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2,3-Diphenyl-3,4-dihydro-2H-1,3-benzothiazin-4-one

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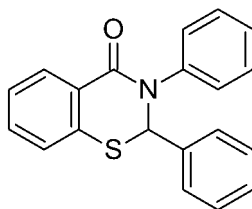
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.051; wR factor = 0.127; data-to-parameter ratio = 19.0.

In the title compound, $\text{C}_{20}\text{H}_{15}\text{NOS}$, the dihedral angle between the phenyl rings is $74.25(6)^\circ$. The six-membered 1,3-thiazine ring has an envelope conformation with the C atom at the 2-position forming the flap. The crystal structure features weak $\text{C}-\text{H}\cdots\text{O}$ interactions, which lead to the formation of a tape motif along [110].

Related literature

For other preparations of the title compound, see: Ponci *et al.* (1963); Kollenz & Ziegler (1970); Oae & Numata (1974); Badea *et al.* (1998). For previously published methods for the preparation of 1,3-thiazin-4-ones by condensation of an imine with a thioacid, see: Kamel *et al.* (2010); Zarghi *et al.* (2009); Zhou *et al.* (2008); Srivastava *et al.* (2002). For the synthesis and crystal structures of related compounds, see: Yennawar & Silverberg (2013, 2014); Yennawar *et al.* (2013).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{15}\text{NOS}$
 $M_r = 317.39$
 Monoclinic, $C2/c$
 $a = 14.799(4)$ Å
 $b = 9.606(3)$ Å
 $c = 22.492(6)$ Å
 $\beta = 98.736(5)^\circ$

$V = 3160.1(15)$ Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.21$ mm⁻¹
 $T = 298$ K
 $0.18 \times 0.16 \times 0.05$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.963$, $T_{\max} = 0.990$
 14747 measured reflections
 3956 independent reflections
 3300 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.127$
 $S = 1.06$
 3956 reflections
 208 parameters
 H-atom parameters not refined
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C10}-\text{H10}\cdots\text{O1}^{\text{i}}$	0.93	2.82	3.422 (2)	124
$\text{C15}-\text{H15}\cdots\text{O1}^{\text{ii}}$	0.93	2.69	3.477 (2)	142

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: XSELL in SHELXTL (Sheldrick, 2008); software used to prepare material for publication: ORTEP-3 for Windows (Farrugia, 2012).

We acknowledge NSF funding (CHEM-0131112) for the X-ray diffractometer, and are thankful to Dr John Tierney for intellectual contributions and to Euticals Inc. for the gift of T3P in 2-methyltetrahydrofuran. RVB, DJC, and ASC acknowledge summer internship support from SP Controls Inc.

Supporting information for this paper is available from the IUCr electronic archives (Reference: FY2112).

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supplementary materials

Acta Cryst. (2014). E70, o465 [doi:10.1107/S1600536814005881]

2,3-Diphenyl-3,4-dihydro-2H-1,3-benzothiazin-4-one

Hemant P. Yennawar, Ryan V. Bendinsky, David J. Coyle, Aaron S. Cali and Lee J. Silverberg

1. Comment

We report here the crystal structure (Fig. 1) of the title compound, which has two phenyl rings connected to the central thiazine ring and a third phenyl ring fused to the thiazine ring. We have recently reported the synthesis and crystal structures of three related compounds: (i) 2-(3-nitrophenyl)-3-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one (Yennawar, Silverberg, Minehan and Tierney, 2013) (ii) 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one (Yennawar & Silverberg, 2014) and (iii) 6,7-diphenyl-5-thia-7-azaspiro[2.6]nonan-8-one (Yennawar & Silverberg, 2013). In order to more directly compare the structure of the benzothiazinone ring to the thiazinone and thiazepanone, we have obtained the crystal structure of the title compound, which, like the latter two compounds does not have a substituent on the *C*-phenyl ring. In the present structure, the dihedral angle between the phenyl rings is 74.25 (6)° - almost perpendicular and within the range of 60–90° in the three other compounds we have reported (see above). The thiazine ring has an envelope conformation with the 2-carbon forming the flap. In the crystal packing (Fig 2), molecules are linked by weak C—H⋯O interactions (Table 1).

The title compound has been synthesized previously (Ponci *et al.*, 1963; Kollenz & Ziegler, 1970; Oae & Numata, 1974; Badea *et al.*, 1998), but not by condensation of *N*-benzylideneaniline with thiosalicylic acid. In our hands, this reaction was not successfully accomplished in refluxing toluene or xylenes, with sodium sulfate in dioxane (Kamel *et al.*, 2010), with *p*-toluenesulfonic acid in refluxing toluene (Zarghi *et al.*, 2009), or with *N,N'*-dicyclohexylcarbodiimide (DCC) in THF (Zhou *et al.*, 2008; Srivastava *et al.*, 2002). The title molecule was finally synthesized by condensation in the presence of 2,4,6-tripropyl-1,3,5,2,4,6-trioxatriphosphorinane-2,4,6-trioxide (T3P) and pyridine, as per our previous reports.

2. Experimental

A two-necked 25 ml roundbottom flask was oven-dried, cooled under N₂, and charged with a stir bar and *N*-benzylideneaniline (1.02 g, 6 mmol). Tetrahydrofuran (2.3 ml) was added, the solid dissolved, and the solution was stirred. Pyridine (1.95 ml, 24 mmol) was added and then thiosalicylic acid (0.931 g, 6 mmol) was added. Finally, 2,4,6-tripropyl-1,3,5,2,4,6-trioxatriphosphorinane-2,4,6-trioxatriphosphorinane-2,4,6-trioxide in 2-methyltetrahydrofuran (50 weight percent; 7.1 ml, 12 mmol) was added. The reaction was stirred at room temperature for 21 h, then poured into a separatory funnel and extracted three times with ethyl acetate. The organic was washed with saturated sodium bicarbonate, water and saturated sodium chloride. The solution was concentrated *in vacuo*, and then the solid was slurried in hot hexanes. After cooling in ice, the solid was collected by vacuum filtration and rinsed with cold hexanes to give a light orange solid (0.8697 g, m.p. 129–133°C). The solid was then recrystallized from ethanol, yielding solid that was still impure by TLC and melting point [0.5921 g, m.p. 132–134°C (lit. 138–140°C; Ponci *et al.*, 1963)]. *R*_f = 0.33 (20% EtOAc/hexanes). Crystals for X-ray crystallography were grown by slow evaporation from toluene.

3. Refinement

The C-bound H atoms were geometrically placed with C—H = 0.93–0.97 Å, and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

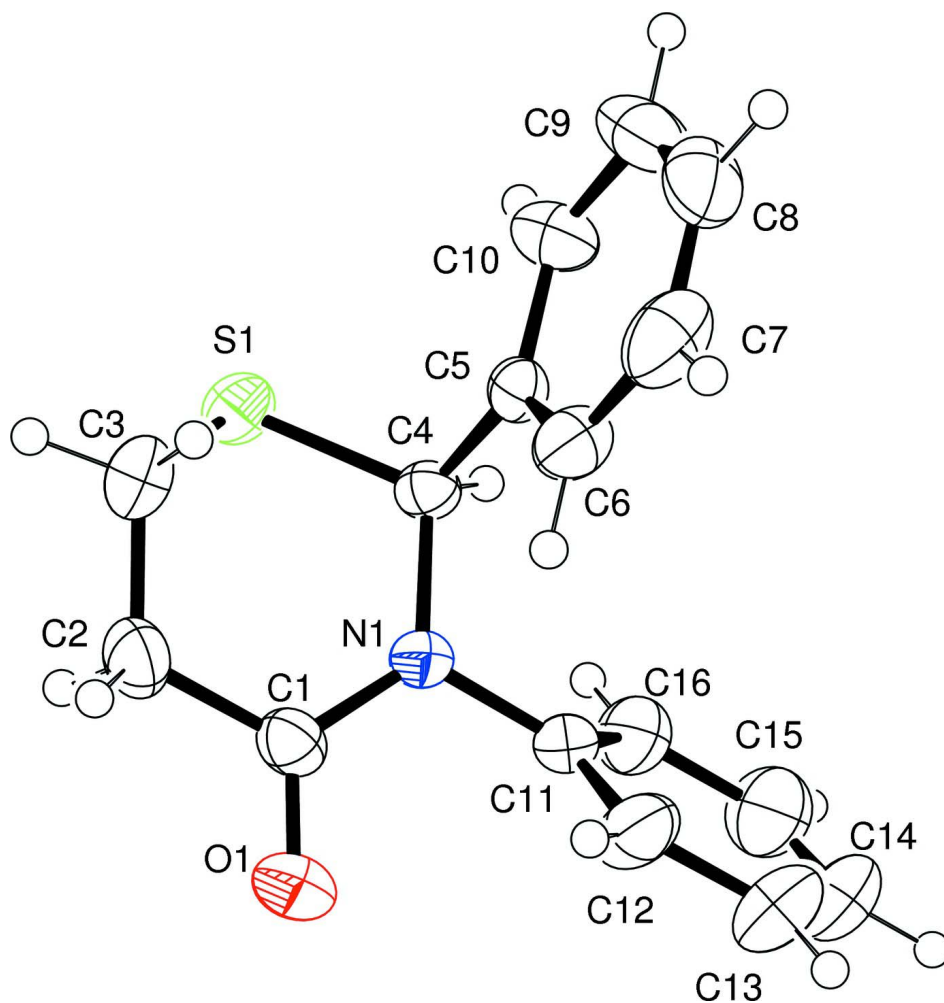
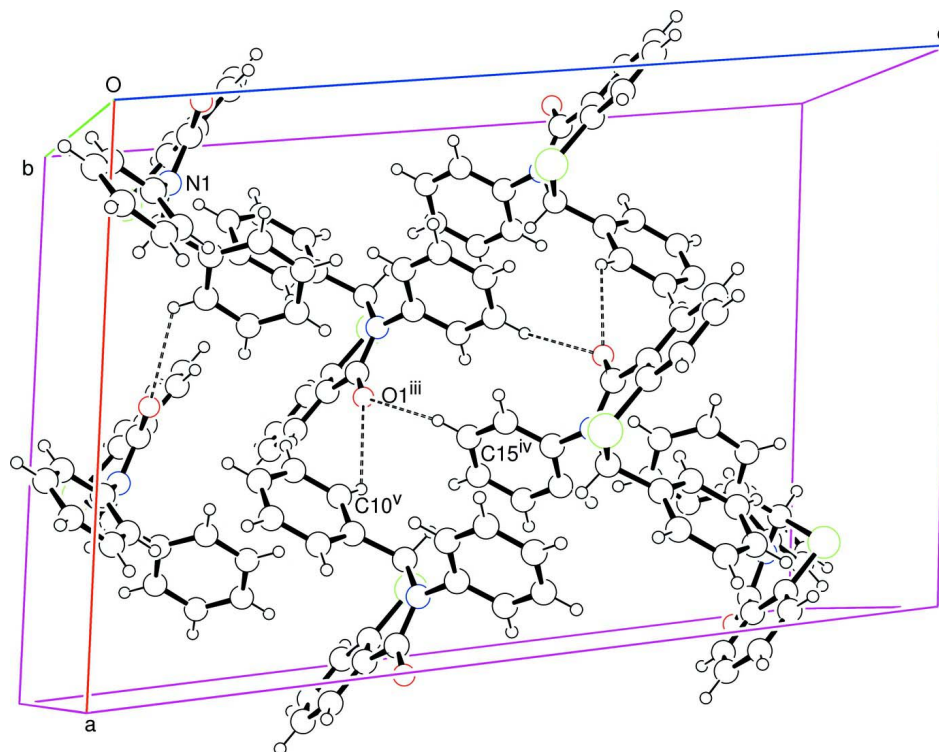


Figure 1

ORTEP view of the title compound. Thermal ellipsoids are drawn at 50% probability.



Symmetry Codes: (iii) $x, -y, z+1/2$; (iv) $x+1/2, -y+1/2, z+1/2$; (v) $1-x, y, -z+1/2$

Figure 2

Crystal packing. C—H...O interactions are shown as dashed lines.

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

Crystal data

$C_{20}H_{15}NOS$

$M_r = 317.39$

Monoclinic, $C2/c$

$a = 14.799$ (4) Å

$b = 9.606$ (3) Å

$c = 22.492$ (6) Å

$\beta = 98.736$ (5)°

$V = 3160.1$ (15) Å³

$Z = 8$

$F(000) = 1328$

$D_x = 1.334$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4538 reflections

$\theta = 2.5$ – 28.0 °

$\mu = 0.21$ mm⁻¹

$T = 298$ K

Block, colourless

$0.18 \times 0.16 \times 0.05$ mm

Data collection

Bruker SMART APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.34 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.963$, $T_{\max} = 0.990$

14747 measured reflections

3956 independent reflections

3300 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 28.4$ °, $\theta_{\min} = 1.8$ °

$h = -18 \rightarrow 19$

$k = -12 \rightarrow 12$

$l = -30 \rightarrow 30$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.127$
 $S = 1.06$
 3956 reflections
 208 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-atom parameters not refined
 $w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 1.8423P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.03935 (11)	0.32561 (17)	0.11547 (7)	0.0434 (4)
C2	0.01411 (11)	0.46594 (17)	0.13594 (7)	0.0434 (4)
C3	0.04910 (12)	0.58978 (17)	0.11600 (7)	0.0460 (4)
C4	0.18665 (11)	0.42745 (15)	0.09649 (7)	0.0406 (3)
H4	0.2301	0.4022	0.0695	0.049*
C5	0.24251 (10)	0.45006 (15)	0.15831 (7)	0.0377 (3)
C6	0.23025 (12)	0.37177 (17)	0.20814 (8)	0.0469 (4)
H6	0.1848	0.3041	0.2048	0.056*
C7	0.28512 (13)	0.3933 (2)	0.26298 (8)	0.0557 (4)
H7	0.2759	0.3408	0.2963	0.067*
C8	0.35351 (13)	0.4925 (2)	0.26842 (9)	0.0559 (4)
H8	0.3903	0.5069	0.3052	0.067*
C9	0.36669 (12)	0.56958 (17)	0.21908 (9)	0.0519 (4)
H9	0.4131	0.6357	0.2225	0.062*
C10	0.31166 (11)	0.54979 (16)	0.16442 (8)	0.0442 (4)
H10	0.3208	0.6034	0.1314	0.053*
C11	0.14103 (11)	0.18738 (16)	0.06409 (7)	0.0393 (3)
C12	0.21265 (16)	0.1052 (2)	0.08852 (9)	0.0657 (6)
H12	0.2492	0.1317	0.1241	0.079*
C13	0.23063 (18)	-0.0177 (2)	0.06018 (10)	0.0759 (7)
H13	0.2795	-0.0733	0.0767	0.091*
C14	0.17703 (15)	-0.05742 (19)	0.00829 (9)	0.0592 (5)
H14	0.1882	-0.1412	-0.0100	0.071*
C15	0.10730 (15)	0.0256 (2)	-0.01663 (9)	0.0670 (6)
H15	0.0715	-0.0005	-0.0525	0.080*
C16	0.08922 (14)	0.1488 (2)	0.01100 (8)	0.0603 (5)

H16	0.0417	0.2057	-0.0065	0.072*
C17	-0.05168 (12)	0.4728 (2)	0.17424 (8)	0.0514 (4)
H17	-0.0753	0.3909	0.1877	0.062*
C18	-0.08209 (14)	0.5990 (2)	0.19226 (9)	0.0619 (5)
H18	-0.1253	0.6023	0.2182	0.074*
C19	-0.04820 (14)	0.7204 (2)	0.17166 (10)	0.0660 (6)
H19	-0.0696	0.8056	0.1834	0.079*
C20	0.01695 (14)	0.7175 (2)	0.13392 (9)	0.0583 (5)
H20	0.0394	0.8002	0.1204	0.070*
N1	0.12016 (9)	0.31448 (13)	0.09313 (6)	0.0408 (3)
O1	-0.01007 (9)	0.22507 (14)	0.11818 (6)	0.0617 (4)
S1	0.13067 (3)	0.58704 (5)	0.06672 (2)	0.05412 (16)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0430 (9)	0.0427 (8)	0.0451 (8)	-0.0043 (7)	0.0089 (7)	-0.0052 (7)
C2	0.0396 (8)	0.0446 (9)	0.0443 (8)	0.0026 (7)	0.0007 (6)	-0.0060 (7)
C3	0.0447 (9)	0.0441 (9)	0.0454 (8)	0.0056 (7)	-0.0049 (7)	-0.0001 (7)
C4	0.0427 (8)	0.0361 (7)	0.0449 (8)	-0.0027 (6)	0.0124 (6)	0.0006 (6)
C5	0.0369 (8)	0.0311 (7)	0.0464 (8)	0.0018 (6)	0.0104 (6)	-0.0028 (6)
C6	0.0459 (9)	0.0435 (8)	0.0521 (9)	-0.0045 (7)	0.0107 (7)	0.0021 (7)
C7	0.0616 (11)	0.0576 (11)	0.0474 (9)	0.0031 (9)	0.0071 (8)	0.0055 (8)
C8	0.0561 (11)	0.0523 (10)	0.0555 (10)	0.0068 (9)	-0.0038 (8)	-0.0108 (8)
C9	0.0457 (9)	0.0382 (8)	0.0700 (11)	-0.0023 (7)	0.0033 (8)	-0.0101 (8)
C10	0.0457 (9)	0.0332 (7)	0.0552 (9)	-0.0018 (6)	0.0119 (7)	-0.0009 (7)
C11	0.0436 (8)	0.0360 (7)	0.0396 (7)	-0.0032 (6)	0.0111 (6)	-0.0018 (6)
C12	0.0748 (14)	0.0642 (12)	0.0528 (10)	0.0207 (10)	-0.0070 (9)	-0.0110 (9)
C13	0.1004 (18)	0.0631 (13)	0.0624 (12)	0.0353 (12)	0.0069 (12)	0.0007 (10)
C14	0.0841 (14)	0.0400 (9)	0.0607 (11)	-0.0032 (9)	0.0341 (10)	-0.0072 (8)
C15	0.0679 (13)	0.0740 (14)	0.0582 (11)	-0.0013 (11)	0.0070 (9)	-0.0278 (10)
C16	0.0606 (11)	0.0658 (12)	0.0513 (10)	0.0133 (10)	-0.0021 (8)	-0.0166 (9)
C17	0.0438 (9)	0.0568 (10)	0.0531 (9)	0.0044 (8)	0.0058 (7)	-0.0069 (8)
C18	0.0518 (11)	0.0707 (13)	0.0622 (11)	0.0164 (9)	0.0056 (9)	-0.0137 (10)
C19	0.0635 (12)	0.0582 (11)	0.0713 (12)	0.0262 (10)	-0.0057 (10)	-0.0156 (10)
C20	0.0636 (12)	0.0425 (9)	0.0630 (11)	0.0104 (8)	-0.0094 (9)	-0.0011 (8)
N1	0.0424 (7)	0.0363 (6)	0.0447 (7)	-0.0045 (5)	0.0101 (5)	-0.0051 (5)
O1	0.0595 (8)	0.0514 (7)	0.0800 (9)	-0.0190 (6)	0.0296 (7)	-0.0166 (6)
S1	0.0616 (3)	0.0450 (2)	0.0554 (3)	-0.0003 (2)	0.0080 (2)	0.01415 (18)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.218 (2)	C10—H10	0.9300
C1—N1	1.370 (2)	C11—C12	1.368 (2)
C1—C2	1.490 (2)	C11—C16	1.368 (2)
C2—C17	1.396 (2)	C11—N1	1.4401 (19)
C2—C3	1.398 (2)	C12—C13	1.386 (3)
C3—C20	1.397 (2)	C12—H12	0.9300
C3—S1	1.7587 (19)	C13—C14	1.362 (3)
C4—N1	1.4590 (19)	C13—H13	0.9300

C4—C5	1.521 (2)	C14—C15	1.356 (3)
C4—S1	1.8211 (16)	C14—H14	0.9300
C4—H4	0.9800	C15—C16	1.382 (3)
C5—C6	1.384 (2)	C15—H15	0.9300
C5—C10	1.393 (2)	C16—H16	0.9300
C6—C7	1.385 (2)	C17—C18	1.376 (3)
C6—H6	0.9300	C17—H17	0.9300
C7—C8	1.382 (3)	C18—C19	1.377 (3)
C7—H7	0.9300	C18—H18	0.9300
C8—C9	1.373 (3)	C19—C20	1.378 (3)
C8—H8	0.9300	C19—H19	0.9300
C9—C10	1.381 (2)	C20—H20	0.9300
C9—H9	0.9300		
O1—C1—N1	121.37 (15)	C12—C11—N1	120.90 (15)
O1—C1—C2	121.40 (15)	C16—C11—N1	119.66 (15)
N1—C1—C2	117.23 (14)	C11—C12—C13	119.86 (18)
C17—C2—C3	119.01 (16)	C11—C12—H12	120.1
C17—C2—C1	117.65 (15)	C13—C12—H12	120.1
C3—C2—C1	123.24 (15)	C14—C13—C12	120.3 (2)
C20—C3—C2	119.70 (17)	C14—C13—H13	119.8
C20—C3—S1	119.44 (14)	C12—C13—H13	119.8
C2—C3—S1	120.83 (13)	C15—C14—C13	119.79 (18)
N1—C4—C5	114.97 (12)	C15—C14—H14	120.1
N1—C4—S1	109.96 (11)	C13—C14—H14	120.1
C5—C4—S1	111.69 (10)	C14—C15—C16	120.32 (18)
N1—C4—H4	106.6	C14—C15—H15	119.8
C5—C4—H4	106.6	C16—C15—H15	119.8
S1—C4—H4	106.6	C11—C16—C15	120.21 (18)
C6—C5—C10	118.58 (15)	C11—C16—H16	119.9
C6—C5—C4	122.85 (14)	C15—C16—H16	119.9
C10—C5—C4	118.51 (14)	C18—C17—C2	120.88 (19)
C5—C6—C7	120.57 (16)	C18—C17—H17	119.6
C5—C6—H6	119.7	C2—C17—H17	119.6
C7—C6—H6	119.7	C17—C18—C19	119.67 (19)
C8—C7—C6	120.27 (17)	C17—C18—H18	120.2
C8—C7—H7	119.9	C19—C18—H18	120.2
C6—C7—H7	119.9	C18—C19—C20	120.95 (18)
C9—C8—C7	119.51 (17)	C18—C19—H19	119.5
C9—C8—H8	120.2	C20—C19—H19	119.5
C7—C8—H8	120.2	C19—C20—C3	119.77 (19)
C8—C9—C10	120.57 (17)	C19—C20—H20	120.1
C8—C9—H9	119.7	C3—C20—H20	120.1
C10—C9—H9	119.7	C1—N1—C11	119.46 (13)
C9—C10—C5	120.50 (16)	C1—N1—C4	122.85 (13)
C9—C10—H10	119.8	C11—N1—C4	117.68 (12)
C5—C10—H10	119.8	C3—S1—C4	95.64 (7)
C12—C11—C16	119.43 (16)		

O1—C1—C2—C17	18.8 (2)	N1—C11—C16—C15	-178.59 (18)
N1—C1—C2—C17	-161.54 (14)	C14—C15—C16—C11	-0.6 (3)
O1—C1—C2—C3	-157.41 (17)	C3—C2—C17—C18	0.1 (3)
N1—C1—C2—C3	22.2 (2)	C1—C2—C17—C18	-176.35 (16)
C17—C2—C3—C20	-0.9 (2)	C2—C17—C18—C19	0.9 (3)
C1—C2—C3—C20	175.31 (15)	C17—C18—C19—C20	-1.0 (3)
C17—C2—C3—S1	-178.93 (12)	C18—C19—C20—C3	0.2 (3)
C1—C2—C3—S1	-2.7 (2)	C2—C3—C20—C19	0.8 (3)
N1—C4—C5—C6	-1.3 (2)	S1—C3—C20—C19	178.82 (14)
S1—C4—C5—C6	124.86 (14)	O1—C1—N1—C11	9.4 (2)
N1—C4—C5—C10	175.80 (13)	C2—C1—N1—C11	-170.25 (13)
S1—C4—C5—C10	-58.01 (16)	O1—C1—N1—C4	-171.62 (16)
C10—C5—C6—C7	0.6 (2)	C2—C1—N1—C4	8.8 (2)
C4—C5—C6—C7	177.72 (15)	C12—C11—N1—C1	-114.62 (19)
C5—C6—C7—C8	-0.7 (3)	C16—C11—N1—C1	66.1 (2)
C6—C7—C8—C9	0.0 (3)	C12—C11—N1—C4	66.3 (2)
C7—C8—C9—C10	0.7 (3)	C16—C11—N1—C4	-113.00 (18)
C8—C9—C10—C5	-0.7 (3)	C5—C4—N1—C1	75.41 (18)
C6—C5—C10—C9	0.1 (2)	S1—C4—N1—C1	-51.67 (17)
C4—C5—C10—C9	-177.16 (14)	C5—C4—N1—C11	-105.55 (15)
C16—C11—C12—C13	-1.6 (3)	S1—C4—N1—C11	127.37 (12)
N1—C11—C12—C13	179.11 (19)	C20—C3—S1—C4	148.70 (14)
C11—C12—C13—C14	-0.4 (4)	C2—C3—S1—C4	-33.26 (15)
C12—C13—C14—C15	1.8 (4)	N1—C4—S1—C3	56.83 (12)
C13—C14—C15—C16	-1.3 (3)	C5—C4—S1—C3	-72.06 (12)
C12—C11—C16—C15	2.1 (3)		

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>
C10—H10...O1 ⁱ	0.93	2.82	3.422 (2)	124
C15—H15...O1 ⁱⁱ	0.93	2.69	3.477 (2)	142

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

supplementary materials

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

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1. Comment

We report here the crystal structure (Fig. 1) of the title compound, which has two phenyl rings connected to the central thiazine ring and a third phenyl ring fused to the thiazine ring. We have recently reported the synthesis and crystal structures of three related compounds: (i) 2-(3-nitrophenyl)-3-phenyl-2,3-dihydro-4*H*-1,3-benzothiazin-4-one (Yennawar, Silverberg, Minehan and Tierney, 2013) (ii) 2,3-diphenyl-2,3,5,6-tetrahydro-4*H*-1,3-thiazin-4-one (Yennawar & Silverberg, 2014) and (iii) 6,7-diphenyl-5-thia-7-azaspiro[2.6]nonan-8-one (Yennawar & Silverberg, 2013). In order to more directly compare the structure of the benzothiazinone ring to the thiazinone and thiazepanone, we have obtained the crystal structure of the title compound, which, like the latter two compounds does not have a substituent on the *C*-phenyl ring. In the present structure, the dihedral angle between the phenyl rings is 74.25 (6)° - almost perpendicular and within the range of 60–90° in the three other compounds we have reported (see above). The thiazine ring has an envelope conformation with the 2-carbon forming the flap. In the crystal packing (Fig 2), molecules are linked by weak C—H⋯O interactions (Table 1).

The title compound has been synthesized previously (Ponci *et al.*, 1963; Kollenz & Ziegler, 1970; Oae & Numata, 1974; Badea *et al.*, 1998), but not by condensation of *N*-benzylideneaniline with thiosalicylic acid. In our hands, this reaction was not successfully accomplished in refluxing toluene or xylenes, with sodium sulfate in dioxane (Kamel *et al.*, 2010), with *p*-toluenesulfonic acid in refluxing toluene (Zarghi *et al.*, 2009), or with *N,N'*-dicyclohexylcarbodiimide (DCC) in THF (Zhou *et al.*, 2008; Srivastava *et al.*, 2002). The title molecule was finally synthesized by condensation in the presence of 2,4,6-tripropyl-1,3,5,2,4,6-trioxatriphosphorinane-2,4,6-trioxide (T3P) and pyridine, as per our previous reports.

2. Experimental

A two-necked 25 ml roundbottom flask was oven-dried, cooled under N₂, and charged with a stir bar and *N*-benzylideneaniline (1.02 g, 6 mmol). Tetrahydrofuran (2.3 ml) was added, the solid dissolved, and the solution was stirred. Pyridine (1.95 ml, 24 mmol) was added and then thiosalicylic acid (0.931 g, 6 mmol) was added. Finally, 2,4,6-tripropyl-1,3,5,2,4,6-trioxatriphosphorinane-2,4,6-trioxatriphosphorinane-2,4,6-trioxide in 2-methyltetrahydrofuran (50 weight percent; 7.1 ml, 12 mmol) was added. The reaction was stirred at room temperature for 21 h, then poured into a separatory funnel and extracted three times with ethyl acetate. The organic was washed with saturated sodium bicarbonate, water and saturated sodium chloride. The solution was concentrated *in vacuo*, and then the solid was slurried in hot hexanes. After cooling in ice, the solid was collected by vacuum filtration and rinsed with cold hexanes to give a light orange solid (0.8697 g, m.p. 129–133°C). The solid was then recrystallized from ethanol, yielding solid that was still impure by TLC and melting point [0.5921 g, m.p. 132–134°C (lit. 138–140°C; Ponci *et al.*, 1963)]. R_f = 0.33 (20% EtOAc/hexanes). Crystals for X-ray crystallography were grown by slow evaporation from toluene.

3. Refinement

The C-bound H atoms were geometrically placed with C—H = 0.93–0.97 Å, and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

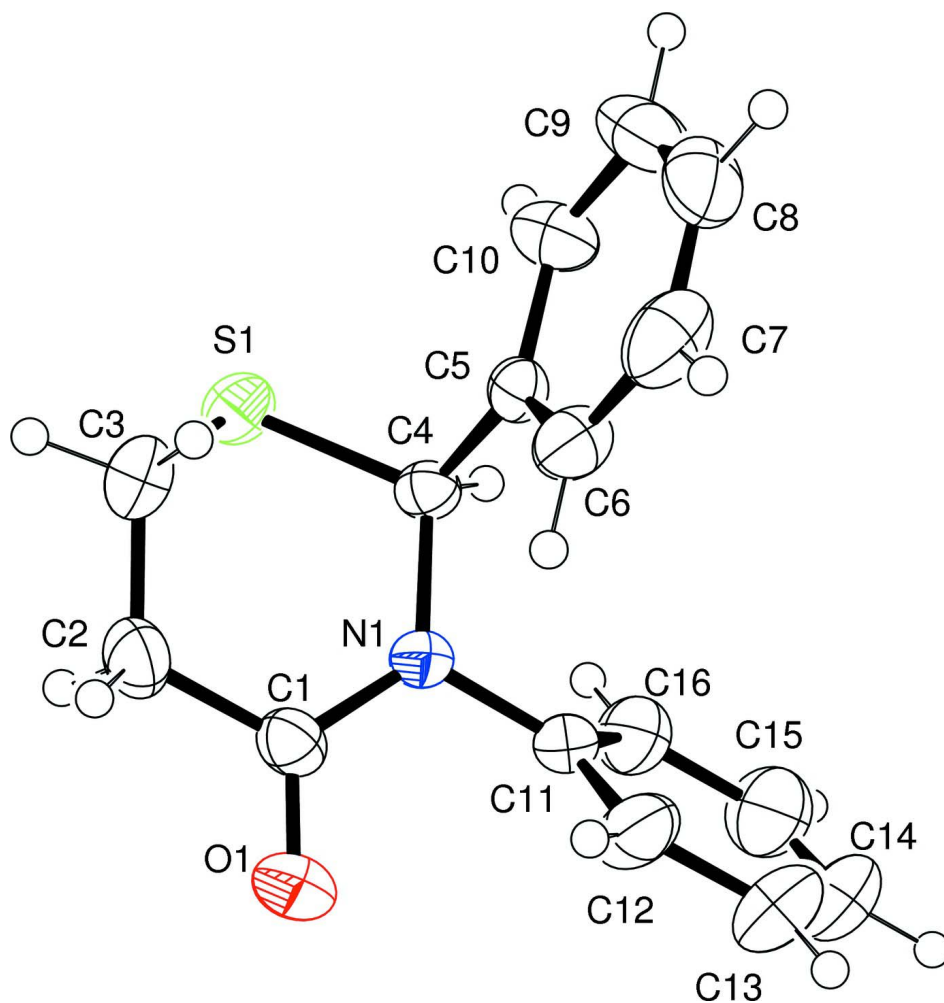
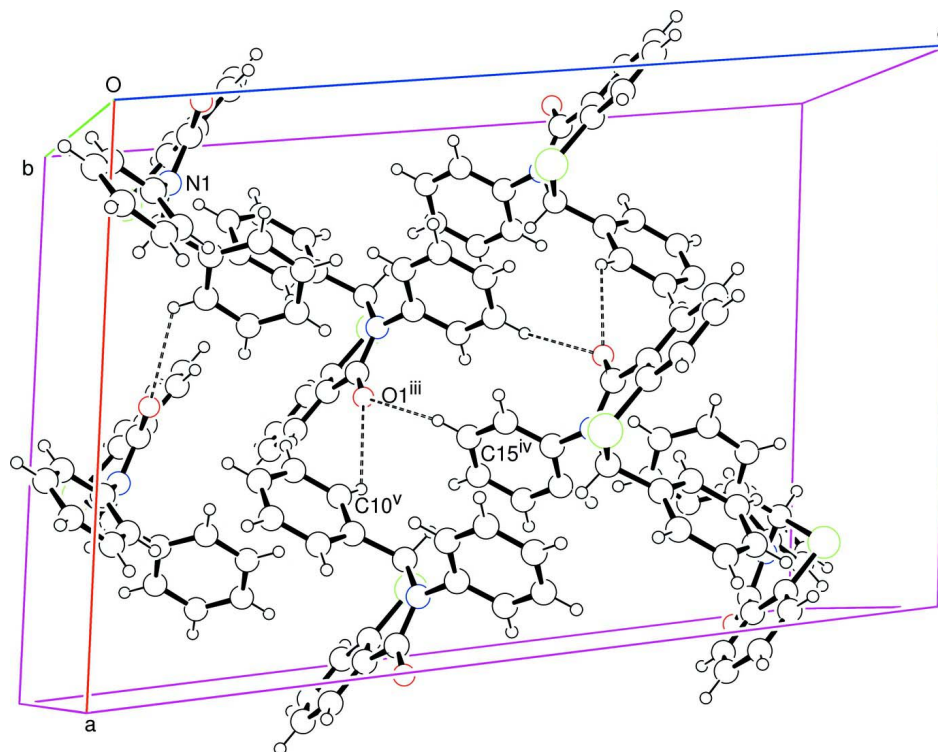


Figure 1

ORTEP view of the title compound. Thermal ellipsoids are drawn at 50% probability.



Symmetry Codes: (iii) $x, -y, z+1/2$; (iv) $x+1/2, -y+1/2, z+1/2$; (v) $1-x, y, -z+1/2$

Figure 2

Crystal packing. C—H...O interactions are shown as dashed lines.

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

Crystal data

$C_{20}H_{15}NOS$

$M_r = 317.39$

Monoclinic, $C2/c$

$a = 14.799$ (4) Å

$b = 9.606$ (3) Å

$c = 22.492$ (6) Å

$\beta = 98.736$ (5)°

$V = 3160.1$ (15) Å³

$Z = 8$

$F(000) = 1328$

$D_x = 1.334$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4538 reflections

$\theta = 2.5$ – 28.0 °

$\mu = 0.21$ mm⁻¹

$T = 298$ K

Block, colourless

$0.18 \times 0.16 \times 0.05$ mm

Data collection

Bruker SMART APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.34 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.963$, $T_{\max} = 0.990$

14747 measured reflections

3956 independent reflections

3300 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 28.4$ °, $\theta_{\min} = 1.8$ °

$h = -18$ → 19

$k = -12$ → 12

$l = -30$ → 30

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.127$
 $S = 1.06$
 3956 reflections
 208 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-atom parameters not refined
 $w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 1.8423P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

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	0.03935 (11)	0.32561 (17)	0.11547 (7)	0.0434 (4)
C2	0.01411 (11)	0.46594 (17)	0.13594 (7)	0.0434 (4)
C3	0.04910 (12)	0.58978 (17)	0.11600 (7)	0.0460 (4)
C4	0.18665 (11)	0.42745 (15)	0.09649 (7)	0.0406 (3)
H4	0.2301	0.4022	0.0695	0.049*
C5	0.24251 (10)	0.45006 (15)	0.15831 (7)	0.0377 (3)
C6	0.23025 (12)	0.37177 (17)	0.20814 (8)	0.0469 (4)
H6	0.1848	0.3041	0.2048	0.056*
C7	0.28512 (13)	0.3933 (2)	0.26298 (8)	0.0557 (4)
H7	0.2759	0.3408	0.2963	0.067*
C8	0.35351 (13)	0.4925 (2)	0.26842 (9)	0.0559 (4)
H8	0.3903	0.5069	0.3052	0.067*
C9	0.36669 (12)	0.56958 (17)	0.21908 (9)	0.0519 (4)
H9	0.4131	0.6357	0.2225	0.062*
C10	0.31166 (11)	0.54979 (16)	0.16442 (8)	0.0442 (4)
H10	0.3208	0.6034	0.1314	0.053*
C11	0.14103 (11)	0.18738 (16)	0.06409 (7)	0.0393 (3)
C12	0.21265 (16)	0.1052 (2)	0.08852 (9)	0.0657 (6)
H12	0.2492	0.1317	0.1241	0.079*
C13	0.23063 (18)	-0.0177 (2)	0.06018 (10)	0.0759 (7)
H13	0.2795	-0.0733	0.0767	0.091*
C14	0.17703 (15)	-0.05742 (19)	0.00829 (9)	0.0592 (5)
H14	0.1882	-0.1412	-0.0100	0.071*
C15	0.10730 (15)	0.0256 (2)	-0.01663 (9)	0.0670 (6)
H15	0.0715	-0.0005	-0.0525	0.080*
C16	0.08922 (14)	0.1488 (2)	0.01100 (8)	0.0603 (5)

H16	0.0417	0.2057	-0.0065	0.072*
C17	-0.05168 (12)	0.4728 (2)	0.17424 (8)	0.0514 (4)
H17	-0.0753	0.3909	0.1877	0.062*
C18	-0.08209 (14)	0.5990 (2)	0.19226 (9)	0.0619 (5)
H18	-0.1253	0.6023	0.2182	0.074*
C19	-0.04820 (14)	0.7204 (2)	0.17166 (10)	0.0660 (6)
H19	-0.0696	0.8056	0.1834	0.079*
C20	0.01695 (14)	0.7175 (2)	0.13392 (9)	0.0583 (5)
H20	0.0394	0.8002	0.1204	0.070*
N1	0.12016 (9)	0.31448 (13)	0.09313 (6)	0.0408 (3)
O1	-0.01007 (9)	0.22507 (14)	0.11818 (6)	0.0617 (4)
S1	0.13067 (3)	0.58704 (5)	0.06672 (2)	0.05412 (16)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0430 (9)	0.0427 (8)	0.0451 (8)	-0.0043 (7)	0.0089 (7)	-0.0052 (7)
C2	0.0396 (8)	0.0446 (9)	0.0443 (8)	0.0026 (7)	0.0007 (6)	-0.0060 (7)
C3	0.0447 (9)	0.0441 (9)	0.0454 (8)	0.0056 (7)	-0.0049 (7)	-0.0001 (7)
C4	0.0427 (8)	0.0361 (7)	0.0449 (8)	-0.0027 (6)	0.0124 (6)	0.0006 (6)
C5	0.0369 (8)	0.0311 (7)	0.0464 (8)	0.0018 (6)	0.0104 (6)	-0.0028 (6)
C6	0.0459 (9)	0.0435 (8)	0.0521 (9)	-0.0045 (7)	0.0107 (7)	0.0021 (7)
C7	0.0616 (11)	0.0576 (11)	0.0474 (9)	0.0031 (9)	0.0071 (8)	0.0055 (8)
C8	0.0561 (11)	0.0523 (10)	0.0555 (10)	0.0068 (9)	-0.0038 (8)	-0.0108 (8)
C9	0.0457 (9)	0.0382 (8)	0.0700 (11)	-0.0023 (7)	0.0033 (8)	-0.0101 (8)
C10	0.0457 (9)	0.0332 (7)	0.0552 (9)	-0.0018 (6)	0.0119 (7)	-0.0009 (7)
C11	0.0436 (8)	0.0360 (7)	0.0396 (7)	-0.0032 (6)	0.0111 (6)	-0.0018 (6)
C12	0.0748 (14)	0.0642 (12)	0.0528 (10)	0.0207 (10)	-0.0070 (9)	-0.0110 (9)
C13	0.1004 (18)	0.0631 (13)	0.0624 (12)	0.0353 (12)	0.0069 (12)	0.0007 (10)
C14	0.0841 (14)	0.0400 (9)	0.0607 (11)	-0.0032 (9)	0.0341 (10)	-0.0072 (8)
C15	0.0679 (13)	0.0740 (14)	0.0582 (11)	-0.0013 (11)	0.0070 (9)	-0.0278 (10)
C16	0.0606 (11)	0.0658 (12)	0.0513 (10)	0.0133 (10)	-0.0021 (8)	-0.0166 (9)
C17	0.0438 (9)	0.0568 (10)	0.0531 (9)	0.0044 (8)	0.0058 (7)	-0.0069 (8)
C18	0.0518 (11)	0.0707 (13)	0.0622 (11)	0.0164 (9)	0.0056 (9)	-0.0137 (10)
C19	0.0635 (12)	0.0582 (11)	0.0713 (12)	0.0262 (10)	-0.0057 (10)	-0.0156 (10)
C20	0.0636 (12)	0.0425 (9)	0.0630 (11)	0.0104 (8)	-0.0094 (9)	-0.0011 (8)
N1	0.0424 (7)	0.0363 (6)	0.0447 (7)	-0.0045 (5)	0.0101 (5)	-0.0051 (5)
O1	0.0595 (8)	0.0514 (7)	0.0800 (9)	-0.0190 (6)	0.0296 (7)	-0.0166 (6)
S1	0.0616 (3)	0.0450 (2)	0.0554 (3)	-0.0003 (2)	0.0080 (2)	0.01415 (18)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.218 (2)	C10—H10	0.9300
C1—N1	1.370 (2)	C11—C12	1.368 (2)
C1—C2	1.490 (2)	C11—C16	1.368 (2)
C2—C17	1.396 (2)	C11—N1	1.4401 (19)
C2—C3	1.398 (2)	C12—C13	1.386 (3)
C3—C20	1.397 (2)	C12—H12	0.9300
C3—S1	1.7587 (19)	C13—C14	1.362 (3)
C4—N1	1.4590 (19)	C13—H13	0.9300

C4—C5	1.521 (2)	C14—C15	1.356 (3)
C4—S1	1.8211 (16)	C14—H14	0.9300
C4—H4	0.9800	C15—C16	1.382 (3)
C5—C6	1.384 (2)	C15—H15	0.9300
C5—C10	1.393 (2)	C16—H16	0.9300
C6—C7	1.385 (2)	C17—C18	1.376 (3)
C6—H6	0.9300	C17—H17	0.9300
C7—C8	1.382 (3)	C18—C19	1.377 (3)
C7—H7	0.9300	C18—H18	0.9300
C8—C9	1.373 (3)	C19—C20	1.378 (3)
C8—H8	0.9300	C19—H19	0.9300
C9—C10	1.381 (2)	C20—H20	0.9300
C9—H9	0.9300		
O1—C1—N1	121.37 (15)	C12—C11—N1	120.90 (15)
O1—C1—C2	121.40 (15)	C16—C11—N1	119.66 (15)
N1—C1—C2	117.23 (14)	C11—C12—C13	119.86 (18)
C17—C2—C3	119.01 (16)	C11—C12—H12	120.1
C17—C2—C1	117.65 (15)	C13—C12—H12	120.1
C3—C2—C1	123.24 (15)	C14—C13—C12	120.3 (2)
C20—C3—C2	119.70 (17)	C14—C13—H13	119.8
C20—C3—S1	119.44 (14)	C12—C13—H13	119.8
C2—C3—S1	120.83 (13)	C15—C14—C13	119.79 (18)
N1—C4—C5	114.97 (12)	C15—C14—H14	120.1
N1—C4—S1	109.96 (11)	C13—C14—H14	120.1
C5—C4—S1	111.69 (10)	C14—C15—C16	120.32 (18)
N1—C4—H4	106.6	C14—C15—H15	119.8
C5—C4—H4	106.6	C16—C15—H15	119.8
S1—C4—H4	106.6	C11—C16—C15	120.21 (18)
C6—C5—C10	118.58 (15)	C11—C16—H16	119.9
C6—C5—C4	122.85 (14)	C15—C16—H16	119.9
C10—C5—C4	118.51 (14)	C18—C17—C2	120.88 (19)
C5—C6—C7	120.57 (16)	C18—C17—H17	119.6
C5—C6—H6	119.7	C2—C17—H17	119.6
C7—C6—H6	119.7	C17—C18—C19	119.67 (19)
C8—C7—C6	120.27 (17)	C17—C18—H18	120.2
C8—C7—H7	119.9	C19—C18—H18	120.2
C6—C7—H7	119.9	C18—C19—C20	120.95 (18)
C9—C8—C7	119.51 (17)	C18—C19—H19	119.5
C9—C8—H8	120.2	C20—C19—H19	119.5
C7—C8—H8	120.2	C19—C20—C3	119.77 (19)
C8—C9—C10	120.57 (17)	C19—C20—H20	120.1
C8—C9—H9	119.7	C3—C20—H20	120.1
C10—C9—H9	119.7	C1—N1—C11	119.46 (13)
C9—C10—C5	120.50 (16)	C1—N1—C4	122.85 (13)
C9—C10—H10	119.8	C11—N1—C4	117.68 (12)
C5—C10—H10	119.8	C3—S1—C4	95.64 (7)
C12—C11—C16	119.43 (16)		

O1—C1—C2—C17	18.8 (2)	N1—C11—C16—C15	-178.59 (18)
N1—C1—C2—C17	-161.54 (14)	C14—C15—C16—C11	-0.6 (3)
O1—C1—C2—C3	-157.41 (17)	C3—C2—C17—C18	0.1 (3)
N1—C1—C2—C3	22.2 (2)	C1—C2—C17—C18	-176.35 (16)
C17—C2—C3—C20	-0.9 (2)	C2—C17—C18—C19	0.9 (3)
C1—C2—C3—C20	175.31 (15)	C17—C18—C19—C20	-1.0 (3)
C17—C2—C3—S1	-178.93 (12)	C18—C19—C20—C3	0.2 (3)
C1—C2—C3—S1	-2.7 (2)	C2—C3—C20—C19	0.8 (3)
N1—C4—C5—C6	-1.3 (2)	S1—C3—C20—C19	178.82 (14)
S1—C4—C5—C6	124.86 (14)	O1—C1—N1—C11	9.4 (2)
N1—C4—C5—C10	175.80 (13)	C2—C1—N1—C11	-170.25 (13)
S1—C4—C5—C10	-58.01 (16)	O1—C1—N1—C4	-171.62 (16)
C10—C5—C6—C7	0.6 (2)	C2—C1—N1—C4	8.8 (2)
C4—C5—C6—C7	177.72 (15)	C12—C11—N1—C1	-114.62 (19)
C5—C6—C7—C8	-0.7 (3)	C16—C11—N1—C1	66.1 (2)
C6—C7—C8—C9	0.0 (3)	C12—C11—N1—C4	66.3 (2)
C7—C8—C9—C10	0.7 (3)	C16—C11—N1—C4	-113.00 (18)
C8—C9—C10—C5	-0.7 (3)	C5—C4—N1—C1	75.41 (18)
C6—C5—C10—C9	0.1 (2)	S1—C4—N1—C1	-51.67 (17)
C4—C5—C10—C9	-177.16 (14)	C5—C4—N1—C11	-105.55 (15)
C16—C11—C12—C13	-1.6 (3)	S1—C4—N1—C11	127.37 (12)
N1—C11—C12—C13	179.11 (19)	C20—C3—S1—C4	148.70 (14)
C11—C12—C13—C14	-0.4 (4)	C2—C3—S1—C4	-33.26 (15)
C12—C13—C14—C15	1.8 (4)	N1—C4—S1—C3	56.83 (12)
C13—C14—C15—C16	-1.3 (3)	C5—C4—S1—C3	-72.06 (12)
C12—C11—C16—C15	2.1 (3)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10—H10 \cdots O1 ⁱ	0.93	2.82	3.422 (2)	124
C15—H15 \cdots O1 ⁱⁱ	0.93	2.69	3.477 (2)	142

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