Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

Crystal structure of (*Z*)-2-benzylidene-4-methyl-2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one

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Received 7 October 2015; accepted 12 October 2015

Edited by E. R. T. Tiekink, University of Malaya, Malaysia

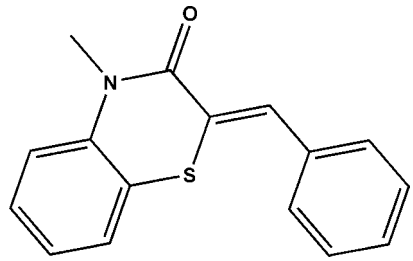
In the title compound, $C_{16}H_{13}NOS$, the 1,4-thiazine ring displays a screw-boat conformation. The conformation about the ethene bond [1.344 (2) Å] is *Z*. The plane of the fused benzene ring makes a dihedral angle of 58.95 (9)° with the pendent phenyl ring, indicating a twisted conformation in the molecule. In the crystal, molecules are linked by pairs of C—H···O hydrogen bonds, forming inversion dimers.

Keywords: crystal structure; thiomorpholin-3-one derivative; benzothiazine derivative; hydrogen bonding.

CCDC reference: 1430755

1. Related literature

For background to the pharmacological activity and potential applications of benzothiazines, see: Schiaffella *et al.* (2006); Gupta *et al.* (2009); Armenise *et al.* (2000); Bansode *et al.* (2009); Dixit *et al.* (2009); Dixit *et al.* (2008); Thomas *et al.* (2003). For medicinal applications; see: Warren *et al.* (1987); Armenise *et al.* (2012); Sabatini *et al.* (2008); Jacquot *et al.* (2001); Kalluraya *et al.* (2005); Munirajasekar *et al.* (2011). For similar compounds, see: Sebbar *et al.* (2014*a*,*b*); Zerzouf *et al.* (2001).



2. Experimental

2.1. Crystal data

$C_{16}H_{13}NOS$
M_r = 267.33
Monoclinic, $P2_1/c$
a = 9.1497 (3) Å
b = 14.7052 (5) Å
c = 10.0037 (3) Å
β = 97.051 (1)°
V = 1335.80 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 296 K
0.36 × 0.31 × 0.26 mm

2.2. Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.670, T_{max} = 0.746
25334 measured reflections
4082 independent reflections
3071 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.153
S = 1.12
4082 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O1^{i}	0.93	2.50	3.404 (2)	164

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Acknowledgements

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements and the University Mohammed V, Rabat, Morocco, for financial support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5394).

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supporting information

Acta Cryst. (2015). E**71**, o862–o863 [doi:10.1107/S2056989015019295]

Crystal structure of (*Z*)-2-benzylidene-4-methyl-2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one

Mohamed Ellouz, Nada Kheira Sebbar, El Mokhtar Essassi, Mohamed Saadi and Lahcen El Ammari

S1. Comment

Over the years, 4*H*-1,4-benzothiazines have constituted an important class of heterocycles which, even when part of a complex molecule, possess a wide spectrum of biological activities (Schiaffella *et al.*, 2006; Gupta *et al.*, 2009; Armenise *et al.*, 2000). Due to the presence of a fold along the nitrogen—sulfur axis, the biological activity of some 1,4-benzothiazines is similar to that of phenothiazines, featuring the same structural specificity (Bansode *et al.*, 2009; Dixit *et al.*, 2009; Dixit *et al.*, 2008; Thomas *et al.*, 2003). Generally, benzothiazine and derivatives have found widespread application as analgesic (Warren *et al.*, 1987), antibacterial (Armenise *et al.*, 2012; Sabatini *et al.*, 2008), anticancer (Jacquot *et al.*, 2001), anticonvulsant (Kalluraya *et al.*, 2005) and anthelmintic (Munirajasekar *et al.*, 2011) agents. As a continuation of our research work devoted to the development of N-substituted benzothiazine and evaluating their potential pharmacological activities, we have checked the action of iodomethane towards (*E*)-2-benzylidene-2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one under phase-transfer catalysis conditions using tetra n-butylammonium bromide (TBAB) as catalyst and potassium carbonate as base (Sebbar *et al.*, 2014*a*, Sebbar *et al.*, 2014*b*; Zerzouf *et al.*, 2001). This led to the characterization of the title compound, Scheme 1.

The molecule of the title compound is build up from two fused six-membered rings linked as shown in Fig.1. The dihedral angle between the (C1 to C6) and (C11 to C16) benzene rings is 58.95 (9)°.

In the crystal, the molecules are linked together by a hydrogen bond (C12–H12···O1) in a way to build a dimer, as shown in Fig. 2 and Table 1.

S2. Experimental

To a solution of 2-(benzylidene)-3,4-dihydro-2*H*-1,4-benzothiazin-3-one (0.5 g, 2 mmol), potassium carbonate (0.55 g, 4 mmol) and tetra *n*-butyl ammonium bromide (0.064 g, 0.2 mmol) in DMF (15 ml) was added iodomethane (0.25 ml, 4 mmol). Stirring was continued at room temperature for 12 h. The mixture was filtered and the solvent removed. The residue was extracted with water. The organic compound was chromatographed on a column of silica gel with ethyl acetate-hexane (9/1) as eluent. Brown crystals were isolated when the solvent was allowed to evaporate (yield: 53%; m.p. = 342 K).

S3. Refinement

The H atoms were located in a difference map and treated as riding with C—H = 0.93 Å (aromatic) and C—H = 0.96 Å (methyl), and with U_{iso}(H) = 1.2 U_{eq} (aromatic) and U_{iso}(H) = 1.5 U_{eq} (methyl).

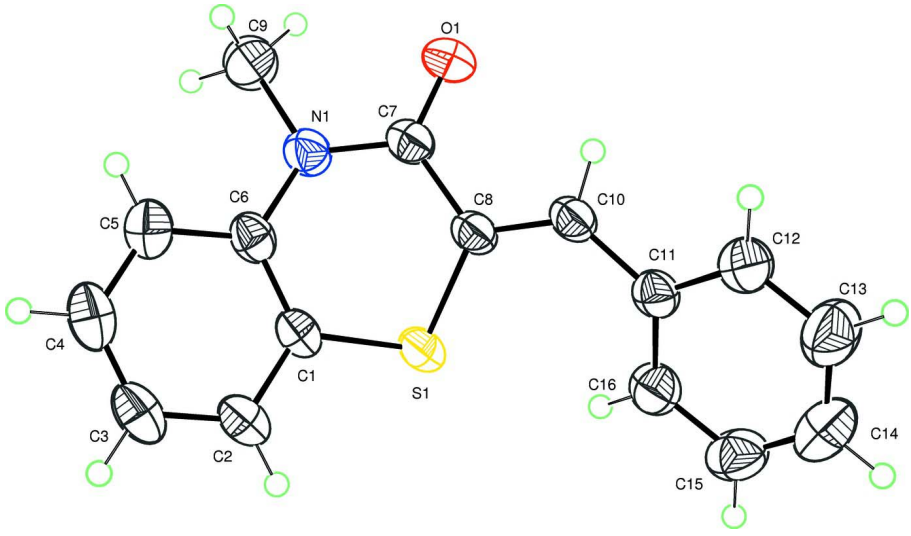

Figure 1

Molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

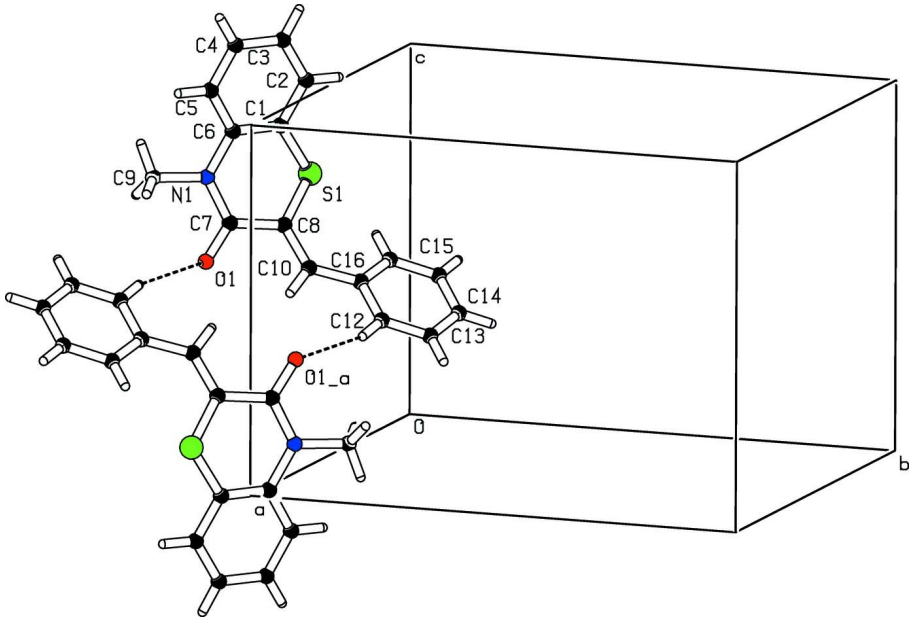

Figure 2

Supramolecular association in the title compound, showing inversion dimers of molecules linked through C12—H12···O1 hydrogen bond (dashed lines).

(*Z*)-2-Benzylidene-4-methyl-2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one

Crystal data

$C_{16}H_{13}NOS$
$M_r = 267.33$
Monoclinic, $P2_1/c$
$a = 9.1497$ (3) Å
$b = 14.7052$ (5) Å
$c = 10.0037$ (3) Å
$\beta = 97.051$ (1)°
$V = 1335.80$ (7) Å^3

$Z = 4$
$F(000) = 560$
$D_x = 1.329$ Mg m^{-3}
Melting point: 342 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4082 reflections

$\theta = 2.2$–$30.5°$
$\mu = 0.23$ mm^{-1}
$T = 296$ K
Prism, brown
0.36 × 0.31 × 0.26 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.670$, $T_{max} = 0.746$

25334 measured reflections
4082 independent reflections
3071 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 30.5°$, $\theta_{min} = 2.2°$
$h = -7 \rightarrow 13$
$k = -20 \rightarrow 21$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.153$
$S = 1.12$
4082 reflections
172 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0757P)^2 + 0.2379P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.73318 (17)	0.52736 (10)	0.43834 (14)	0.0426 (3)
C2	0.6952 (2)	0.48778 (12)	0.55605 (16)	0.0513 (4)
H2	0.6186	0.4459	0.5516	0.062*
C3	0.7715 (2)	0.51089 (13)	0.67964 (16)	0.0579 (5)
H3	0.7445	0.4862	0.7586	0.070*
C4	0.8872 (2)	0.57054 (13)	0.68451 (17)	0.0596 (4)
H4	0.9383	0.5863	0.7675	0.071*
C5	0.9292 (2)	0.60761 (12)	0.56848 (17)	0.0549 (4)
H5	1.0100	0.6464	0.5737	0.066*
C6	0.85101 (17)	0.58725 (10)	0.44272 (14)	0.0425 (3)
C7	0.86549 (16)	0.58789 (10)	0.19776 (14)	0.0435 (3)
C8	0.75763 (16)	0.51143 (10)	0.17601 (14)	0.0408 (3)
C9	0.9905 (3)	0.70539 (13)	0.3367 (2)	0.0689 (5)
H9A	1.0003	0.7272	0.4278	0.103*
H9B	1.0853	0.6874	0.3142	0.103*
H9C	0.9512	0.7528	0.2768	0.103*

C10	0.75953 (16)	0.45936 (11)	0.06583 (14)	0.0430 (3)
H10	0.8336	0.4735	0.0133	0.052*
C11	0.66396 (17)	0.38420 (10)	0.01526 (14)	0.0432 (3)
C12	0.7201 (2)	0.32215 (12)	−0.07117 (18)	0.0563 (4)
H12	0.8150	0.3299	−0.0935	0.068*
C13	0.6368 (3)	0.24967 (15)	−0.1236 (3)	0.0770 (6)
H13	0.6763	0.2088	−0.1804	0.092*
C14	0.4954 (3)	0.23710 (15)	−0.0928 (2)	0.0744 (6)
H14	0.4397	0.1879	−0.1283	0.089*
C15	0.4378 (2)	0.29778 (15)	−0.0094 (2)	0.0663 (5)
H15	0.3427	0.2893	0.0121	0.080*
C16	0.51940 (18)	0.37157 (13)	0.04339 (17)	0.0550 (4)
H16	0.4778	0.4130	0.0979	0.066*
N1	0.89082 (15)	0.62709 (9)	0.32356 (13)	0.0466 (3)
O1	0.92759 (14)	0.61644 (9)	0.10488 (12)	0.0591 (3)
S1	0.62428 (4)	0.50183 (3)	0.28662 (4)	0.05206 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0506 (8)	0.0451 (7)	0.0335 (6)	0.0048 (6)	0.0115 (6)	0.0010 (5)
C2	0.0614 (10)	0.0554 (9)	0.0395 (7)	0.0038 (7)	0.0162 (7)	0.0053 (6)
C3	0.0759 (12)	0.0629 (10)	0.0359 (8)	0.0138 (8)	0.0103 (8)	0.0093 (7)
C4	0.0735 (11)	0.0637 (10)	0.0390 (7)	0.0134 (9)	−0.0027 (7)	−0.0030 (7)
C5	0.0626 (10)	0.0528 (9)	0.0479 (8)	0.0018 (7)	0.0014 (7)	−0.0070 (7)
C6	0.0537 (8)	0.0380 (7)	0.0367 (7)	0.0064 (6)	0.0090 (6)	−0.0005 (5)
C7	0.0451 (7)	0.0476 (8)	0.0390 (7)	−0.0009 (6)	0.0101 (6)	0.0039 (6)
C8	0.0405 (7)	0.0504 (8)	0.0322 (6)	−0.0003 (6)	0.0079 (5)	0.0045 (5)
C9	0.0953 (15)	0.0483 (9)	0.0663 (11)	−0.0214 (9)	0.0228 (10)	−0.0069 (8)
C10	0.0425 (7)	0.0545 (8)	0.0327 (6)	−0.0003 (6)	0.0077 (5)	0.0038 (6)
C11	0.0459 (7)	0.0500 (8)	0.0328 (6)	−0.0006 (6)	0.0019 (5)	0.0059 (5)
C12	0.0566 (9)	0.0543 (9)	0.0589 (10)	−0.0009 (7)	0.0107 (8)	−0.0052 (7)
C13	0.0818 (14)	0.0585 (11)	0.0914 (16)	−0.0055 (10)	0.0138 (12)	−0.0195 (10)
C14	0.0754 (13)	0.0584 (11)	0.0862 (14)	−0.0158 (10)	−0.0033 (11)	−0.0036 (10)
C15	0.0503 (9)	0.0789 (13)	0.0677 (11)	−0.0139 (9)	−0.0012 (8)	0.0097 (10)
C16	0.0465 (8)	0.0704 (11)	0.0474 (8)	−0.0025 (7)	0.0033 (6)	−0.0006 (8)
N1	0.0583 (8)	0.0404 (6)	0.0429 (6)	−0.0045 (5)	0.0130 (5)	0.0001 (5)
O1	0.0633 (7)	0.0710 (8)	0.0461 (6)	−0.0177 (6)	0.0191 (5)	0.0037 (5)
S1	0.0473 (2)	0.0749 (3)	0.0361 (2)	−0.01066 (18)	0.01376 (16)	−0.00324 (16)

Geometric parameters (Å, º)

C1—C6	1.389 (2)	C9—N1	1.465 (2)
C1—C2	1.395 (2)	C9—H9A	0.9600
C1—S1	1.7510 (15)	C9—H9B	0.9600
C2—C3	1.386 (3)	C9—H9C	0.9600
C2—H2	0.9300	C10—C11	1.461 (2)
C3—C4	1.371 (3)	C10—H10	0.9300

C3—H3	0.9300	C11—C12	1.398 (2)
C4—C5	1.379 (3)	C11—C16	1.398 (2)
C4—H4	0.9300	C12—C13	1.376 (3)
C5—C6	1.401 (2)	C12—H12	0.9300
C5—H5	0.9300	C13—C14	1.379 (3)
C6—N1	1.4154 (19)	C13—H13	0.9300
C7—O1	1.2215 (18)	C14—C15	1.371 (3)
C7—N1	1.3777 (19)	C14—H14	0.9300
C7—C8	1.494 (2)	C15—C16	1.385 (3)
C8—C10	1.344 (2)	C15—H15	0.9300
C8—S1	1.7505 (15)	C16—H16	0.9300

C6—C1—C2	120.73 (14)	H9A—C9—H9C	109.5
C6—C1—S1	121.35 (11)	H9B—C9—H9C	109.5
C2—C1—S1	117.89 (13)	C8—C10—C11	130.37 (14)
C3—C2—C1	120.00 (17)	C8—C10—H10	114.8
C3—C2—H2	120.0	C11—C10—H10	114.8
C1—C2—H2	120.0	C12—C11—C16	117.82 (15)
C4—C3—C2	119.42 (16)	C12—C11—C10	117.27 (14)
C4—C3—H3	120.3	C16—C11—C10	124.87 (15)
C2—C3—H3	120.3	C13—C12—C11	120.86 (18)
C3—C4—C5	121.10 (16)	C13—C12—H12	119.6
C3—C4—H4	119.5	C11—C12—H12	119.6
C5—C4—H4	119.5	C12—C13—C14	120.6 (2)
C4—C5—C6	120.48 (17)	C12—C13—H13	119.7
C4—C5—H5	119.8	C14—C13—H13	119.7
C6—C5—H5	119.8	C15—C14—C13	119.39 (19)
C1—C6—C5	118.21 (14)	C15—C14—H14	120.3
C1—C6—N1	121.00 (13)	C13—C14—H14	120.3
C5—C6—N1	120.79 (15)	C14—C15—C16	120.82 (19)
O1—C7—N1	120.60 (14)	C14—C15—H15	119.6
O1—C7—C8	120.56 (14)	C16—C15—H15	119.6
N1—C7—C8	118.81 (12)	C15—C16—C11	120.45 (18)
C10—C8—C7	118.25 (13)	C15—C16—H16	119.8
C10—C8—S1	123.55 (12)	C11—C16—H16	119.8
C7—C8—S1	117.89 (11)	C7—N1—C6	124.35 (12)
N1—C9—H9A	109.5	C7—N1—C9	116.37 (13)
N1—C9—H9B	109.5	C6—N1—C9	118.12 (13)
H9A—C9—H9B	109.5	C8—S1—C1	99.44 (7)
N1—C9—H9C	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1^{i}	0.93	2.50	3.404 (2)	164

Symmetry code: (i) −*x*+2, −*y*+1, −*z*.