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(*Z*)-4-*n*-Butyl-2-(4-chlorobenzylidene)-2*H*-1,4-benzothiazin-3(4*H*)-one

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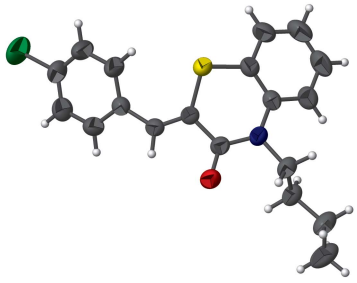
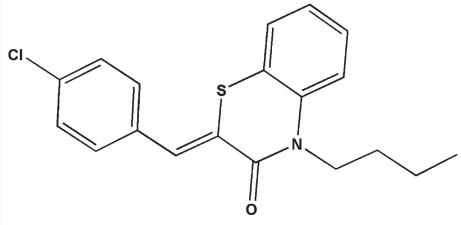
In the title compound, $C_{19}H_{18}ClNOS$, the thiazin-3-one ring adopts a slightly distorted screw-boat conformation. An intramolecular C—H···S hydrogen bond encloses an *S*(6) ring and affects the overall conformation of the molecule. The dihedral angle between the two phenyl rings is 52.3 (2)°. In the crystal, weak C—H···O intermolecular interactions stabilize the crystal packing.

3D view



Chemical scheme



Structure description

1,4-Benzothiazine-containing compounds are important due to their potential applications in the treatment of diabetes complications, by inhibiting aldose reductase (Aotsuka *et al.*, 1994). They are also used as analgesics (Wammack *et al.*, 2002), Ca^{2+} antagonists (Fujimura *et al.*, 1996), and have antimicrobial properties (Zia-ur-Rehman *et al.*, 2009). As a continuation of our previous work on the synthesis of new 1,4-benzothiazine derivatives (Sebbar *et al.*,2016*a*,*b*; Ellouz *et al.*,2017*a*,*b*), we report here the synthesis and crystal structure of the title compound (Fig. 1). This was prepared by reacting (*Z*)-2-(4-chlorobenzylidene)-2*H*-1,4-benzothiazin-3(4*H*)-one with 1-bromobutane, under phase-transfer catalysis conditions using tetra-*n*-butyl ammonium bromide (TBAB) as a catalyst and potassium carbonate as the base.

The title compound crystallizes with one independent molecule in the asymmetric unit (Fig. 1). The thiazine-3-one ring of the [1,4]thiazin-3-one moiety adopts a slightly distorted screw-boat conformation [puckering parameters: Q = 0.468 (7) Å, θ = 69.8 (7)° and φ = 25.949 (3)°]. An intramolecular C15—H15···S1 hydrogen bond forms an *S*(6)

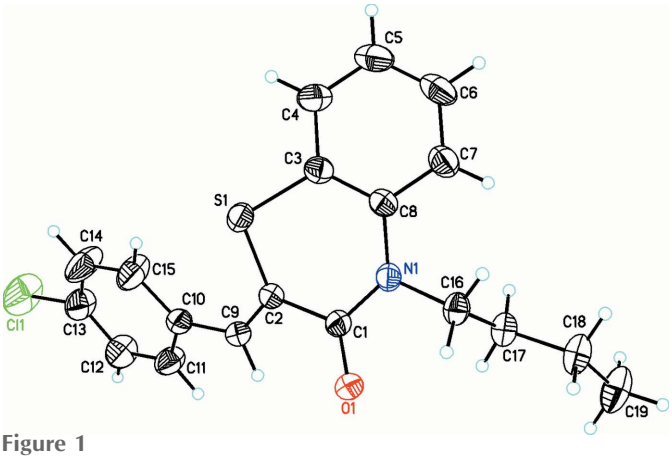


Figure 1
Structure of the title compound, showing the atom-numbering scheme, with displacement ellipsoids drawn at the 30% probability level.

ring motif, and at least partially determines the conformation of the molecule. The dihedral angle between the phenyl rings is 52.3 (2)°.

In the crystal, weak C—H···O hydrogen bonds involving O1 as the acceptor, Table 1, link the molecules into a two-dimensional network (Fig. 2).

Synthesis and crystallization

To a solution of (*Z*)-2-(4-chlorobenzylidene)-2*H*-1,4-benzothiazin-3(4*H*)-one (0.49 g,1.5mmol), potassium carbonate (0.41 g,3 mmol) and tetra-*n*-butyl ammonium bromide (0.048 g,0.15 mmol) in DMF (18 ml) was added 1-bromobutane (3 mmol). Stirring was continued at room temperature for 24 h. The mixture was filtered and the solvent removed. The residue was then washed with water. The organic compound obtained was chromatographed on a column of silica gel with ethyl acetate–hexane (9/1) as the eluent. Colorless prismatic crystals were isolated when the solvent was allowed to evaporate (yield = 51%).

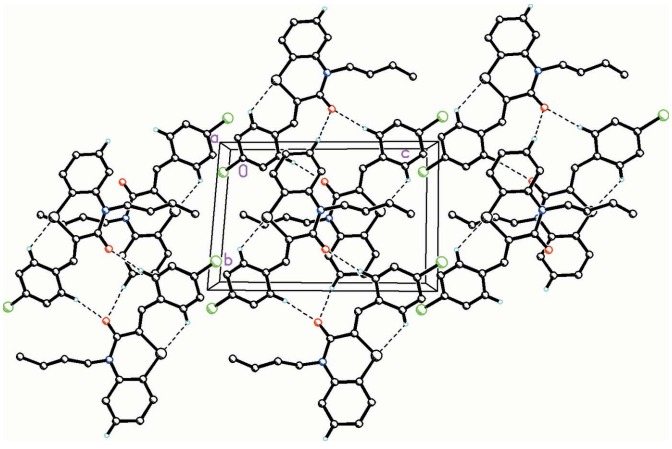


Figure 2
The molecular packing of the title compound, viewed along the *a* axis. Dashed lines indicate weak intra- and intermolecular hydrogen bonds. H atoms not involved in packing have been omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1^{i}	0.93	2.53	3.410 (3)	158
C11—H11···O1^{ii}	0.93	2.39	3.290 (3)	162
C15—H15···S1	0.93	2.55	3.219 (3)	129

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{18}ClNOS$
M_r	343.85
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	8.8898 (8), 8.9885 (9), 12.1973 (10)
α, β, γ (°)	89.908 (8), 84.490 (7), 63.534 (9)
V (Å^3)	867.53 (15)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	3.09
Crystal size (mm)	0.34 × 0.32 × 0.14
Data collection	
Diffractometer	Rigaku Oxford Diffraction
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min}, T_{max}	0.382, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5496, 3279, 2807
R_{int}	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.615
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.146, 1.03
No. of reflections	3279
No. of parameters	210
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.32

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170870 [https://doi.org/10.1107/S2414314617008707]

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Crystal data

$C_{19}H_{18}ClNOS$
M_r = 343.85
Triclinic, $P\overline{1}$
a = 8.8898 (8) Å
b = 8.9885 (9) Å
c = 12.1973 (10) Å
α = 89.908 (8)°
β = 84.490 (7)°
γ = 63.534 (9)°
V = 867.53 (15) Å^3
Z = 2
$F(000)$ = 360
D_x = 1.316 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2206 reflections
θ = 5.5–70.9°
μ = 3.09 mm^{-1}
T = 293 K
Prism, colourless
0.34 × 0.32 × 0.14 mm

Data collection

Rigaku Oxford Diffraction diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku Oxford Diffraction, 2015)
T_{min} = 0.382, T_{max} = 1.000
5496 measured reflections
3279 independent reflections
2807 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 71.5°, θ_{min} = 3.6°
h = −10→10
k = −11→10
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.146
S = 1.03
3279 reflections
210 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0787P)^2 + 0.2112P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0051 (9)

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.09239 (15)	1.18172 (14)	−0.02253 (9)	0.1305 (5)
S1	0.73755 (8)	0.52007 (7)	0.23423 (4)	0.0561 (2)
O1	0.7170 (2)	0.7404 (2)	0.50943 (14)	0.0629 (5)
N1	0.7797 (2)	0.4711 (2)	0.47968 (14)	0.0461 (4)
C1	0.7158 (3)	0.6329 (3)	0.44911 (17)	0.0467 (5)
C2	0.6464 (3)	0.6753 (3)	0.34053 (17)	0.0447 (4)
C3	0.7545 (3)	0.3470 (3)	0.30826 (19)	0.0502 (5)
C4	0.7543 (3)	0.2137 (3)	0.2500 (2)	0.0667 (7)
H4	0.7439	0.2195	0.1747	0.080*
C5	0.7696 (4)	0.0737 (4)	0.3035 (3)	0.0785 (8)
H5	0.7724	−0.0163	0.2643	0.094*
C6	0.7806 (4)	0.0673 (3)	0.4149 (3)	0.0784 (8)
H6	0.7886	−0.0266	0.4513	0.094*
C7	0.7800 (3)	0.1990 (3)	0.4738 (2)	0.0644 (6)
H7	0.7868	0.1934	0.5494	0.077*
C8	0.7693 (3)	0.3403 (3)	0.42042 (18)	0.0470 (5)
C9	0.5312 (3)	0.8316 (3)	0.32848 (17)	0.0487 (5)
H9	0.5091	0.9029	0.3895	0.058*
C10	0.4338 (3)	0.9109 (3)	0.23699 (18)	0.0505 (5)
C11	0.2955 (4)	1.0643 (3)	0.2594 (2)	0.0692 (7)
H11	0.2722	1.1131	0.3301	0.083*
C12	0.1918 (4)	1.1465 (4)	0.1810 (3)	0.0783 (8)
H12	0.0998	1.2496	0.1985	0.094*
C13	0.2237 (4)	1.0772 (4)	0.0775 (3)	0.0783 (8)
C14	0.3614 (6)	0.9286 (4)	0.0506 (3)	0.1164 (16)
H14	0.3849	0.8826	−0.0209	0.140*
C15	0.4653 (5)	0.8471 (4)	0.1298 (2)	0.0944 (12)
H15	0.5595	0.7462	0.1106	0.113*
C16	0.8499 (3)	0.4380 (3)	0.58673 (17)	0.0513 (5)
H16A	0.9118	0.5024	0.5944	0.062*
H16B	0.9298	0.3213	0.5868	0.062*
C17	0.7187 (3)	0.4791 (3)	0.68574 (19)	0.0560 (6)
H17A	0.6484	0.4242	0.6754	0.067*
H17B	0.6467	0.5981	0.6918	0.067*
C18	0.8018 (4)	0.4236 (4)	0.7914 (2)	0.0733 (8)
H18A	0.8518	0.3032	0.7922	0.088*
H18B	0.8921	0.4561	0.7923	0.088*
C19	0.6794 (5)	0.4967 (5)	0.8941 (2)	0.1002 (12)
H19A	0.5910	0.4630	0.8945	0.150*

H19B	0.6314	0.6159	0.8947	0.150*
H19C	0.7382	0.4577	0.9582	0.150*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1339 (9)	0.1157 (8)	0.0988 (7)	−0.0060 (6)	−0.0706 (7)	0.0190 (6)
S1	0.0688 (4)	0.0496 (3)	0.0409 (3)	−0.0181 (3)	−0.0068 (2)	0.0018 (2)
O1	0.0915 (12)	0.0568 (10)	0.0514 (9)	−0.0403 (9)	−0.0224 (8)	0.0055 (7)
N1	0.0508 (9)	0.0494 (10)	0.0414 (9)	−0.0247 (8)	−0.0084 (7)	0.0074 (7)
C1	0.0537 (11)	0.0504 (11)	0.0423 (10)	−0.0286 (9)	−0.0069 (8)	0.0036 (9)
C2	0.0519 (11)	0.0448 (11)	0.0418 (10)	−0.0253 (9)	−0.0057 (8)	0.0028 (8)
C3	0.0483 (11)	0.0447 (11)	0.0558 (12)	−0.0189 (9)	−0.0062 (9)	0.0007 (9)
C4	0.0734 (16)	0.0558 (14)	0.0703 (16)	−0.0281 (12)	−0.0084 (13)	−0.0105 (12)
C5	0.089 (2)	0.0566 (15)	0.094 (2)	−0.0385 (14)	0.0012 (16)	−0.0144 (15)
C6	0.0883 (19)	0.0477 (14)	0.101 (2)	−0.0353 (13)	0.0089 (16)	0.0044 (14)
C7	0.0722 (15)	0.0531 (13)	0.0659 (15)	−0.0282 (12)	0.0025 (12)	0.0074 (11)
C8	0.0428 (10)	0.0425 (11)	0.0544 (12)	−0.0185 (8)	−0.0017 (8)	0.0026 (9)
C9	0.0598 (12)	0.0453 (11)	0.0436 (11)	−0.0254 (9)	−0.0068 (9)	0.0006 (8)
C10	0.0579 (12)	0.0445 (11)	0.0482 (11)	−0.0215 (9)	−0.0089 (9)	0.0025 (9)
C11	0.0773 (16)	0.0573 (14)	0.0561 (14)	−0.0139 (12)	−0.0132 (12)	−0.0074 (11)
C12	0.0662 (16)	0.0634 (16)	0.0769 (18)	−0.0019 (13)	−0.0160 (14)	0.0033 (14)
C13	0.0865 (19)	0.0689 (17)	0.0680 (17)	−0.0186 (14)	−0.0388 (15)	0.0127 (13)
C14	0.154 (3)	0.073 (2)	0.0598 (18)	0.011 (2)	−0.045 (2)	−0.0094 (15)
C15	0.116 (2)	0.0600 (16)	0.0530 (15)	0.0116 (16)	−0.0220 (16)	−0.0036 (12)
C16	0.0461 (11)	0.0623 (13)	0.0461 (11)	−0.0237 (10)	−0.0110 (9)	0.0111 (9)
C17	0.0521 (12)	0.0673 (14)	0.0468 (12)	−0.0246 (10)	−0.0075 (9)	0.0119 (10)
C18	0.0680 (15)	0.096 (2)	0.0485 (13)	−0.0281 (14)	−0.0131 (11)	0.0150 (13)
C19	0.098 (2)	0.141 (3)	0.0465 (15)	−0.040 (2)	−0.0061 (15)	0.0107 (17)

Geometric parameters (Å, º)

Cl1—C13	1.740 (3)	C10—C15	1.380 (4)
S1—C2	1.756 (2)	C11—H11	0.9300
S1—C3	1.753 (2)	C11—C12	1.368 (4)
O1—C1	1.220 (3)	C12—H12	0.9300
N1—C1	1.371 (3)	C12—C13	1.355 (4)
N1—C8	1.424 (3)	C13—C14	1.363 (4)
N1—C16	1.474 (3)	C14—H14	0.9300
C1—C2	1.492 (3)	C14—C15	1.374 (4)
C2—C9	1.337 (3)	C15—H15	0.9300
C3—C4	1.395 (3)	C16—H16A	0.9700
C3—C8	1.386 (3)	C16—H16B	0.9700
C4—H4	0.9300	C16—C17	1.517 (3)
C4—C5	1.375 (4)	C17—H17A	0.9700
C5—H5	0.9300	C17—H17B	0.9700
C5—C6	1.371 (5)	C17—C18	1.519 (3)
C6—H6	0.9300	C18—H18A	0.9700

C6—C7	1.384 (4)	C18—H18B	0.9700
C7—H7	0.9300	C18—C19	1.512 (4)
C7—C8	1.395 (3)	C19—H19A	0.9600
C9—H9	0.9300	C19—H19B	0.9600
C9—C10	1.462 (3)	C19—H19C	0.9600
C10—C11	1.383 (3)		

C3—S1—C2	99.73 (10)	C11—C12—H12	120.1
C1—N1—C8	124.60 (18)	C13—C12—C11	119.8 (3)
C1—N1—C16	115.97 (17)	C13—C12—H12	120.1
C8—N1—C16	119.20 (17)	C12—C13—Cl1	119.9 (2)
O1—C1—N1	120.5 (2)	C12—C13—C14	120.3 (3)
O1—C1—C2	120.45 (19)	C14—C13—Cl1	119.9 (2)
N1—C1—C2	119.09 (18)	C13—C14—H14	120.2
C1—C2—S1	116.82 (15)	C13—C14—C15	119.6 (3)
C9—C2—S1	124.85 (17)	C15—C14—H14	120.2
C9—C2—C1	118.04 (19)	C10—C15—H15	119.1
C4—C3—S1	117.58 (19)	C14—C15—C10	121.9 (3)
C8—C3—S1	121.84 (17)	C14—C15—H15	119.1
C8—C3—C4	120.6 (2)	N1—C16—H16A	108.7
C3—C4—H4	119.9	N1—C16—H16B	108.7
C5—C4—C3	120.1 (3)	N1—C16—C17	114.37 (17)
C5—C4—H4	119.9	H16A—C16—H16B	107.6
C4—C5—H5	120.1	C17—C16—H16A	108.7
C6—C5—C4	119.8 (3)	C17—C16—H16B	108.7
C6—C5—H5	120.1	C16—C17—H17A	109.4
C5—C6—H6	119.7	C16—C17—H17B	109.4
C5—C6—C7	120.7 (3)	C16—C17—C18	111.19 (19)
C7—C6—H6	119.7	H17A—C17—H17B	108.0
C6—C7—H7	119.8	C18—C17—H17A	109.4
C6—C7—C8	120.4 (3)	C18—C17—H17B	109.4
C8—C7—H7	119.8	C17—C18—H18A	109.0
C3—C8—N1	121.16 (19)	C17—C18—H18B	109.0
C3—C8—C7	118.4 (2)	H18A—C18—H18B	107.8
C7—C8—N1	120.4 (2)	C19—C18—C17	112.9 (2)
C2—C9—H9	114.1	C19—C18—H18A	109.0
C2—C9—C10	131.8 (2)	C19—C18—H18B	109.0
C10—C9—H9	114.1	C18—C19—H19A	109.5
C11—C10—C9	117.3 (2)	C18—C19—H19B	109.5
C15—C10—C9	126.4 (2)	C18—C19—H19C	109.5
C15—C10—C11	116.4 (2)	H19A—C19—H19B	109.5
C10—C11—H11	119.0	H19A—C19—H19C	109.5
C12—C11—C10	122.1 (2)	H19B—C19—H19C	109.5
C12—C11—H11	119.0		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1^{i}	0.93	2.53	3.410 (3)	158
C11—H11···O1^{ii}	0.93	2.39	3.290 (3)	162
C15—H15···S1	0.93	2.55	3.219 (3)	129

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