourgoin-Legay & Boudet (1969) have reported the air oxidation of 2-alkyl-4*H*-1,3-benzothiazines to give the sulf-oxides, although the analogous 2-aryl compounds were less prone to air oxidation.

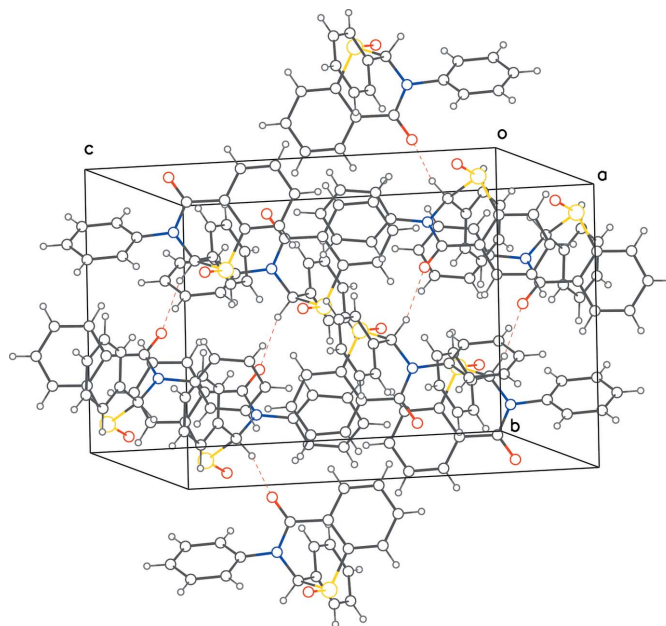
In this article, we report the crystal structure of the product from one of the reactions using tin, the title compound, namely 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one 1-oxide, (I). To the best of our knowledge, this is the first reported crystal structure of an *S*-oxide of a 2,3-dihydro-4*H*-1,3-benzothiazin-4-one.

## 2. Structural commentary

In the title racemic compound, the planes of the two phenyl substituents form dihedral angles of 48.97 (15) and 69.26 (15)° with that of the fused benzene ring of the parent benzothiazine system (Fig. 1). The O atom on the S atom is pseudo-axial and *trans* to the 2-phenyl ring, just as in 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one 1-oxide (Yennawar, Yang & Silverberg, 2016). The thiazine ring has a screw-boat conformation, with a puckering amplitude of 0.686 (2) Å and  $\theta = 65.6$  (2)° (Cremer & Pople, 1975). The thiazine ring in 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one 1-oxide (Yennawar, Yang & Silverberg, 2016) was in an envelope conformation. The overall molecular configuration is quite similar to the structure of 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one (Yennawar *et al.*, 2014).



**Figure 1**  
 The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.



**Figure 2**  
 Crystal packing diagram showing  $C-H\cdots O$  contacts as dotted red lines between molecules of (I) which form chains along the *b*-axis direction.

## 3. Supramolecular features

The crystal lattice has layers of molecules comprising alternating enantiomers, extending along the *a*-axis direction and lying in the *ac* plane. The layers are linked across the *b*-cell direction through intermolecular  $C1-H\cdots O2^i$  hydrogen bonds (Fig. 2, Table 1) between molecules of the same chirality [symmetry code: (i)  $-x + \frac{1}{2}, y - \frac{3}{2}, -z + \frac{1}{2}$ ]. While  $C-H\cdots O$  interactions are also present in our two earlier structures (Yennawar *et al.*, 2014; Yennawar, Yang & Silverberg, 2016), the differences in either the donor C or acceptor O atoms make them unique in each case. In the present structure, the chiral C atom donates the proton to the O atom at position 4 ( $\cdots O-C$ ) of the thiazine ring, while in our 2016 structure, the acceptor O atom was the one at position 1 ( $\cdots O-S$ ). In the 2014 structure, the two benzene-ring C atoms are the donors to the only O atom ( $\cdots O-C$ ) on the thiazine ring.

## 4. Database survey

A literature search found no prior reports of a crystal structure of an *S*-oxide of a 2,3-dihydro-4*H*-1,3-benzothiazin-4-one. We have previously reported the crystal structures of 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one 1-oxide (Yennawar, Yang & Silverberg, 2016) and 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one (Yennawar *et al.*, 2014).

## 5. Synthesis and crystallization

A 2 ml reactival with a stir bar was charged with 0.1004 g of 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one and 0.95 ml of acetone and stirred. The benzothiazinone did not fully dissolve. A 10 ml round-bottomed flask was charged with

0.1212 g of triphenyltin chloride and 2.0 ml of acetone and stirred. The contents of the 2 ml vial were added to the 10 ml flask and the vial was rinsed with an additional 0.5 ml of acetone, giving a clear solution, which was stirred for 2 h and then allowed to stand without stirring for 3 d. The solution was filtered through Celite and then concentrated under vacuum, giving a white solid. The solid was recrystallized from cyclohexane to give a yellow solid (yield 0.0755 g, 72%). Crystals suitable for X-ray analysis were obtained by slow evaporation from an acetone solution.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms were placed geometrically and allowed to ride on their parent C atoms during refinement, with C–H distances of 0.98 (methine) or 0.93 Å (aromatic) and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . Although of no particular significance in this racemic compound, the enantiomer chosen was the C1(S) one.

## Funding information

Funding for this research was provided by: Penn State Schuylkill; National Science Foundation (grant No. CHEM-0131112) for the X-ray diffractometer.

## References

- Bourgoin-Legay, D. & Boudet, R. (1969). *Bull. Soc. Chim. Fr.* **7**, 2524–2530.
- Bourhis, L. J., Dolomanov, O. V., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2015). *Acta Cryst.* **A71**, 59–75.
- Bruker (2001). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Eng, G., Whalen, D., Zhang, Y. Z., Tierney, J., Jiang, X. & May, L. (1996). *Appl. Organomet. Chem.* **10**, 495–499.
- Gududuru, V., Hurh, E., Dalton, J. T. & Miller, D. D. (2004). *Bioorg. Med. Chem. Lett.* **14**, 5289–5293.
- Hou, Y., Xing, S., Shao, J., Yin, Z., Hao, L., Yang, T., Zhang, H., Zhu, M., Chen, H. & Li, X. (2016). *Carbohydr. Res.* **429**, 105–112.
- Jeng, F., Li, X., Shao, J., Zhu, M., Li, Y., Hua, C. & Xiaoliu, L. (2015). *Chin. J. Org. Chem.* **35**, 1370–1374.
- Kamel, M. M., Ali, H. I., Anwar, M. M., Mohamed, N. A. & Soliman, A. M. M. (2010). *Eur. J. Med. Chem.* **45**, 572–580.
- Li, Q., Li, C., Lu, X., Zhang, H. & Zhu, H. (2012). *Eur. J. Med. Chem.* **50**, 288–295.
- Mandour, A. H., El-Sawy, E. R., Ebid, M. S. & El-Sayed, Z. G. (2007). *Egypt. J. Chem.* **50**, 555–568.

**Table 2**

Experimental details.

Crystal data	
Chemical formula	C <sub>20</sub> H <sub>15</sub> NO <sub>2</sub> S
$M_r$	333.39
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	298
$a, b, c$ (Å)	9.1505 (16), 11.2712 (19), 16.379 (3)
$\beta$ (°)	103.997 (6)
$V$ (Å <sup>3</sup> )	1639.2 (5)
$Z$	4
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.21
Crystal size (mm)	0.20 × 0.16 × 0.14
Data collection	
Diffractometer	Bruker CCD area detector
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2001)
$T_{\text{min}}, T_{\text{max}}$	0.790, 0.9
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	12730, 4036, 3701
$R_{\text{int}}$	0.025
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.090, 0.231, 1.65
No. of reflections	4036
No. of parameters	217
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.61, -0.34

Computer programs: *SMART* (Bruker, 2001), *SAINT* (Bruker, 2001), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL97* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

- Mei, Z., Wang, L., Lu, W., Pang, C., Maeda, T., Peng, W., Kaiser, M., El Sayed, I. & Inokuchi, T. (2013). *J. Med. Chem.* **56**, 1431–1442.
- Nofal, Z. M., Soliman, E. A., El-Karim, A., El-Zahar, M. I., Srour, A. M., Sethumadhavan, S. & Maher, T. J. (2014). *J. Heterocycl. Chem.* **51**, 1797–1806.
- Popiolek, L., Biernasiuk, A. & Malm, A. (2016). *J. Heterocycl. Chem.* **53**, 479–486.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Surrey, A. R. (1963*a*). US Patent 3082209.
- Surrey, A. R. (1963*b*). US Patent 3093639.
- Surrey, A. R., Webb, W. G. & Gesler, R. M. (1958). *J. Am. Chem. Soc.* **80**, 3469–3471.
- Wang, S., Fang, K., Dong, G., Chen, S., Liu, N., Miao, Z., Yao, J., Li, J., Zhang, W. & Sheng, C. (2015). *J. Med. Chem.* **58**, 6678–6696.
- Yennawar, H. P., Bendinsky, R. V., Coyle, D. J., Cali, A. S. & Silverberg, L. J. (2014). *Acta Cryst.* **E70**, o465.
- Yennawar, H. P., Fox, R. & Silverberg, L. J. (2016). *Acta Cryst.* **E72**, 276–279.
- Yennawar, H. P., Yang, Z. & Silverberg, L. J. (2016). *Acta Cryst.* **E72**, 1541–1543.
- Zarghi, A., Zebardast, T., Daraie, B. & Hedayati, M. (2009). *Bioorg. Med. Chem.* **17**, 5369–5373.

## supporting information

*Acta Cryst.* (2017). E73, 1189-1191 [https://doi.org/10.1107/S2056989017010313]

## Crystal structure of 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one 1-oxide

Hemant P. Yennawar, Ryan Fox, Quentin J. Moyer, Ziwei Yang and Lee J. Silverberg

### Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SMART* (Bruker, 2001); program(s) used to solve structure: *olex2.solve* (Bourhis *et al.*, 2015); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

### *rac*-2,3-Diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one 1-oxide

#### Crystal data

C<sub>20</sub>H<sub>15</sub>NO<sub>2</sub>S

*M<sub>r</sub>* = 333.39

Monoclinic, *P2<sub>1</sub>/n*

*a* = 9.1505 (16) Å

*b* = 11.2712 (19) Å

*c* = 16.379 (3) Å

β = 103.997 (6)°

*V* = 1639.2 (5) Å<sup>3</sup>

*Z* = 4

*F*(000) = 696

*D<sub>x</sub>* = 1.351 Mg m<sup>-3</sup>

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 975 reflections

θ = 2.9–28.1°

μ = 0.21 mm<sup>-1</sup>

*T* = 298 K

Block, colorless

0.20 × 0.16 × 0.14 mm

#### Data collection

Bruker CCD area detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2001)

*T<sub>min</sub>* = 0.790, *T<sub>max</sub>* = 0.9

12730 measured reflections

4036 independent reflections

3701 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.025

θ<sub>max</sub> = 28.4°, θ<sub>min</sub> = 2.2°

*h* = -12→11

*k* = -14→14

*l* = -21→21

#### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.090

*wR*(*F*<sup>2</sup>) = 0.231

*S* = 1.65

4036 reflections

217 parameters

0 restraints

Primary atom site location: iterative

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.1*P*)<sup>2</sup>]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> < 0.001

Δρ<sub>max</sub> = 0.61 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.34 e Å<sup>-3</sup>

*Special details*

**Experimental.** The data collection nominally covered a full sphere of reciprocal space by a combination of 4 sets of  $\omega$  scans each set at different  $\phi$  and/or  $2\theta$  angles and each scan (10 s exposure) covering  $-0.300^\circ$  degrees in  $\omega$ . The crystal to detector distance was 5.82 cm.

**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.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2072 (3)	0.6272 (2)	0.31199 (16)	0.0321 (5)
H1	0.1959	0.5611	0.2720	0.039*
C2	0.3510 (3)	0.8148 (2)	0.32892 (15)	0.0295 (5)
C3	0.3394 (3)	0.8157 (2)	0.41882 (15)	0.0303 (5)
C4	0.3293 (3)	0.7126 (2)	0.46419 (16)	0.0341 (6)
C5	0.3268 (3)	0.7173 (3)	0.54783 (18)	0.0473 (7)
H5	0.3222	0.6477	0.5776	0.057*
C6	0.3311 (4)	0.8261 (3)	0.58734 (19)	0.0516 (8)
H6	0.3298	0.8298	0.6439	0.062*
C7	0.3373 (3)	0.9288 (3)	0.54312 (18)	0.0467 (7)
H7	0.3384	1.0019	0.5696	0.056*
C8	0.3420 (3)	0.9239 (2)	0.45941 (17)	0.0384 (6)
H8	0.3470	0.9939	0.4301	0.046*
C9	0.0503 (3)	0.6611 (2)	0.31841 (15)	0.0339 (6)
C10	-0.0061 (3)	0.7748 (3)	0.30135 (18)	0.0434 (7)
H10	0.0541	0.8338	0.2868	0.052*
C11	-0.1521 (4)	0.8012 (3)	0.3059 (2)	0.0562 (9)
H11	-0.1896	0.8776	0.2938	0.067*
C12	-0.2407 (4)	0.7160 (4)	0.3280 (2)	0.0648 (10)
H12	-0.3379	0.7346	0.3316	0.078*
C13	-0.1875 (4)	0.6036 (4)	0.3447 (3)	0.0673 (10)
H13	-0.2485	0.5458	0.3599	0.081*
C14	-0.0414 (4)	0.5746 (3)	0.3393 (2)	0.0526 (8)
H14	-0.0061	0.4974	0.3498	0.063*
C15	0.3197 (3)	0.7045 (2)	0.19894 (16)	0.0320 (5)
C16	0.2398 (4)	0.7703 (3)	0.13225 (17)	0.0446 (7)
H16	0.1642	0.8216	0.1393	0.053*
C17	0.2729 (5)	0.7592 (3)	0.05491 (19)	0.0574 (9)
H17	0.2198	0.8038	0.0096	0.069*
C18	0.3845 (4)	0.6825 (3)	0.0442 (2)	0.0582 (9)
H18	0.4082	0.6770	-0.0077	0.070*
C19	0.4602 (4)	0.6144 (3)	0.1105 (2)	0.0548 (8)
H19	0.5333	0.5610	0.1030	0.066*

C20	0.4279 (3)	0.6249 (3)	0.18879 (18)	0.0430 (7)
H20	0.4787	0.5787	0.2338	0.052*
N1	0.2910 (2)	0.71883 (18)	0.28149 (13)	0.0304 (5)
O1	0.4735 (2)	0.5462 (2)	0.39909 (15)	0.0535 (6)
O2	0.4139 (2)	0.89553 (17)	0.30137 (12)	0.0421 (5)
S1	0.32220 (8)	0.57148 (6)	0.41423 (4)	0.0392 (3)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0354 (12)	0.0251 (11)	0.0372 (13)	-0.0058 (9)	0.0112 (10)	-0.0019 (10)
C2	0.0296 (11)	0.0270 (11)	0.0303 (12)	0.0005 (9)	0.0041 (9)	0.0034 (9)
C3	0.0273 (11)	0.0307 (12)	0.0309 (13)	-0.0032 (9)	0.0032 (9)	0.0014 (9)
C4	0.0332 (12)	0.0328 (12)	0.0351 (13)	0.0028 (10)	0.0059 (10)	0.0033 (10)
C5	0.0507 (16)	0.0536 (17)	0.0366 (15)	0.0039 (13)	0.0087 (12)	0.0134 (13)
C6	0.0555 (18)	0.068 (2)	0.0303 (14)	0.0079 (15)	0.0083 (13)	-0.0011 (14)
C7	0.0493 (16)	0.0513 (17)	0.0372 (15)	0.0007 (13)	0.0062 (12)	-0.0131 (13)
C8	0.0415 (14)	0.0347 (14)	0.0370 (14)	-0.0045 (10)	0.0058 (11)	-0.0013 (11)
C9	0.0342 (12)	0.0395 (14)	0.0284 (12)	-0.0097 (10)	0.0079 (10)	-0.0089 (10)
C10	0.0387 (14)	0.0467 (15)	0.0431 (15)	0.0005 (12)	0.0065 (12)	-0.0077 (13)
C11	0.0443 (17)	0.070 (2)	0.0495 (18)	0.0094 (15)	0.0029 (14)	-0.0180 (16)
C12	0.0364 (15)	0.101 (3)	0.058 (2)	-0.0051 (18)	0.0132 (14)	-0.032 (2)
C13	0.0468 (18)	0.089 (3)	0.072 (2)	-0.0270 (19)	0.0265 (17)	-0.011 (2)
C14	0.0475 (16)	0.0529 (19)	0.0606 (19)	-0.0145 (14)	0.0196 (15)	-0.0049 (15)
C15	0.0343 (12)	0.0315 (12)	0.0312 (12)	-0.0057 (9)	0.0101 (10)	-0.0017 (10)
C16	0.0587 (18)	0.0383 (14)	0.0378 (15)	0.0025 (12)	0.0136 (13)	0.0016 (12)
C17	0.085 (2)	0.0500 (18)	0.0366 (16)	-0.0038 (17)	0.0139 (15)	0.0080 (14)
C18	0.078 (2)	0.063 (2)	0.0414 (17)	-0.0122 (17)	0.0293 (17)	-0.0071 (15)
C19	0.0516 (18)	0.065 (2)	0.0541 (19)	0.0021 (15)	0.0252 (15)	-0.0110 (16)
C20	0.0379 (14)	0.0530 (17)	0.0380 (15)	0.0047 (12)	0.0089 (11)	-0.0052 (12)
N1	0.0350 (10)	0.0270 (10)	0.0305 (10)	-0.0046 (8)	0.0106 (8)	-0.0016 (8)
O1	0.0475 (12)	0.0473 (12)	0.0673 (14)	0.0153 (9)	0.0169 (10)	0.0093 (10)
O2	0.0545 (12)	0.0346 (10)	0.0385 (10)	-0.0164 (8)	0.0139 (9)	0.0009 (8)
S1	0.0453 (4)	0.0278 (4)	0.0463 (4)	0.0023 (2)	0.0145 (3)	0.0098 (3)

*Geometric parameters (Å, °)*

C1—H1	0.9800	C10—C11	1.388 (4)
C1—C9	1.514 (3)	C11—H11	0.9300
C1—N1	1.446 (3)	C11—C12	1.361 (5)
C1—S1	1.858 (3)	C12—H12	0.9300
C2—C3	1.502 (3)	C12—C13	1.361 (6)
C2—N1	1.367 (3)	C13—H13	0.9300
C2—O2	1.220 (3)	C13—C14	1.400 (5)
C3—C4	1.394 (3)	C14—H14	0.9300
C3—C8	1.386 (3)	C15—C16	1.375 (4)
C4—C5	1.377 (4)	C15—C20	1.375 (4)
C4—S1	1.783 (3)	C15—N1	1.447 (3)

C5—H5	0.9300	C16—H16	0.9300
C5—C6	1.383 (4)	C16—C17	1.378 (4)
C6—H6	0.9300	C17—H17	0.9300
C6—C7	1.374 (4)	C17—C18	1.381 (5)
C7—H7	0.9300	C18—H18	0.9300
C7—C8	1.383 (4)	C18—C19	1.373 (5)
C8—H8	0.9300	C19—H19	0.9300
C9—C10	1.384 (4)	C19—C20	1.389 (4)
C9—C14	1.383 (4)	C20—H20	0.9300
C10—H10	0.9300	O1—S1	1.491 (2)
C9—C1—H1	106.9	C12—C11—H11	119.8
C9—C1—S1	111.28 (17)	C11—C12—H12	119.9
N1—C1—H1	106.9	C13—C12—C11	120.2 (3)
N1—C1—C9	115.8 (2)	C13—C12—H12	119.9
N1—C1—S1	108.68 (16)	C12—C13—H13	119.8
S1—C1—H1	106.9	C12—C13—C14	120.4 (3)
N1—C2—C3	116.7 (2)	C14—C13—H13	119.8
O2—C2—C3	120.6 (2)	C9—C14—C13	119.8 (3)
O2—C2—N1	122.7 (2)	C9—C14—H14	120.1
C4—C3—C2	123.1 (2)	C13—C14—H14	120.1
C8—C3—C2	118.6 (2)	C16—C15—N1	120.0 (2)
C8—C3—C4	118.3 (2)	C20—C15—C16	121.0 (3)
C3—C4—S1	119.9 (2)	C20—C15—N1	119.0 (2)
C5—C4—C3	121.2 (2)	C15—C16—H16	120.4
C5—C4—S1	118.9 (2)	C15—C16—C17	119.2 (3)
C4—C5—H5	120.2	C17—C16—H16	120.4
C4—C5—C6	119.6 (3)	C16—C17—H17	119.8
C6—C5—H5	120.2	C16—C17—C18	120.5 (3)
C5—C6—H6	120.0	C18—C17—H17	119.8
C7—C6—C5	120.0 (3)	C17—C18—H18	120.1
C7—C6—H6	120.0	C19—C18—C17	119.8 (3)
C6—C7—H7	119.9	C19—C18—H18	120.1
C6—C7—C8	120.3 (3)	C18—C19—H19	119.9
C8—C7—H7	119.9	C18—C19—C20	120.2 (3)
C3—C8—H8	119.7	C20—C19—H19	119.9
C7—C8—C3	120.6 (2)	C15—C20—C19	119.2 (3)
C7—C8—H8	119.7	C15—C20—H20	120.4
C10—C9—C1	122.2 (2)	C19—C20—H20	120.4
C14—C9—C1	118.8 (3)	C1—N1—C15	118.32 (19)
C14—C9—C10	119.0 (3)	C2—N1—C1	122.9 (2)
C9—C10—H10	119.9	C2—N1—C15	118.7 (2)
C9—C10—C11	120.3 (3)	C4—S1—C1	93.56 (11)
C11—C10—H10	119.9	O1—S1—C1	105.12 (12)
C10—C11—H11	119.8	O1—S1—C4	108.31 (13)
C12—C11—C10	120.4 (3)		
C1—C9—C10—C11	178.3 (2)	C15—C16—C17—C18	0.4 (5)

C1—C9—C14—C13	-179.3 (3)	C16—C15—C20—C19	2.4 (4)
C2—C3—C4—C5	176.6 (2)	C16—C15—N1—C1	105.0 (3)
C2—C3—C4—S1	-2.5 (3)	C16—C15—N1—C2	-78.4 (3)
C2—C3—C8—C7	-177.6 (2)	C16—C17—C18—C19	1.7 (5)
C3—C2—N1—C1	5.9 (3)	C17—C18—C19—C20	-1.9 (5)
C3—C2—N1—C15	-170.5 (2)	C18—C19—C20—C15	-0.2 (5)
C3—C4—C5—C6	1.3 (4)	C20—C15—C16—C17	-2.5 (4)
C3—C4—S1—C1	-36.3 (2)	C20—C15—N1—C1	-75.2 (3)
C3—C4—S1—O1	70.9 (2)	C20—C15—N1—C2	101.3 (3)
C4—C3—C8—C7	1.1 (4)	N1—C1—C9—C10	-4.0 (3)
C4—C5—C6—C7	0.3 (5)	N1—C1—C9—C14	173.9 (2)
C5—C4—S1—C1	144.5 (2)	N1—C1—S1—C4	60.72 (18)
C5—C4—S1—O1	-108.3 (2)	N1—C1—S1—O1	-49.3 (2)
C5—C6—C7—C8	-1.2 (5)	N1—C2—C3—C4	26.3 (3)
C6—C7—C8—C3	0.5 (4)	N1—C2—C3—C8	-155.1 (2)
C8—C3—C4—C5	-2.0 (4)	N1—C15—C16—C17	177.3 (3)
C8—C3—C4—S1	178.89 (19)	N1—C15—C20—C19	-177.4 (2)
C9—C1—N1—C2	73.2 (3)	O2—C2—C3—C4	-152.3 (3)
C9—C1—N1—C15	-110.4 (2)	O2—C2—C3—C8	26.3 (3)
C9—C1—S1—C4	-67.89 (18)	O2—C2—N1—C1	-175.5 (2)
C9—C1—S1—O1	-177.94 (17)	O2—C2—N1—C15	8.1 (4)
C9—C10—C11—C12	0.7 (4)	S1—C1—C9—C10	120.8 (2)
C10—C9—C14—C13	-1.4 (4)	S1—C1—C9—C14	-61.4 (3)
C10—C11—C12—C13	-0.8 (5)	S1—C1—N1—C2	-52.8 (3)
C11—C12—C13—C14	-0.1 (6)	S1—C1—N1—C15	123.56 (19)
C12—C13—C14—C9	1.3 (5)	S1—C4—C5—C6	-179.5 (2)
C14—C9—C10—C11	0.5 (4)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C1—H1...O2 <sup>i</sup>	0.98	2.31	3.240 (3)	157

Symmetry code: (i)  $-x+1/2, y-1/2, -z+1/2$ .