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ISSN 2414-3146

2-[(2*Z*)-2-Benzylidene-3-oxo-3,4-dihydro-2*H*-1,4-benzothiazin-4-yl]acetic acid

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Received 27 May 2016
Accepted 29 May 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

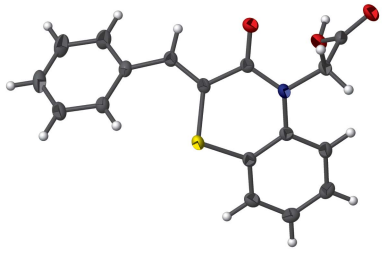
Keywords: crystal structure; hydrogen bonding; benzothiazine.

CCDC reference: 1482425

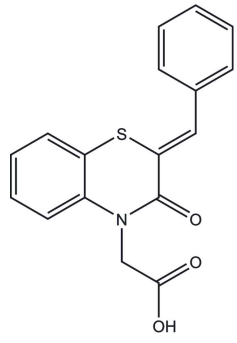
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{13}NO_3S$, the thiazine ring displays a screw-boat conformation. The dihedral angle between the terminal benzene ring and the mean plane of the 1,4-benzothiazine fused-ring system is 51.52 (6)°. In the crystal, inversion dimers linked by pairs of O—H···O_t (t = thiazine) hydrogen bonds generate $R_2^2(14)$ loops. The dimers are linked by pairwise C—H···O hydrogen bonds, resulting in [010] chains.

3D view



Chemical scheme



Structure description

As a continuation of our studies of substituted 1,4-benzothiazine derivatives (Ellouz *et al.*, 2015; Sebbar *et al.*, 2015), we report the synthesis of a 1,4-benzothiazine derivative (Fig. 1) by the hydrolysis reaction with aqueous solution of potassium hydroxide of ethyl 2-(3-oxo-2,3-dihydro[1,4]-benzothiazin-4-yl)acetate in ethanol.

A puckering analysis of the heterocyclic ring gave parameters Q = 0.430 (1) Å, θ = 106.1 (2)° and φ = 167.5 (2)°. The dihedral angle between the C1–C6 and C10–C15 rings is 56.70 (5)°. In the crystal, pair-wise O2—H2*A*···O1^{i} [symmetry code: (i) $2-x, 1-y, 2-z$) hydrogen bonds form inversion dimers, which are connected into chains running parallel to the *b* axis by pair-wise C16—H16*A*···O3^{ii} [symmetry code: (ii) $2-x, -y, 2-z$) hydrogen bonds (Table 1 and Fig. 2).

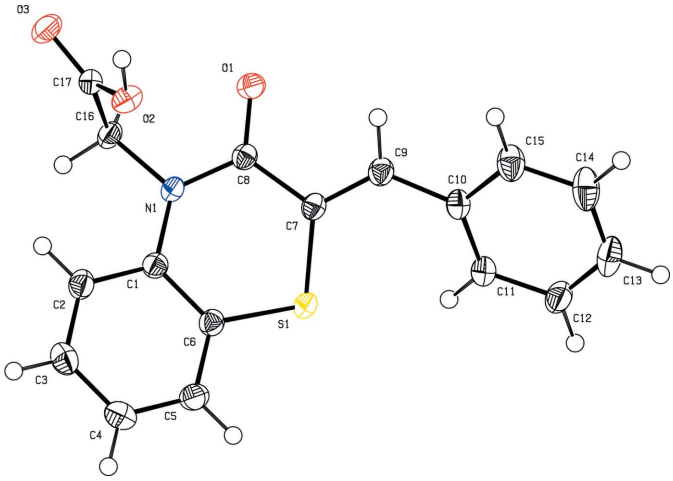


Figure 1
The title molecule with 50% probability ellipsoids.

Synthesis and crystallization

A solution of potassium hydroxide (12.5 mmol) in water (5 ml) was added to the solution of ethyl (*Z*)-2-(2-benzylidene-2,3-dihydro-[1,4]-benzothiazin-3-one-4-yl)acetate(3.07 - mmol) in ethanol (10 ml). The resulting reaction mixture was stirred at room temperature for 6 h and the reaction completion was checked by TLC. The reaction mixture was poured into water and acidified with 3 *M* HCl to form the title compound as a colorless solid. The solid product was purified by recrystallization from ethanol solution to afford colorless crystals in 80% yield.

Refinement

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

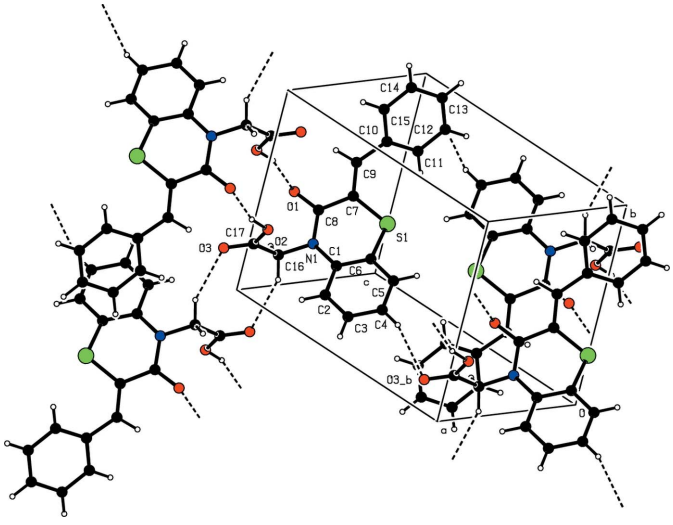


Figure 2
A part of the unit cell showing intermolecular hydrogen bonds (dotted lines) in the crystal structure of the title compound.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O1^{i}	0.92 (2)	1.78 (2)	2.6591 (13)	161 (2)
C16—H16*A*···O3^{ii}	0.99	2.47	3.3583 (16)	149

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{13}NO_3S$
M_r	311.34
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, *b*, *c* (Å)	8.6760 (3), 8.7882 (3), 9.8142 (4)
α, β, γ (°)	78.200 (1), 73.311 (1), 86.242 (1)
V (Å^3)	701.61 (4)
Z	2
Radiation type	Cu *K*α
μ (mm^{-1})	2.16
Crystal size (mm)	0.17 × 0.11 × 0.09
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.68, 0.83
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20850, 2816, 2698
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.032, 0.083, 1.04
No. of reflections	2816
No. of parameters	203
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.32

Computer programs: *APEX2* and *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Acknowledgements

The support of NSF–MRI Grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

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full crystallographic data

IUCrData (2016). **1**, x160863 [doi:10.1107/S2414314616008634]

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Crystal data

$C_{17}H_{13}NO_3S$
M_r = 311.34
Triclinic, $P\overline{1}$
a = 8.6760 (3) Å
b = 8.7882 (3) Å
c = 9.8142 (4) Å
α = 78.200 (1)°
β = 73.311 (1)°
γ = 86.242 (1)°
V = 701.61 (4) Å^3
Z = 2
$F(000)$ = 324
D_x = 1.474 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9893 reflections
θ = 4.8–74.7°
μ = 2.16 mm^{-1}
T = 150 K
Block, colourless
0.17 × 0.11 × 0.09 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2015)
T_{min} = 0.68, T_{max} = 0.83
20850 measured reflections
2816 independent reflections
2698 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 74.6°, θ_{min} = 4.8°
h = −10→10
k = −9→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 1.04
2816 reflections
203 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 0.2923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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.

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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å). All were included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.61329 (4)	0.52854 (4)	0.64991 (3)	0.02465 (11)
O1	0.73459 (11)	0.46100 (11)	1.01298 (10)	0.0243 (2)
O2	1.08484 (12)	0.38446 (11)	0.88403 (10)	0.0264 (2)
H2A	1.159 (3)	0.417 (3)	0.922 (2)	0.056 (6)*
O3	1.06908 (13)	0.17480 (12)	1.06181 (11)	0.0322 (2)
N1	0.80930 (13)	0.32446 (12)	0.83337 (11)	0.0207 (2)
C1	0.85510 (15)	0.31621 (15)	0.68359 (14)	0.0206 (3)
C2	0.98019 (16)	0.21791 (16)	0.62797 (15)	0.0253 (3)
H2	1.0367	0.1572	0.6903	0.030*
C3	1.02251 (17)	0.20820 (17)	0.48272 (16)	0.0291 (3)
H3	1.1070	0.1400	0.4465	0.035*
C4	0.94287 (18)	0.29701 (17)	0.38981 (15)	0.0294 (3)
H4	0.9717	0.2894	0.2904	0.035*
C5	0.82079 (17)	0.39689 (16)	0.44328 (15)	0.0264 (3)
H5	0.7671	0.4596	0.3797	0.032*
C6	0.77588 (15)	0.40624 (15)	0.58941 (14)	0.0217 (3)
C7	0.66020 (15)	0.57311 (15)	0.79937 (14)	0.0207 (3)
C8	0.73771 (15)	0.45155 (15)	0.88811 (13)	0.0202 (3)
C9	0.61808 (15)	0.70741 (15)	0.84638 (15)	0.0230 (3)
H9	0.6471	0.7150	0.9310	0.028*
C10	0.53440 (15)	0.84350 (15)	0.78719 (14)	0.0228 (3)
C11	0.42571 (16)	0.83932 (16)	0.70696 (15)	0.0263 (3)
H11	0.4039	0.7434	0.6861	0.032*
C12	0.34936 (18)	0.97462 (18)	0.65750 (16)	0.0318 (3)
H12	0.2765	0.9708	0.6022	0.038*
C13	0.37898 (18)	1.11481 (17)	0.68845 (17)	0.0346 (3)
H13	0.3277	1.2072	0.6532	0.042*
C14	0.48335 (19)	1.12025 (18)	0.7708 (2)	0.0376 (4)
H14	0.5021	1.2161	0.7937	0.045*
C15	0.56053 (17)	0.98580 (17)	0.81981 (18)	0.0319 (3)
H15	0.6320	0.9903	0.8762	0.038*
C16	0.86963 (16)	0.20705 (15)	0.93505 (14)	0.0229 (3)
H16A	0.8936	0.1106	0.8952	0.027*
H16B	0.7832	0.1831	1.0274	0.027*

C17	1.01887 (16)	0.25291 (15)	0.96737 (14)	0.0222 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02793 (18)	0.02328 (18)	0.02754 (18)	0.00799 (12)	−0.01553 (13)	−0.00721 (12)
O1	0.0268 (5)	0.0251 (5)	0.0222 (4)	0.0002 (4)	−0.0094 (4)	−0.0041 (3)
O2	0.0276 (5)	0.0275 (5)	0.0256 (5)	−0.0022 (4)	−0.0124 (4)	−0.0007 (4)
O3	0.0417 (6)	0.0271 (5)	0.0335 (5)	0.0042 (4)	−0.0232 (5)	−0.0019 (4)
N1	0.0226 (5)	0.0191 (5)	0.0218 (5)	0.0035 (4)	−0.0101 (4)	−0.0029 (4)
C1	0.0213 (6)	0.0196 (6)	0.0225 (6)	−0.0009 (5)	−0.0082 (5)	−0.0041 (5)
C2	0.0232 (6)	0.0237 (6)	0.0306 (7)	0.0037 (5)	−0.0104 (5)	−0.0059 (5)
C3	0.0259 (7)	0.0283 (7)	0.0334 (7)	0.0029 (5)	−0.0055 (6)	−0.0116 (6)
C4	0.0328 (7)	0.0307 (7)	0.0251 (7)	−0.0020 (6)	−0.0062 (6)	−0.0089 (5)
C5	0.0308 (7)	0.0261 (7)	0.0244 (6)	−0.0006 (5)	−0.0116 (5)	−0.0036 (5)
C6	0.0230 (6)	0.0190 (6)	0.0246 (6)	0.0001 (5)	−0.0091 (5)	−0.0040 (5)
C7	0.0183 (6)	0.0205 (6)	0.0236 (6)	0.0009 (5)	−0.0073 (5)	−0.0031 (5)
C8	0.0187 (6)	0.0200 (6)	0.0225 (6)	−0.0007 (5)	−0.0072 (5)	−0.0032 (5)
C9	0.0201 (6)	0.0230 (7)	0.0272 (6)	0.0016 (5)	−0.0088 (5)	−0.0053 (5)
C10	0.0197 (6)	0.0207 (6)	0.0265 (6)	0.0026 (5)	−0.0046 (5)	−0.0046 (5)
C11	0.0264 (6)	0.0246 (7)	0.0289 (7)	0.0061 (5)	−0.0095 (5)	−0.0069 (5)
C12	0.0297 (7)	0.0339 (8)	0.0305 (7)	0.0104 (6)	−0.0105 (6)	−0.0038 (6)
C13	0.0296 (7)	0.0242 (7)	0.0411 (8)	0.0068 (6)	−0.0042 (6)	0.0035 (6)
C14	0.0306 (7)	0.0202 (7)	0.0595 (10)	0.0005 (6)	−0.0096 (7)	−0.0071 (6)
C15	0.0254 (7)	0.0257 (7)	0.0473 (9)	0.0013 (5)	−0.0125 (6)	−0.0100 (6)
C16	0.0259 (6)	0.0195 (6)	0.0236 (6)	0.0027 (5)	−0.0107 (5)	−0.0007 (5)
C17	0.0255 (6)	0.0219 (6)	0.0207 (6)	0.0061 (5)	−0.0086 (5)	−0.0061 (5)

Geometric parameters (Å, º)

S1—C7	1.7519 (13)	C7—C9	1.3449 (18)
S1—C6	1.7546 (13)	C7—C8	1.4878 (17)
O1—C8	1.2376 (16)	C9—C10	1.4626 (18)
O2—C17	1.3282 (17)	C9—H9	0.9500
O2—H2A	0.92 (2)	C10—C11	1.3976 (19)
O3—C17	1.2052 (16)	C10—C15	1.400 (2)
N1—C8	1.3729 (16)	C11—C12	1.3905 (19)
N1—C1	1.4242 (16)	C11—H11	0.9500
N1—C16	1.4637 (15)	C12—C13	1.384 (2)
C1—C6	1.3954 (18)	C12—H12	0.9500
C1—C2	1.3970 (18)	C13—C14	1.384 (2)
C2—C3	1.385 (2)	C13—H13	0.9500
C2—H2	0.9500	C14—C15	1.387 (2)
C3—C4	1.385 (2)	C14—H14	0.9500
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.384 (2)	C16—C17	1.5154 (18)
C4—H4	0.9500	C16—H16A	0.9900
C5—C6	1.3923 (19)	C16—H16B	0.9900

C5—H5	0.9500

C7—S1—C6	100.41 (6)	C7—C9—H9	115.1
C17—O2—H2A	109.4 (14)	C10—C9—H9	115.1
C8—N1—C1	124.44 (10)	C11—C10—C15	118.42 (12)
C8—N1—C16	114.83 (10)	C11—C10—C9	124.55 (12)
C1—N1—C16	119.46 (10)	C15—C10—C9	116.96 (12)
C6—C1—C2	118.67 (12)	C12—C11—C10	120.45 (13)
C6—C1—N1	120.63 (11)	C12—C11—H11	119.8
C2—C1—N1	120.71 (11)	C10—C11—H11	119.8
C3—C2—C1	120.52 (12)	C13—C12—C11	120.32 (14)
C3—C2—H2	119.7	C13—C12—H12	119.8
C1—C2—H2	119.7	C11—C12—H12	119.8
C4—C3—C2	120.62 (13)	C12—C13—C14	119.93 (13)
C4—C3—H3	119.7	C12—C13—H13	120.0
C2—C3—H3	119.7	C14—C13—H13	120.0
C5—C4—C3	119.30 (13)	C13—C14—C15	120.01 (14)
C5—C4—H4	120.4	C13—C14—H14	120.0
C3—C4—H4	120.4	C15—C14—H14	120.0
C4—C5—C6	120.59 (13)	C14—C15—C10	120.83 (14)
C4—C5—H5	119.7	C14—C15—H15	119.6
C6—C5—H5	119.7	C10—C15—H15	119.6
C5—C6—C1	120.28 (12)	N1—C16—C17	115.04 (11)
C5—C6—S1	117.61 (10)	N1—C16—H16A	108.5
C1—C6—S1	122.06 (10)	C17—C16—H16A	108.5
C9—C7—C8	117.37 (12)	N1—C16—H16B	108.5
C9—C7—S1	123.59 (10)	C17—C16—H16B	108.5
C8—C7—S1	118.80 (9)	H16A—C16—H16B	107.5
O1—C8—N1	119.20 (11)	O3—C17—O2	124.70 (13)
O1—C8—C7	120.81 (11)	O3—C17—C16	121.37 (12)
N1—C8—C7	119.97 (11)	O2—C17—C16	113.93 (11)
C7—C9—C10	129.89 (13)		

C8—N1—C1—C6	−26.36 (19)	C16—N1—C8—C7	−175.22 (11)
C16—N1—C1—C6	167.24 (12)	C9—C7—C8—O1	13.88 (18)
C8—N1—C1—C2	153.63 (12)	S1—C7—C8—O1	−160.73 (10)
C16—N1—C1—C2	−12.77 (18)	C9—C7—C8—N1	−168.25 (12)
C6—C1—C2—C3	−1.1 (2)	S1—C7—C8—N1	17.15 (16)
N1—C1—C2—C3	178.87 (12)	C8—C7—C9—C10	−176.82 (12)
C1—C2—C3—C4	0.7 (2)	S1—C7—C9—C10	−2.5 (2)
C2—C3—C4—C5	0.5 (2)	C7—C9—C10—C11	27.5 (2)
C3—C4—C5—C6	−1.2 (2)	C7—C9—C10—C15	−155.58 (14)
C4—C5—C6—C1	0.8 (2)	C15—C10—C11—C12	1.8 (2)
C4—C5—C6—S1	−176.86 (11)	C9—C10—C11—C12	178.69 (13)
C2—C1—C6—C5	0.36 (19)	C10—C11—C12—C13	−0.6 (2)
N1—C1—C6—C5	−179.65 (12)	C11—C12—C13—C14	−0.9 (2)
C2—C1—C6—S1	177.95 (10)	C12—C13—C14—C15	1.2 (2)
N1—C1—C6—S1	−2.06 (17)	C13—C14—C15—C10	0.0 (2)

C7—S1—C6—C5	−153.85 (11)	C11—C10—C15—C14	−1.5 (2)
C7—S1—C6—C1	28.50 (12)	C9—C10—C15—C14	−178.61 (14)
C6—S1—C7—C9	150.49 (12)	C8—N1—C16—C17	−72.15 (14)
C6—S1—C7—C8	−35.26 (11)	C1—N1—C16—C17	95.52 (14)
C1—N1—C8—O1	−164.28 (11)	N1—C16—C17—O3	171.05 (12)
C16—N1—C8—O1	2.69 (17)	N1—C16—C17—O2	−8.42 (16)
C1—N1—C8—C7	17.81 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O1^{i}	0.92 (2)	1.78 (2)	2.6591 (13)	161 (2)
C16—H16*A*···O3^{ii}	0.99	2.47	3.3583 (16)	149

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