



Crystal structure of 4-benzyl-2H-benzo-[b][1,4]thiazin-3(4H)-one

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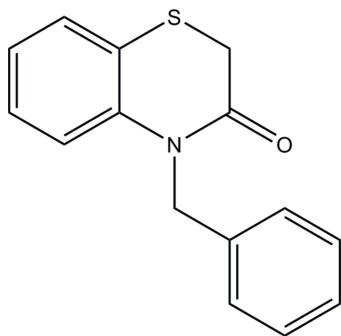
In the title compound, C₁₅H₁₃NOS, the thiazine ring adopts a twisted boat conformation and the dihedral angle between the aromatic rings is 86.54 (4)°. In the crystal, molecules are linked by weak C—H...O interactions, resulting in chains along [010].

Keywords: crystal structure; 1,4-benzothiazine derivatives; C—H...O interactions.

CCDC reference: 1438105

1. Related literature

For related structures and background to 1,4-benzothiazine derivatives, see: Zerzouf *et al.* (2001); Sebbar *et al.* (2015).



2. Experimental

2.1. Crystal data

C₁₅H₁₃NOS
 $M_r = 255.32$
 Monoclinic, $P2_1/c$
 $a = 10.8711$ (7) Å
 $b = 5.3815$ (3) Å
 $c = 21.1997$ (13) Å
 $\beta = 93.128$ (1)°
 $V = 1238.39$ (13) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.25$ mm⁻¹
 $T = 150$ K
 $0.31 \times 0.19 \times 0.15$ mm

2.2. Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2015)
 $T_{\min} = 0.88$, $T_{\max} = 0.96$
 22588 measured reflections
 3318 independent reflections
 2731 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.107$
 $S = 1.06$
 3318 reflections
 163 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.43$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C7-H7A\cdots O1^i$	0.99	2.55	3.2504 (14)	128
$C7-H7B\cdots O1^{ii}$	0.99	2.53	3.4403 (15)	152

Symmetry codes: (i) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, y + 1, z$.

Data collection: APEX2 (Bruker, 2015); cell refinement: SAINT (Bruker, 2015); data reduction: SAINT; program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2014 (Sheldrick, 2015b); molecular graphics: DIAMOND (Brandenburg & Putz, 2012); software used to prepare material for publication: SHELXTL (Sheldrick, 2015b).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7545).

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supporting information

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

N. K. Sebbar, M. Ellouz, E. M. Essassi, Y. Ouzidan and J. T. Mague

S1. Comment

As a continuation of our research devoted to the development of substituted 1,4-benzothiazine derivatives (Zerzouf *et al.*, 2001; Sebbar *et al.*, 2015), we report the synthesis of a 1,4-benzothiazine derivative by reaction of benzyl chloride with 2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one in the presence of tetra-*n*-butylammonium bromide as catalyst and potassium carbonate as base (Scheme 1).

In the title compound, the heterocyclic ring has puckering parameters $Q = 0.6272(10) \text{ \AA}$, $\theta = 63.91(10)^\circ$ and $\varphi = 325.56(11)^\circ$. The dihedral angle between the rings C1—C6 and C10—C15 is $86.54(4)^\circ$. Weak C—H $\cdots$ O interactions (Table 1) form chains running parallel to the *b* axis (Fig. 2).

S2. Experimental

To a solution of 2*H*-benzo[*b*][1,4]thiazin-3(4*H*)-one (0.543 g, 3.29 mmol), benzyl chloride (0.76 ml, 6.58 mmol) and potassium carbonate (0.91 g, 6.58 mmol) in DMF (15 ml) was added a catalytic amount of tetra-*n*-butylammonium bromide (0.11 g, 0.33 mmol) and the mixture was stirred for 24 h. The solid material was removed by filtration and the solvent evaporated under vacuum. The solid product was purified by recrystallization from ethanol to afford colorless crystals in 75% yield.

S3. Refinement

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.

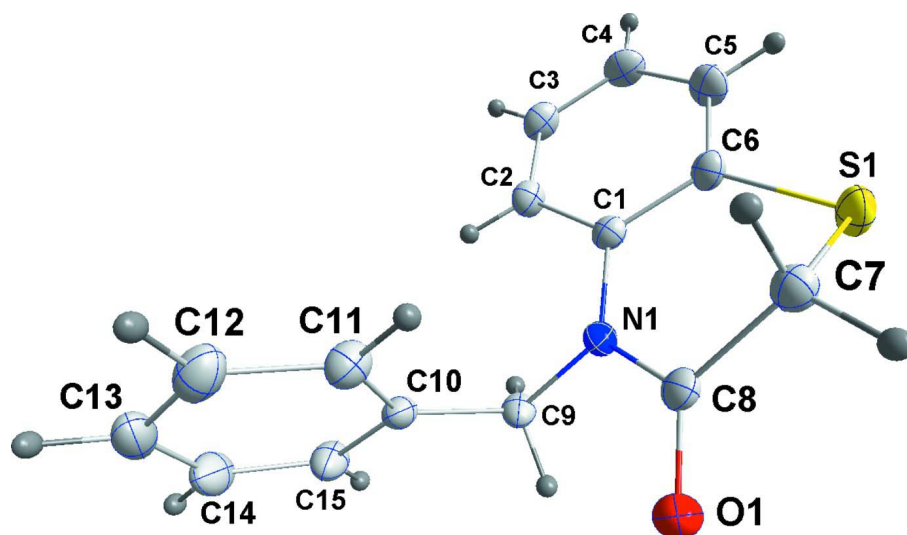


Figure 1
Perspective view of the molecule with 50% probability ellipsoids.

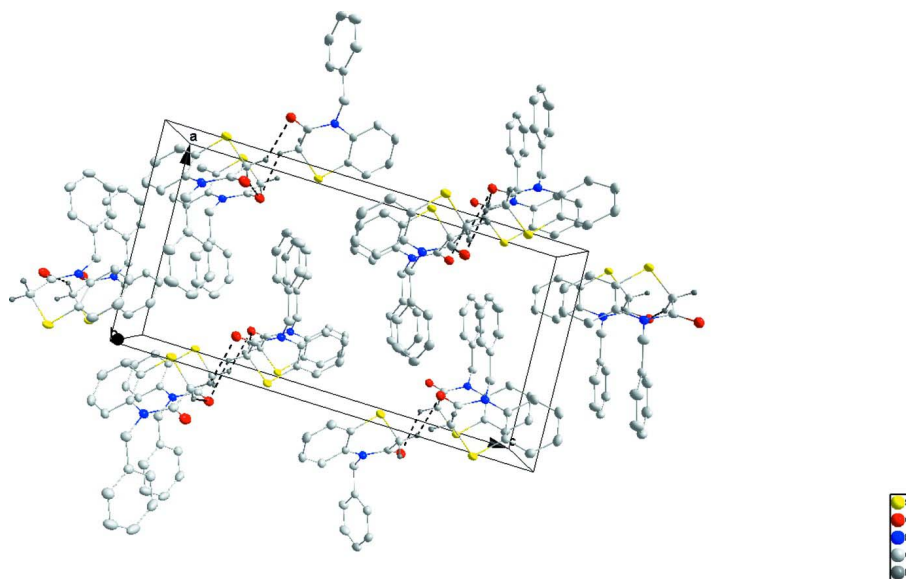


Figure 2
Packing viewed down the *b* axis. Intermolecular C—H...O interactions are shown by dotted lines.

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

Crystal data

$C_{15}H_{13}NOS$

$M_r = 255.32$

Monoclinic, $P2_1/c$

$a = 10.8711 (7) \text{ \AA}$

$b = 5.3815 (3) \text{ \AA}$

$c = 21.1997 (13) \text{ \AA}$

$\beta = 93.128 (1)^\circ$

$V = 1238.39 (13) \text{ \AA}^3$

$Z = 4$

$F(000) = 536$

$D_x = 1.369 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9273 reflections

$\theta = 2.6\text{--}29.0^\circ$

$\mu = 0.25 \text{ mm}^{-1}$

$T = 150 \text{ K}$

Block, colourless

$0.31 \times 0.19 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEX CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 8.3333 pixels mm⁻¹
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2015)
 $T_{\min} = 0.88$, $T_{\max} = 0.96$

22588 measured reflections
 3318 independent reflections
 2731 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$
 $\theta_{\max} = 29.1^\circ$, $\theta_{\min} = 1.9^\circ$
 $h = -14 \rightarrow 14$
 $k = -7 \rightarrow 7$
 $l = -28 \rightarrow 28$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.107$
 $S = 1.06$
 3318 reflections
 163 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 constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.0734P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω , collected at $\varphi = 0.00$, 90.00 and 180.00° and 2 sets of 800 frames, each of width 0.45° in φ , collected at $\omega = -30.00$ and 210.00° . The scan time was 20 sec/frame.

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. H-atoms attached to carbon were placed in calculated positions (C—H = $0.95 - 0.99 \text{ \AA}$). 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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	1.01122 (3)	0.51867 (6)	0.64271 (2)	0.02520 (11)
O1	0.82014 (8)	0.06255 (17)	0.72781 (4)	0.0263 (2)
N1	0.77044 (9)	0.22455 (18)	0.63087 (4)	0.0188 (2)
C1	0.78669 (10)	0.4044 (2)	0.58307 (5)	0.0183 (2)
C2	0.69629 (11)	0.4345 (2)	0.53375 (5)	0.0228 (2)
H2	0.6216	0.3430	0.5342	0.027*
C3	0.71505 (12)	0.5966 (2)	0.48439 (5)	0.0266 (3)
H3	0.6537	0.6136	0.4510	0.032*
C4	0.82274 (12)	0.7341 (2)	0.48348 (5)	0.0283 (3)
H4	0.8359	0.8435	0.4493	0.034*
C5	0.91096 (12)	0.7109 (3)	0.53267 (5)	0.0273 (3)

H5	0.9839	0.8080	0.5325	0.033*
C6	0.89443 (10)	0.5470 (2)	0.58242 (5)	0.0204 (2)
C7	0.90903 (11)	0.4571 (2)	0.70494 (5)	0.0202 (2)
H7A	0.9579	0.4299	0.7451	0.024*
H7B	0.8549	0.6026	0.7104	0.024*
C8	0.83135 (10)	0.2310 (2)	0.68959 (5)	0.0191 (2)
C9	0.68427 (11)	0.0192 (2)	0.61862 (6)	0.0206 (2)
H9A	0.7166	−0.1296	0.6415	0.025*
H9B	0.6814	−0.0193	0.5729	0.025*
C10	0.55414 (10)	0.0666 (2)	0.63773 (5)	0.0185 (2)
C11	0.52211 (11)	0.2662 (2)	0.67490 (5)	0.0239 (3)
H11	0.5828	0.3854	0.6879	0.029*
C12	0.40231 (12)	0.2935 (3)	0.69330 (5)	0.0290 (3)
H12	0.3813	0.4312	0.7186	0.035*
C13	0.31340 (12)	0.1201 (3)	0.67478 (6)	0.0302 (3)
H13	0.2314	0.1387	0.6874	0.036*
C14	0.34421 (12)	−0.0800 (3)	0.63797 (7)	0.0319 (3)
H14	0.2835	−0.1998	0.6255	0.038*
C15	0.46353 (11)	−0.1061 (2)	0.61924 (6)	0.0261 (3)
H15	0.4839	−0.2430	0.5935	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01580 (17)	0.0344 (2)	0.02528 (17)	−0.00305 (11)	−0.00034 (12)	0.00452 (11)
O1	0.0243 (4)	0.0259 (5)	0.0282 (4)	−0.0015 (4)	−0.0014 (3)	0.0087 (3)
N1	0.0177 (4)	0.0169 (5)	0.0215 (4)	−0.0014 (4)	−0.0006 (3)	−0.0006 (3)
C1	0.0192 (5)	0.0181 (5)	0.0178 (5)	0.0024 (4)	0.0019 (4)	−0.0025 (4)
C2	0.0227 (6)	0.0244 (6)	0.0210 (5)	−0.0001 (5)	−0.0022 (4)	−0.0040 (4)
C3	0.0296 (6)	0.0309 (7)	0.0189 (5)	0.0040 (5)	−0.0033 (5)	−0.0025 (5)
C4	0.0343 (7)	0.0302 (7)	0.0209 (5)	0.0020 (5)	0.0051 (5)	0.0054 (5)
C5	0.0243 (6)	0.0318 (7)	0.0263 (6)	−0.0026 (5)	0.0060 (5)	0.0039 (5)
C6	0.0171 (5)	0.0245 (6)	0.0198 (5)	0.0022 (4)	0.0019 (4)	−0.0011 (4)
C7	0.0200 (5)	0.0219 (6)	0.0186 (5)	−0.0010 (4)	−0.0006 (4)	0.0009 (4)
C8	0.0157 (5)	0.0195 (6)	0.0220 (5)	0.0034 (4)	0.0011 (4)	0.0006 (4)
C9	0.0195 (6)	0.0145 (5)	0.0276 (6)	−0.0002 (4)	0.0003 (4)	−0.0032 (4)
C10	0.0187 (5)	0.0167 (5)	0.0200 (5)	0.0008 (4)	−0.0010 (4)	0.0021 (4)
C11	0.0239 (6)	0.0234 (6)	0.0242 (5)	0.0017 (5)	−0.0014 (4)	−0.0022 (4)
C12	0.0302 (7)	0.0345 (7)	0.0228 (5)	0.0097 (6)	0.0050 (5)	−0.0008 (5)
C13	0.0218 (6)	0.0410 (8)	0.0283 (6)	0.0048 (5)	0.0055 (5)	0.0115 (6)
C14	0.0218 (6)	0.0306 (7)	0.0430 (7)	−0.0051 (5)	−0.0014 (5)	0.0061 (6)
C15	0.0236 (6)	0.0196 (6)	0.0349 (6)	−0.0012 (5)	−0.0004 (5)	−0.0023 (5)

Geometric parameters (Å, °)

S1—C6	1.7583 (11)	C7—H7A	0.9900
S1—C7	1.8013 (12)	C7—H7B	0.9900
O1—C8	1.2262 (14)	C9—C10	1.5144 (16)

N1—C8	1.3781 (13)	C9—H9A	0.9900
N1—C1	1.4191 (14)	C9—H9B	0.9900
N1—C9	1.4628 (14)	C10—C11	1.3880 (16)
C1—C6	1.4012 (16)	C10—C15	1.3947 (16)
C1—C2	1.4047 (15)	C11—C12	1.3876 (17)
C2—C3	1.3860 (18)	C11—H11	0.9500
C2—H2	0.9500	C12—C13	1.3848 (19)
C3—C4	1.3859 (19)	C12—H12	0.9500
C3—H3	0.9500	C13—C14	1.382 (2)
C4—C5	1.3833 (16)	C13—H13	0.9500
C4—H4	0.9500	C14—C15	1.3843 (18)
C5—C6	1.3940 (17)	C14—H14	0.9500
C5—H5	0.9500	C15—H15	0.9500
C7—C8	1.5066 (16)		
C6—S1—C7	95.66 (5)	O1—C8—N1	121.21 (10)
C8—N1—C1	123.74 (9)	O1—C8—C7	121.93 (10)
C8—N1—C9	116.79 (9)	N1—C8—C7	116.84 (9)
C1—N1—C9	119.46 (9)	N1—C9—C10	115.07 (9)
C6—C1—C2	118.72 (10)	N1—C9—H9A	108.5
C6—C1—N1	121.15 (9)	C10—C9—H9A	108.5
C2—C1—N1	120.07 (10)	N1—C9—H9B	108.5
C3—C2—C1	120.60 (11)	C10—C9—H9B	108.5
C3—C2—H2	119.7	H9A—C9—H9B	107.5
C1—C2—H2	119.7	C11—C10—C15	118.70 (11)
C4—C3—C2	120.38 (11)	C11—C10—C9	123.32 (10)
C4—C3—H3	119.8	C15—C10—C9	117.92 (10)
C2—C3—H3	119.8	C12—C11—C10	120.65 (12)
C5—C4—C3	119.52 (11)	C12—C11—H11	119.7
C5—C4—H4	120.2	C10—C11—H11	119.7
C3—C4—H4	120.2	C13—C12—C11	120.02 (12)
C4—C5—C6	121.00 (12)	C13—C12—H12	120.0
C4—C5—H5	119.5	C11—C12—H12	120.0
C6—C5—H5	119.5	C14—C13—C12	119.89 (12)
C5—C6—C1	119.76 (10)	C14—C13—H13	120.1
C5—C6—S1	119.15 (9)	C12—C13—H13	120.1
C1—C6—S1	121.08 (9)	C13—C14—C15	120.04 (12)
C8—C7—S1	110.56 (8)	C13—C14—H14	120.0
C8—C7—H7A	109.5	C15—C14—H14	120.0
S1—C7—H7A	109.5	C14—C15—C10	120.69 (12)
C8—C7—H7B	109.5	C14—C15—H15	119.7
S1—C7—H7B	109.5	C10—C15—H15	119.7
H7A—C7—H7B	108.1		
C8—N1—C1—C6	23.43 (16)	C1—N1—C8—O1	−175.23 (10)
C9—N1—C1—C6	−156.89 (11)	C9—N1—C8—O1	5.09 (15)
C8—N1—C1—C2	−159.32 (11)	C1—N1—C8—C7	6.40 (15)
C9—N1—C1—C2	20.36 (15)	C9—N1—C8—C7	−173.29 (10)

C6—C1—C2—C3	1.99 (17)	S1—C7—C8—O1	131.04 (10)
N1—C1—C2—C3	−175.32 (11)	S1—C7—C8—N1	−50.60 (12)
C1—C2—C3—C4	−0.93 (19)	C8—N1—C9—C10	88.08 (12)
C2—C3—C4—C5	−0.81 (19)	C1—N1—C9—C10	−91.62 (12)
C3—C4—C5—C6	1.48 (19)	N1—C9—C10—C11	−12.01 (16)
C4—C5—C6—C1	−0.40 (19)	N1—C9—C10—C15	170.98 (10)
C4—C5—C6—S1	178.35 (10)	C15—C10—C11—C12	0.00 (17)
C2—C1—C6—C5	−1.33 (17)	C9—C10—C11—C12	−176.99 (11)
N1—C1—C6—C5	175.96 (11)	C10—C11—C12—C13	0.24 (18)
C2—C1—C6—S1	179.95 (9)	C11—C12—C13—C14	0.01 (18)
N1—C1—C6—S1	−2.77 (15)	C12—C13—C14—C15	−0.51 (19)
C7—S1—C6—C5	147.46 (11)	C13—C14—C15—C10	0.75 (19)
C7—S1—C6—C1	−33.80 (10)	C11—C10—C15—C14	−0.49 (18)
C6—S1—C7—C8	57.87 (9)	C9—C10—C15—C14	176.66 (11)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C7—H7 <i>A</i> $\cdots$ O1 ⁱ	0.99	2.55	3.2504 (14)	128
C7—H7 <i>B</i> $\cdots$ O1 ⁱⁱ	0.99	2.53	3.4403 (15)	152

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