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Technology, Australia**Keywords:** 1,3-thiazin-4-one; twisted half-chair
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crystal structure.**CCDC reference:** 1571357**Supporting information:** this article has
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Crystal structure of (1*S*,2*S*,5*R*)-5-acetylamino-4-oxo-2,3-diphenyl-1,3-thiazinan-1-ium-1-olate

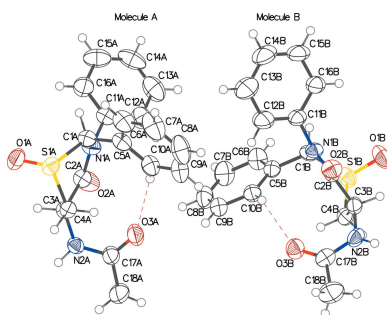
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The asymmetric unit of the enantiomerically pure title compound, C₁₈H₁₈N₂O₃S, comprises two independent molecules (*A* and *B*) having almost identical conformations. When overlaid, the alignment-r.m.s. deviation value is 0.30 Å. The six-membered heterocycle has a twisted half-chair conformation in both molecules. The O atom on the S atom of the ring is pseudo-axial on the thiazine ring and *trans* to both a phenyl group substituent and the acetamide group in each case. The two benzene rings in each molecule are almost orthogonal to each other, with interplanar dihedral angles of 83.79 (17) and 86.95 (16)°. The acetamide group is pseudo-equatorial and a phenyl ring is pseudo-axial on the thiazine ring. Both molecules show a weak intramolecular C—H···O interaction between H-atom donors of one of the phenyl rings and the acetamide group. In the crystal, an intermolecular N—H···O(thiazine) hydrogen bond links *B* molecules along the 2₁ (*b*) screw axis and, in addition, an N—H···O(acetamide) hydrogen bond links *A* and *B* molecules across *a*. A two-dimensional layered structure lying parallel to (001) is generated, also involving weak intermolecular C—H···O interactions.

1. Chemical context

The 1,3-thiazin-4-ones are a group of six-membered heterocycles with a wide range of biological activity (Ryabukhin *et al.*, 1996). Surrey's research (Surrey *et al.*, 1958; Surrey, 1963*a,b*) resulted in the discovery of two drugs, the anti-anxiety and muscle relaxant chlormezanone [2-(4-chlorophenyl)-3-methyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one 1,1-dioxide] (O'Neil, 2006; Tanaka & Horayama, 2005) and muscle relaxant dichloromezanone [2-(3,4-dichlorophenyl)-3-methyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one 1,1-dioxide] (Elks & Ganellin, 1990). These sulfoxides showed greater activity than the sulfides from which they were synthesized (Surrey *et al.*, 1958). Surrey also prepared a variety of other sulfoxides and sulfones of 3-alkyl-2-aryl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-ones (Surrey, 1963*a,b*). We have reported previously the crystal structure of the first *N*-aryl sulfoxide in this family, racemic 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one 1-oxide (Yennawar *et al.*, 2016).

A sulfoxide typically has an S—O bond that is between a double bond and a single bond, with one of the lone pairs that was on the sulfide coordinating to the O atom, while O atom contributes electrons from a lone pair to a *d* orbital of the S atom. The geometry of a sulfoxide is pyramidal, with a high energy barrier for inversion, making it possible to isolate stable enantiomers (Bentley, 2005). Herein, we report the crystal structure of the sulfoxide of *N*-[(2*S*,5*R*)-4-oxo-2,3-diphenyl-1,3-thiazinan-5-yl]acetamide (Yennawar, Singh & Silverberg, 2015), C₁₈H₁₈N₂O₃S, prepared using the method



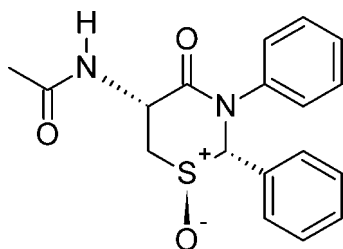
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Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2A-H2A\cdots O2B^i$	0.91 (3)	2.25 (3)	3.137 (3)	164 (2)
$N2B-H2B\cdots O1B^{ii}$	0.79 (3)	2.14 (3)	2.916 (3)	168 (3)
$C10A-H10A\cdots O3A$	0.93	2.42	3.259 (3)	149
$C10B-H10B\cdots O3B$	0.93	2.44	3.232 (4)	143
$C4B-H4BB\cdots O2A^{iii}$	0.97	2.25	3.116 (3)	148

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

we have reported previously for the oxidation of other 2,3-diphenyl-1,3-thiazin-4-ones (Yennawar *et al.*, 2016; Yennawar, Noble *et al.*, 2017) and 1,3-thiazolidinones (Yennawar, Hullihen *et al.*, 2015; Cannon *et al.*, 2015). The oxidation of the confirmed enantiopure sulfide *N*-[(2*S*,5*R*)-4-oxo-2,3-diphenyl-1,3-thiazinan-5-yl]acetamide 0.375-hydrate (Yennawar, Singh & Silverberg, 2015), derived from *N*-acetyl-L-cysteine, yielded a single stereoisomer as the only product.



2. Structural commentary

The crystal structure of the title compound has two independent homochiral molecules (*A* and *B*) in the asymmetric unit (Fig. 1), which have almost identical conformational features, having an alignment-r.m.s. deviation value of 0.3 Å. Both have

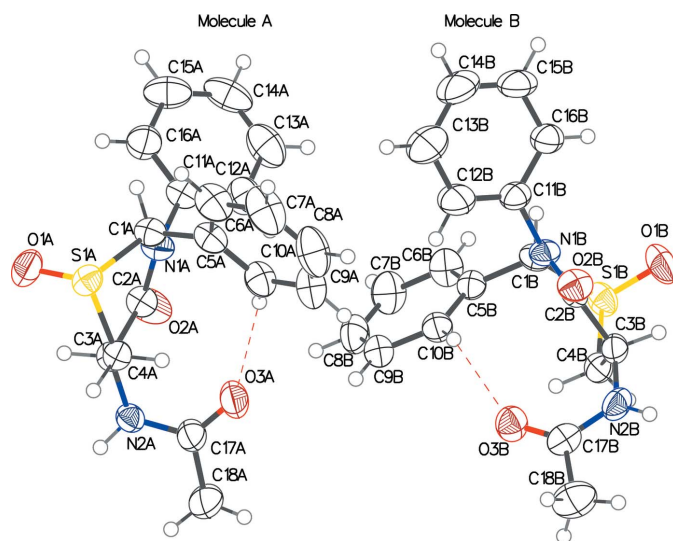


Figure 1
The molecular structures of the two independent molecules (*A* and *B*) in the asymmetric unit of the title compound, with displacement ellipsoids drawn at the 50% probability level. Dashed lines indicate intramolecular $C-H\cdots O$ interactions.

the thiazine rings in a twisted half-chair configuration, with puckering amplitudes = 0.6753 (19)/0.653 (2) Å and $\theta = 131.05$ (17)/135.66 (18)° in molecules *A/B*, respectively (Cremer & Pople, 1975). The O atom on the S atom of the ring is pseudo-axial on the thiazine ring and *trans* to both the 2-phenyl group and the acetamide group in each case. The two phenyl rings in each molecule are almost orthogonal to one another, with dihedral angles of 83.79 (17) and 86.95 (16)° in molecules *A* and *B*, respectively. The acetamide group is pseudo-equatorial and the 2-phenyl group is pseudo-axial on the thiazine ring. A weak intramolecular $C-H\cdots O$ hydrogen bond between the 2-phenyl ring and the O atom of the acetamide group is seen in both molecules ($C10A-H\cdots O3A$ and $C10B-H\cdots O3B$), as detailed in Table 1.

We reported previously the crystal structure of the starting sulfide, *N*-[(2*S*,5*R*)-4-oxo-2,3-diphenyl-1,3-thiazinan-5-yl]acetamide 0.375-hydrate (Yennawar, Singh & Silverberg, 2015), which also had two independent homochiral molecules in the asymmetric unit. However, they were not identical: in one molecule, the thiazine ring was in a half-chair conformation in which the 2-phenyl ring was nearly pseudo-axial and the acetamide group was nearly pseudo-equatorial. The other molecule had the thiazine ring in a boat conformation in which both substituents were pseudo-equatorial.

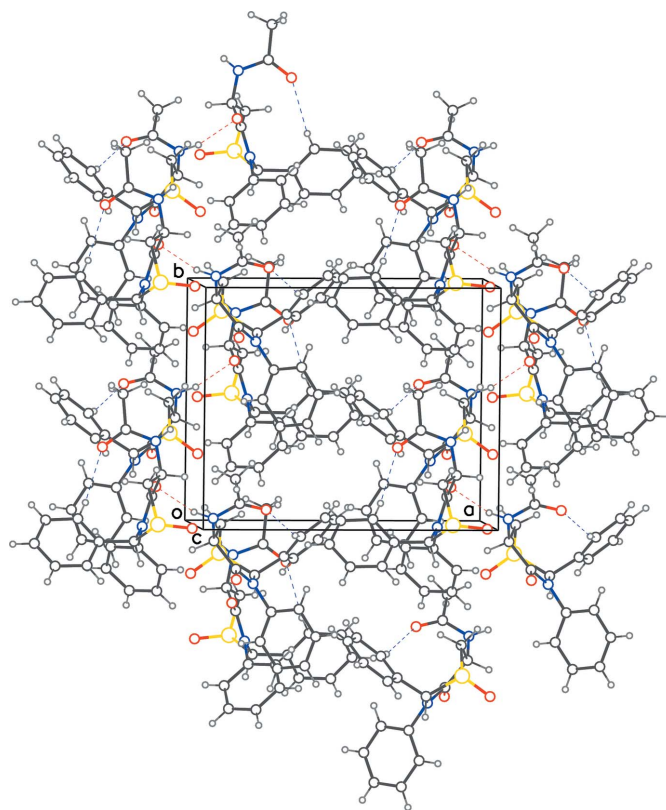


Figure 2
Crystal packing diagram with red dotted lines for intermolecular $N-H\cdots O$ contacts between 2_1 -related molecules, forming helical chains along the *b*-axis direction, as well as the interaction with an independent molecule. Blue dotted lines represent the intramolecular $C-H\cdots O$ contacts.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₈ H ₁₈ N ₂ O ₃ S
<i>M_r</i>	342.40
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁
Temperature (K)	298
<i>a</i> , <i>b</i> , <i>c</i> (Å)	12.872 (6), 10.139 (5), 13.460 (6)
β (°)	103.104 (9)
<i>V</i> (Å ³)	1710.8 (14)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.21
Crystal size (mm)	0.23 × 0.20 × 0.19
Data collection	
Diffractometer	Bruker SCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2016)
<i>T_{min}</i> , <i>T_{max}</i>	0.309, 0.900
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	15296, 8079, 6949
<i>R_{int}</i>	0.031
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.666
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.049, 0.128, 1.02
No. of reflections	8079
No. of parameters	443
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.37, -0.27
Absolute structure	Flack (1983), 4160 Friedel pairs
Absolute structure parameter	0.07 (6)

Computer programs: *SMART* (Bruker, 2016), *SAINT* (Bruker, 2016), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

3. Supramolecular features

In the crystal, the *B* molecule and its 2₁-related symmetry neighbours form a continuous hydrogen-bonded chain along the *b*-cell direction through N—H \cdots O interactions involving the acetamide N atom and the thiazin-1-ium-1-olate O atoms [N2*B*—H \cdots O1*B*ⁱⁱⁱ; symmetry code: (ii) $-x, y + \frac{1}{2}, -z$; Table 1] (Fig. 2). Molecules *A* and *B* interact, wherein the O atom in the 4-position of molecule *B* accepts a proton from the acetamide N atom of molecule *A* [N2*A*—H \cdots O1*B*ⁱ; symmetry code: (i) $x + 1, y, z$]. The sulfoxide O atom of molecule *A* does not participate in any hydrogen bonding. A two-dimensional sheet structure lying parallel to (001) is generated. No benzene ring in either of the molecules participates in face-to-face π – π stacking interactions.

4. Database survey

Crystal structures of a number of 1,3-thiazolidin-4-one 1-oxides have been reported (Wang *et al.*, 2010; Johnson *et al.*, 1983; Chen *et al.*, 2011; Colombo *et al.*, 2008; Yennawar, Hullihen *et al.*, 2015) and the structure of chlormezanone [2-(4-chlorophenyl)-3-methyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one 1,1-dioxide] has also been reported (Tanaka & Horayama, 2005). We have reported previously the crystal

structure of 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one 1-oxide (Yennawar *et al.*, 2016). We have also reported recently the crystal structures of 2,3-diphenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one 1-oxide (Yennawar, Fox *et al.*, 2017) and 2,3-diphenyl-2,3-dihydro-4*H*-pyrido[3,2-*e*][1,3]thiazin-4-one 1-oxide (Yennawar, Noble *et al.*, 2017).

5. Synthesis and crystallization

A 5 ml round-bottomed flask was charged with 53.9 mg of *N*-[(2*S*,5*R*)-4-oxo-2,3-diphenyl-1,3-thiazin-5-yl]acetamide 0.375-hydrate, whose configuration was established previously (Yennawar, Singh & Silverberg, 2015), and 1.4 ml of methanol and stirred. A solution of 79.5 mg of Oxone[®] and 1 ml of distilled water was added dropwise and the mixture was stirred until the reaction was complete, as determined by thin-layer chromatography (TLC). The solids were dissolved by the addition of 5 ml of distilled water. The solution was extracted with 10 ml of dichloromethane. The organic layer was washed with 5 ml of distilled water and then with 5 ml of saturated sodium chloride. The solution was dried over Na₂SO₄ and concentrated under vacuum giving a crude solid. This was chromatographed on flash silica gel, eluting with a gradient of 0–60% acetone in ethyl acetate, giving 55.8 mg of product [98.6% yield; m.p. 449–452 K; *R_F* = 0.20 (30% acetone/70% ethyl acetate)]. Crystals suitable for X-ray crystallography were grown by slow evaporation from propan-2-ol.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms, excepting those on N atoms, were placed geometrically and allowed to ride on their parent C atoms during refinement, with C—H distances of 0.93 (aromatic), 0.96 (methyl), 0.97 or (methylene) and 0.98 Å (methyl), and with *U*_{iso}(H) = 1.2*U*_{eq}(aromatic or methylene C) or 1.5*U*_{eq}(methyl C). H atoms on N atoms were located in a difference Fourier map and were refined isotropically. The absolute configuration for the chiral centres in the molecule was determined as (1*S*,2*S*,5*R*) (for the arbitrarily numbered atoms C1*A*/*B*, C3*A*/*B*), with a Flack absolute structure parameter (Flack, 1983) of 0.07 (6) for 4160 Friedel pairs.

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Acta Cryst. (2017). E73, 1417-1420 [https://doi.org/10.1107/S2056989017012488]

Crystal structure of (1*S*,2*S*,5*R*)-5-acetylamino-4-oxo-2,3-diphenyl-1,3-thiazinan-1-ium-1-olate

Hemant P. Yennawar, Duncan J. Noble and Lee J. Silverberg

Computing details

Data collection: *SMART* (Bruker, 2016); cell refinement: *SAINTE* (Bruker, 2016); data reduction: *SAINTE* (Bruker, 2016); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); 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).

(1*S*,2*S*,5*R*)-5-Acetylamino-4-oxo-2,3-diphenyl-1,3-thiazinan-1-ium-1-olate

Crystal data

C₁₈H₁₈N₂O₃S

M_r = 342.40

Monoclinic, *P2*₁

a = 12.872 (6) Å

b = 10.139 (5) Å

c = 13.460 (6) Å

β = 103.104 (9)°

V = 1710.8 (14) Å³

Z = 4

F(000) = 720

D_x = 1.329 Mg m⁻³

Melting point = 449–452 K

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 7433 reflections

θ = 2.5–28.2°

μ = 0.21 mm⁻¹

T = 298 K

Block, colorless

0.23 × 0.20 × 0.19 mm

Data collection

Bruker SCD area detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2016)

*T*_{min} = 0.309, *T*_{max} = 0.900

15296 measured reflections

8079 independent reflections

6949 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.031

θ_{max} = 28.2°, θ_{min} = 2.0°

h = -16→17

k = -13→13

l = -17→17

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.049

wR(*F*²) = 0.128

S = 1.02

8079 reflections

443 parameters

1 restraint

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

$$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.37 \text{ e } \text{Å}^{-3}$

$$\Delta\rho_{\min} = -0.27 \text{ e } \text{Å}^{-3}$$

Absolute structure: Flack (1983), 4160 Friedel pairs
 Absolute structure parameter: 0.07 (6)

Special details

Experimental. The data collection nominally covered a full sphere of reciprocal space by a combination of 4 sets of ω scans each set at different φ and/or 2θ angles and each scan (10 s exposure) covering -0.300° degrees in ω . 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 $F^2 > 2\text{sigma}(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1A	0.79355 (19)	0.2939 (3)	0.48969 (16)	0.0397 (5)
H1A	0.7893	0.2095	0.5241	0.048*
C2A	0.86711 (19)	0.3246 (3)	0.33058 (17)	0.0393 (5)
C3A	0.93950 (18)	0.4386 (2)	0.37719 (15)	0.0344 (5)
H3A	1.0109	0.4009	0.4010	0.041*
C4A	0.91189 (19)	0.5047 (2)	0.46957 (15)	0.0356 (5)
H4AA	0.8426	0.5468	0.4499	0.043*
H4AB	0.9645	0.5718	0.4964	0.043*
C5A	0.69426 (19)	0.3706 (3)	0.49564 (17)	0.0431 (5)
C6A	0.6572 (2)	0.3596 (4)	0.5857 (2)	0.0598 (8)
H6A	0.6895	0.3011	0.6365	0.072*
C7A	0.5723 (3)	0.4365 (5)	0.5981 (2)	0.0759 (11)
H7A	0.5479	0.4297	0.6579	0.091*
C8A	0.5243 (3)	0.5217 (5)	0.5243 (3)	0.0763 (10)
H8A	0.4674	0.5728	0.5340	0.092*
C9A	0.5592 (2)	0.5335 (4)	0.4343 (2)	0.0636 (8)
H9A	0.5262	0.5926	0.3841	0.076*
C10A	0.6433 (2)	0.4567 (3)	0.4200 (2)	0.0497 (6)
H10A	0.6658	0.4627	0.3592	0.060*
C11A	0.7454 (2)	0.1476 (2)	0.34101 (17)	0.0398 (5)
C16A	0.7794 (2)	0.0238 (3)	0.3750 (2)	0.0563 (7)
H16A	0.8400	0.0136	0.4273	0.068*
C15A	0.7227 (3)	-0.0862 (3)	0.3309 (3)	0.0719 (10)
H15A	0.7447	-0.1701	0.3546	0.086*
C14A	0.6350 (3)	-0.0716 (4)	0.2531 (3)	0.0709 (10)
H14A	0.5983	-0.1455	0.2228	0.085*
C13A	0.6014 (3)	0.0507 (4)	0.2197 (3)	0.0734 (10)
H13A	0.5411	0.0602	0.1671	0.088*

C12A	0.6561 (2)	0.1616 (4)	0.2634 (2)	0.0588 (7)
H12A	0.6325	0.2453	0.2403	0.071*
C17A	0.8618 (2)	0.5983 (3)	0.2465 (2)	0.0487 (6)
C18A	0.8817 (3)	0.7020 (4)	0.1734 (3)	0.0784 (11)
H18A	0.9497	0.7427	0.1999	0.118*
H18B	0.8266	0.7676	0.1648	0.118*
H18C	0.8814	0.6621	0.1087	0.118*
N1A	0.80481 (17)	0.26229 (19)	0.38641 (14)	0.0384 (4)
N2A	0.94797 (16)	0.5352 (2)	0.29910 (14)	0.0392 (4)
H2A	1.014 (2)	0.561 (3)	0.2912 (19)	0.036 (7)*
O1A	1.00529 (16)	0.2968 (2)	0.56800 (15)	0.0581 (5)
O2A	0.87065 (18)	0.2840 (2)	0.24629 (13)	0.0615 (6)
O3A	0.77158 (16)	0.5727 (3)	0.25622 (17)	0.0702 (7)
S1A	0.91010 (5)	0.38151 (6)	0.56562 (4)	0.04051 (15)
C1B	0.22990 (19)	0.4488 (2)	0.06076 (17)	0.0389 (5)
H1B	0.2075	0.3562	0.0584	0.047*
C2B	0.16558 (17)	0.6316 (2)	0.16162 (17)	0.0357 (5)
C3B	0.13964 (19)	0.7241 (3)	0.06778 (18)	0.0410 (5)
H3B	0.0625	0.7164	0.0407	0.049*
C4B	0.1904 (2)	0.6890 (3)	-0.02038 (17)	0.0418 (5)
H4BA	0.2672	0.6983	0.0009	0.050*
H4BB	0.1649	0.7488	-0.0769	0.050*
C5B	0.34898 (18)	0.4469 (3)	0.06755 (16)	0.0377 (5)
C6B	0.3913 (2)	0.3500 (3)	0.0150 (2)	0.0528 (7)
H6B	0.3468	0.2870	-0.0229	0.063*
C7B	0.4994 (3)	0.3473 (3)	0.0189 (2)	0.0615 (8)
H7B	0.5269	0.2836	-0.0177	0.074*
C8B	0.5669 (2)	0.4381 (4)	0.0764 (2)	0.0578 (7)
H8B	0.6397	0.4350	0.0791	0.069*
C9B	0.5264 (2)	0.5332 (3)	0.1298 (2)	0.0499 (6)
H9B	0.5721	0.5941	0.1692	0.060*
C10B	0.4174 (2)	0.5390 (3)	0.12537 (18)	0.0424 (5)
H10B	0.3903	0.6043	0.1609	0.051*
C11B	0.2058 (2)	0.4150 (2)	0.23392 (18)	0.0419 (5)
C12B	0.2934 (3)	0.4162 (4)	0.3126 (2)	0.0722 (10)
H12B	0.3466	0.4787	0.3143	0.087*
C13B	0.3032 (4)	0.3236 (5)	0.3903 (3)	0.0893 (13)
H13B	0.3635	0.3232	0.4436	0.107*
C14B	0.2239 (3)	0.2330 (4)	0.3880 (3)	0.0775 (11)
H14B	0.2307	0.1705	0.4397	0.093*
C15B	0.1358 (3)	0.2339 (3)	0.3110 (3)	0.0669 (9)
H15B	0.0818	0.1729	0.3107	0.080*
C16B	0.1249 (2)	0.3260 (3)	0.2317 (2)	0.0505 (6)
H16B	0.0644	0.3268	0.1787	0.061*
C17B	0.2575 (2)	0.9072 (3)	0.13509 (19)	0.0458 (6)
C18B	0.2671 (4)	1.0542 (3)	0.1506 (3)	0.0763 (10)
H18D	0.3007	1.0727	0.2204	0.114*
H18E	0.1974	1.0932	0.1342	0.114*

H18F	0.3095	1.0903	0.1069	0.114*
N1B	0.19754 (16)	0.5070 (2)	0.14898 (14)	0.0381 (4)
N2B	0.15836 (19)	0.8610 (2)	0.09612 (17)	0.0469 (5)
H2B	0.110 (2)	0.910 (3)	0.089 (2)	0.042 (8)*
O1B	0.04145 (15)	0.5076 (3)	-0.06247 (17)	0.0666 (6)
O2B	0.14719 (15)	0.66808 (19)	0.24289 (14)	0.0484 (4)
O3B	0.33503 (15)	0.83467 (19)	0.15418 (15)	0.0530 (5)
S1B	0.15756 (5)	0.52300 (7)	-0.06050 (4)	0.04663 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.0472 (13)	0.0435 (13)	0.0292 (10)	-0.0069 (11)	0.0101 (9)	-0.0013 (9)
C2A	0.0442 (12)	0.0432 (13)	0.0316 (10)	-0.0011 (10)	0.0108 (9)	-0.0049 (9)
C3A	0.0338 (11)	0.0389 (12)	0.0300 (10)	-0.0003 (9)	0.0065 (8)	-0.0027 (9)
C4A	0.0397 (11)	0.0343 (12)	0.0319 (9)	0.0014 (9)	0.0062 (9)	-0.0056 (8)
C5A	0.0441 (12)	0.0518 (14)	0.0355 (11)	-0.0110 (12)	0.0135 (10)	-0.0064 (11)
C6A	0.0595 (16)	0.083 (2)	0.0409 (13)	-0.0101 (16)	0.0195 (12)	-0.0040 (14)
C7A	0.0571 (18)	0.125 (3)	0.0529 (16)	-0.0064 (19)	0.0280 (15)	-0.0207 (19)
C8A	0.0464 (17)	0.110 (3)	0.073 (2)	0.0079 (19)	0.0156 (15)	-0.025 (2)
C9A	0.0417 (14)	0.084 (2)	0.0621 (16)	0.0076 (16)	0.0050 (12)	-0.0074 (17)
C10A	0.0429 (13)	0.0638 (18)	0.0428 (12)	-0.0026 (13)	0.0106 (10)	-0.0038 (12)
C11A	0.0426 (13)	0.0425 (13)	0.0367 (11)	-0.0081 (11)	0.0137 (9)	-0.0063 (10)
C16A	0.0598 (17)	0.0445 (15)	0.0607 (16)	-0.0018 (14)	0.0055 (13)	-0.0077 (13)
C15A	0.100 (3)	0.0442 (18)	0.076 (2)	-0.0166 (17)	0.029 (2)	-0.0106 (14)
C14A	0.084 (2)	0.070 (2)	0.0665 (19)	-0.0399 (19)	0.0320 (19)	-0.0292 (17)
C13A	0.062 (2)	0.094 (3)	0.0601 (18)	-0.0213 (19)	0.0051 (15)	-0.0221 (18)
C12A	0.0604 (18)	0.0622 (19)	0.0468 (14)	-0.0062 (14)	-0.0028 (13)	-0.0074 (13)
C17A	0.0390 (13)	0.0625 (18)	0.0442 (13)	-0.0003 (12)	0.0085 (11)	0.0123 (12)
C18A	0.0573 (18)	0.097 (3)	0.081 (2)	0.0043 (19)	0.0149 (17)	0.050 (2)
N1A	0.0482 (11)	0.0372 (11)	0.0311 (9)	-0.0086 (9)	0.0115 (8)	-0.0076 (7)
N2A	0.0349 (10)	0.0446 (12)	0.0388 (9)	-0.0033 (9)	0.0102 (8)	0.0027 (9)
O1A	0.0572 (11)	0.0548 (12)	0.0561 (11)	0.0131 (10)	-0.0005 (9)	0.0087 (9)
O2A	0.0785 (14)	0.0743 (14)	0.0388 (9)	-0.0276 (12)	0.0280 (10)	-0.0233 (9)
O3A	0.0353 (10)	0.103 (2)	0.0692 (13)	-0.0017 (10)	0.0059 (9)	0.0341 (13)
S1A	0.0493 (3)	0.0405 (3)	0.0289 (2)	0.0010 (3)	0.0029 (2)	-0.0018 (2)
C1B	0.0428 (12)	0.0363 (12)	0.0386 (11)	-0.0073 (10)	0.0114 (10)	-0.0046 (9)
C2B	0.0284 (10)	0.0408 (12)	0.0396 (11)	-0.0031 (9)	0.0111 (9)	0.0040 (9)
C3B	0.0320 (11)	0.0487 (14)	0.0423 (12)	0.0031 (10)	0.0083 (9)	0.0088 (10)
C4B	0.0400 (12)	0.0535 (15)	0.0296 (10)	-0.0044 (11)	0.0030 (9)	0.0073 (10)
C5B	0.0390 (12)	0.0417 (13)	0.0334 (10)	0.0012 (10)	0.0099 (9)	0.0018 (9)
C6B	0.0530 (15)	0.0572 (18)	0.0474 (14)	0.0036 (13)	0.0099 (11)	-0.0136 (12)
C7B	0.0560 (16)	0.070 (2)	0.0626 (17)	0.0151 (15)	0.0223 (14)	-0.0136 (15)
C8B	0.0397 (14)	0.074 (2)	0.0604 (16)	0.0124 (14)	0.0134 (12)	0.0068 (15)
C9B	0.0410 (13)	0.0488 (15)	0.0568 (14)	-0.0004 (12)	0.0047 (11)	0.0037 (13)
C10B	0.0423 (12)	0.0381 (13)	0.0465 (12)	0.0000 (10)	0.0093 (10)	-0.0022 (10)
C11B	0.0459 (13)	0.0422 (14)	0.0414 (12)	0.0020 (10)	0.0178 (10)	0.0077 (9)
C12B	0.070 (2)	0.082 (3)	0.0588 (17)	-0.0207 (17)	0.0013 (15)	0.0301 (17)

C13B	0.096 (3)	0.104 (3)	0.061 (2)	-0.010 (2)	0.0021 (19)	0.040 (2)
C14B	0.090 (3)	0.075 (2)	0.075 (2)	0.011 (2)	0.034 (2)	0.0384 (19)
C15B	0.067 (2)	0.0498 (18)	0.097 (2)	0.0029 (15)	0.046 (2)	0.0232 (17)
C16B	0.0485 (14)	0.0436 (15)	0.0639 (17)	0.0020 (12)	0.0224 (13)	0.0070 (12)
C17B	0.0572 (16)	0.0417 (15)	0.0410 (12)	0.0062 (12)	0.0160 (11)	0.0010 (10)
C18B	0.098 (3)	0.0469 (19)	0.084 (2)	0.0031 (17)	0.021 (2)	-0.0066 (16)
N1B	0.0428 (10)	0.0384 (11)	0.0360 (9)	-0.0021 (8)	0.0148 (8)	0.0055 (8)
N2B	0.0459 (12)	0.0432 (13)	0.0541 (12)	0.0160 (11)	0.0163 (10)	0.0089 (10)
O1B	0.0388 (10)	0.0814 (16)	0.0722 (13)	-0.0158 (11)	-0.0029 (9)	-0.0075 (12)
O2B	0.0558 (11)	0.0522 (11)	0.0435 (9)	0.0023 (8)	0.0246 (8)	0.0015 (8)
O3B	0.0478 (10)	0.0490 (11)	0.0607 (11)	0.0041 (8)	0.0093 (9)	-0.0076 (8)
S1B	0.0416 (3)	0.0590 (4)	0.0362 (3)	-0.0103 (3)	0.0023 (2)	-0.0070 (3)

Geometric parameters (Å, °)

C1A—H1A	0.9800	C1B—H1B	0.9800
C1A—C5A	1.514 (4)	C1B—C5B	1.515 (3)
C1A—N1A	1.465 (3)	C1B—N1B	1.468 (3)
C1A—S1A	1.841 (3)	C1B—S1B	1.846 (3)
C2A—C3A	1.527 (3)	C2B—C3B	1.547 (3)
C2A—N1A	1.371 (3)	C2B—N1B	1.351 (3)
C2A—O2A	1.217 (3)	C2B—O2B	1.227 (3)
C3A—H3A	0.9800	C3B—H3B	0.9800
C3A—C4A	1.524 (3)	C3B—C4B	1.521 (3)
C3A—N2A	1.459 (3)	C3B—N2B	1.445 (4)
C4A—H4AA	0.9700	C4B—H4BA	0.9700
C4A—H4AB	0.9700	C4B—H4BB	0.9700
C4A—S1A	1.801 (2)	C4B—S1B	1.788 (3)
C5A—C6A	1.404 (3)	C5B—C6B	1.392 (4)
C5A—C10A	1.388 (4)	C5B—C10B	1.394 (4)
C6A—H6A	0.9300	C6B—H6B	0.9300
C6A—C7A	1.383 (5)	C6B—C7B	1.380 (4)
C7A—H7A	0.9300	C7B—H7B	0.9300
C7A—C8A	1.355 (6)	C7B—C8B	1.377 (5)
C8A—H8A	0.9300	C8B—H8B	0.9300
C8A—C9A	1.389 (5)	C8B—C9B	1.374 (4)
C9A—H9A	0.9300	C9B—H9B	0.9300
C9A—C10A	1.382 (4)	C9B—C10B	1.392 (4)
C10A—H10A	0.9300	C10B—H10B	0.9300
C11A—C16A	1.372 (4)	C11B—C12B	1.361 (4)
C11A—C12A	1.375 (4)	C11B—C16B	1.372 (4)
C11A—N1A	1.449 (3)	C11B—N1B	1.460 (3)
C16A—H16A	0.9300	C12B—H12B	0.9300
C16A—C15A	1.390 (5)	C12B—C13B	1.389 (5)
C15A—H15A	0.9300	C13B—H13B	0.9300
C15A—C14A	1.362 (6)	C13B—C14B	1.369 (6)
C14A—H14A	0.9300	C14B—H14B	0.9300
C14A—C13A	1.356 (6)	C14B—C15B	1.353 (5)

C13A—H13A	0.9300	C15B—H15B	0.9300
C13A—C12A	1.385 (5)	C15B—C16B	1.401 (4)
C12A—H12A	0.9300	C16B—H16B	0.9300
C17A—C18A	1.502 (4)	C17B—C18B	1.505 (4)
C17A—N2A	1.336 (3)	C17B—N2B	1.350 (4)
C17A—O3A	1.226 (3)	C17B—O3B	1.219 (3)
C18A—H18A	0.9600	C18B—H18D	0.9600
C18A—H18B	0.9600	C18B—H18E	0.9600
C18A—H18C	0.9600	C18B—H18F	0.9600
N2A—H2A	0.91 (3)	N2B—H2B	0.79 (3)
O1A—S1A	1.491 (2)	O1B—S1B	1.497 (2)
C5A—C1A—H1A	106.6	C5B—C1B—H1B	106.0
C5A—C1A—S1A	108.30 (17)	C5B—C1B—S1B	111.05 (15)
N1A—C1A—H1A	106.6	N1B—C1B—H1B	106.0
N1A—C1A—C5A	115.4 (2)	N1B—C1B—C5B	115.16 (19)
N1A—C1A—S1A	112.87 (15)	N1B—C1B—S1B	111.87 (17)
S1A—C1A—H1A	106.6	S1B—C1B—H1B	106.0
N1A—C2A—C3A	120.12 (19)	N1B—C2B—C3B	118.6 (2)
O2A—C2A—C3A	119.4 (2)	O2B—C2B—C3B	119.7 (2)
O2A—C2A—N1A	120.3 (2)	O2B—C2B—N1B	121.3 (2)
C2A—C3A—H3A	106.2	C2B—C3B—H3B	105.6
C4A—C3A—C2A	115.76 (18)	C4B—C3B—C2B	116.3 (2)
C4A—C3A—H3A	106.2	C4B—C3B—H3B	105.6
N2A—C3A—C2A	110.52 (18)	N2B—C3B—C2B	112.0 (2)
N2A—C3A—H3A	106.2	N2B—C3B—H3B	105.6
N2A—C3A—C4A	111.2 (2)	N2B—C3B—C4B	110.8 (2)
C3A—C4A—H4AA	109.9	C3B—C4B—H4BA	109.7
C3A—C4A—H4AB	109.9	C3B—C4B—H4BB	109.7
C3A—C4A—S1A	108.90 (16)	C3B—C4B—S1B	109.99 (17)
H4AA—C4A—H4AB	108.3	H4BA—C4B—H4BB	108.2
S1A—C4A—H4AA	109.9	S1B—C4B—H4BA	109.7
S1A—C4A—H4AB	109.9	S1B—C4B—H4BB	109.7
C6A—C5A—C1A	117.5 (3)	C6B—C5B—C1B	119.1 (2)
C10A—C5A—C1A	123.3 (2)	C6B—C5B—C10B	119.1 (2)
C10A—C5A—C6A	119.1 (3)	C10B—C5B—C1B	121.8 (2)
C5A—C6A—H6A	120.2	C5B—C6B—H6B	119.9
C7A—C6A—C5A	119.5 (3)	C7B—C6B—C5B	120.1 (3)
C7A—C6A—H6A	120.2	C7B—C6B—H6B	119.9
C6A—C7A—H7A	119.6	C6B—C7B—H7B	119.6
C8A—C7A—C6A	120.8 (3)	C8B—C7B—C6B	120.7 (3)
C8A—C7A—H7A	119.6	C8B—C7B—H7B	119.6
C7A—C8A—H8A	119.6	C7B—C8B—H8B	120.1
C7A—C8A—C9A	120.7 (3)	C9B—C8B—C7B	119.8 (3)
C9A—C8A—H8A	119.6	C9B—C8B—H8B	120.1
C8A—C9A—H9A	120.3	C8B—C9B—H9B	119.8
C10A—C9A—C8A	119.4 (3)	C8B—C9B—C10B	120.4 (3)
C10A—C9A—H9A	120.3	C10B—C9B—H9B	119.8

C5A—C10A—H10A	119.8	C5B—C10B—H10B	120.0
C9A—C10A—C5A	120.5 (3)	C9B—C10B—C5B	119.9 (2)
C9A—C10A—H10A	119.8	C9B—C10B—H10B	120.0
C16A—C11A—C12A	119.8 (3)	C12B—C11B—C16B	120.9 (3)
C16A—C11A—N1A	119.7 (2)	C12B—C11B—N1B	120.3 (2)
C12A—C11A—N1A	120.5 (3)	C16B—C11B—N1B	118.8 (2)
C11A—C16A—H16A	120.2	C11B—C12B—H12B	120.1
C11A—C16A—C15A	119.6 (3)	C11B—C12B—C13B	119.9 (3)
C15A—C16A—H16A	120.2	C13B—C12B—H12B	120.1
C16A—C15A—H15A	119.9	C12B—C13B—H13B	120.1
C14A—C15A—C16A	120.3 (3)	C14B—C13B—C12B	119.8 (4)
C14A—C15A—H15A	119.9	C14B—C13B—H13B	120.1
C15A—C14A—H14A	120.0	C13B—C14B—H14B	119.9
C13A—C14A—C15A	120.0 (3)	C15B—C14B—C13B	120.3 (3)
C13A—C14A—H14A	120.0	C15B—C14B—H14B	119.9
C14A—C13A—H13A	119.7	C14B—C15B—H15B	119.7
C14A—C13A—C12A	120.6 (3)	C14B—C15B—C16B	120.7 (3)
C12A—C13A—H13A	119.7	C16B—C15B—H15B	119.7
C11A—C12A—C13A	119.7 (3)	C11B—C16B—C15B	118.5 (3)
C11A—C12A—H12A	120.2	C11B—C16B—H16B	120.7
C13A—C12A—H12A	120.2	C15B—C16B—H16B	120.7
N2A—C17A—C18A	116.0 (2)	N2B—C17B—C18B	116.0 (3)
O3A—C17A—C18A	121.6 (3)	O3B—C17B—C18B	122.0 (3)
O3A—C17A—N2A	122.4 (2)	O3B—C17B—N2B	121.9 (2)
C17A—C18A—H18A	109.5	C17B—C18B—H18D	109.5
C17A—C18A—H18B	109.5	C17B—C18B—H18E	109.5
C17A—C18A—H18C	109.5	C17B—C18B—H18F	109.5
H18A—C18A—H18B	109.5	H18D—C18B—H18E	109.5
H18A—C18A—H18C	109.5	H18D—C18B—H18F	109.5
H18B—C18A—H18C	109.5	H18E—C18B—H18F	109.5
C2A—N1A—C1A	127.96 (19)	C2B—N1B—C1B	128.89 (19)
C2A—N1A—C11A	117.19 (18)	C2B—N1B—C11B	117.93 (19)
C11A—N1A—C1A	114.79 (18)	C11B—N1B—C1B	113.1 (2)
C3A—N2A—H2A	120.0 (16)	C3B—N2B—H2B	120 (2)
C17A—N2A—C3A	121.0 (2)	C17B—N2B—C3B	121.5 (2)
C17A—N2A—H2A	118.6 (16)	C17B—N2B—H2B	119 (2)
C4A—S1A—C1A	94.46 (11)	C4B—S1B—C1B	94.54 (11)
O1A—S1A—C1A	107.22 (13)	O1B—S1B—C1B	105.93 (12)
O1A—S1A—C4A	105.71 (11)	O1B—S1B—C4B	105.70 (14)

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>
N2A—H2A...O2B ⁱ	0.91 (3)	2.25 (3)	3.137 (3)	164 (2)
N2B—H2B...O1B ⁱⁱ	0.79 (3)	2.14 (3)	2.916 (3)	168 (3)
C10A—H10A...O3A	0.93	2.42	3.259 (3)	149

<i>C10B—H10B···O3B</i>	0.93	2.44	3.232 (4)	143
<i>C4B—H4BB···O2Aⁱⁱⁱ</i>	0.97	2.25	3.116 (3)	148

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