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

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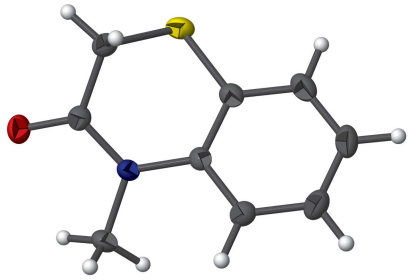
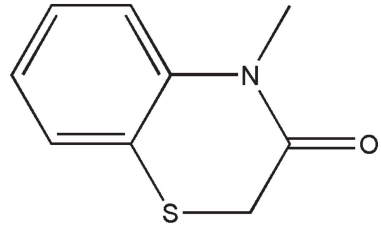
Structural data: full structural data are available from iucrdata.iucr.org

In the crystal of the title compound, C_9H_9NOS, the molecules are linked by C—H···O hydrogen bonds to generate bilayers lying parallel to (001).

3D view



Chemical scheme



Structure description

As a continuation of our studies of *N*-substituted benzothiazines (Sebbar *et al.*, 2014, 2016; Ellouz *et al.*, 2015), we now describe the synthesis and structure of the title compound, (Fig. 1).

A puckering analysis of the heterocyclic ring gave the parameters Q = 0.668 (1) Å, θ = 113.2 (1)° and φ = 146.9°: atoms C1, C6, S1 and N1 are roughly coplanar (r.m.s. deviation = 0.046 Å) and atoms C7 and C8 deviate in the same sense [by 0.476 (1) and 1.111 (1) Å, respectively] from the mean plane. In the crystal, the molecules are linked by C—H···O hydrogen bonds (Table 1) to form bilayers oriented parallel to (001) (Figs. 2 and 3).

Synthesis and crystallization

To a solution of 3,4-dihydro-2*H*-1,4-benzothiazin-3-one (2 mmol), potassium carbonate (4 mmol) and tetra *n*-butyl ammonium bromide (0.2 mmol) in DMF (15 ml) was added iodomethane (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 of the title compound were isolated when the solvent was allowed to evaporate (yield 57%; m.p. 370 K).

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O1^{i}	0.983 (18)	2.301 (18)	3.2819 (16)	175.0 (15)
C9—H9B···O1^{ii}	0.998 (19)	2.62 (2)	3.6157 (17)	173.3 (15)
C9—H9C···O1^{iii}	0.995 (17)	2.512 (17)	3.4265 (16)	152.6 (14)

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

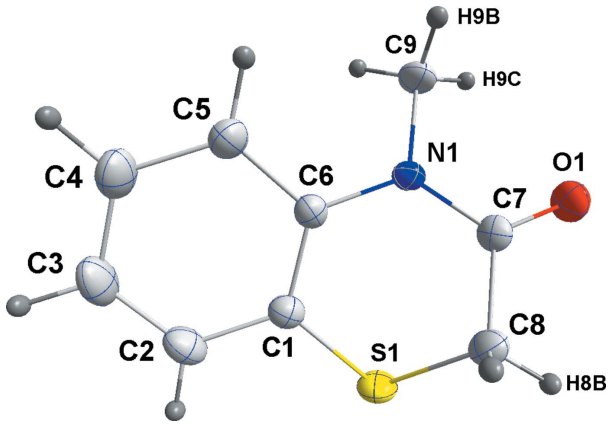


Figure 1
The title molecule with 50% probability ellipsoids.

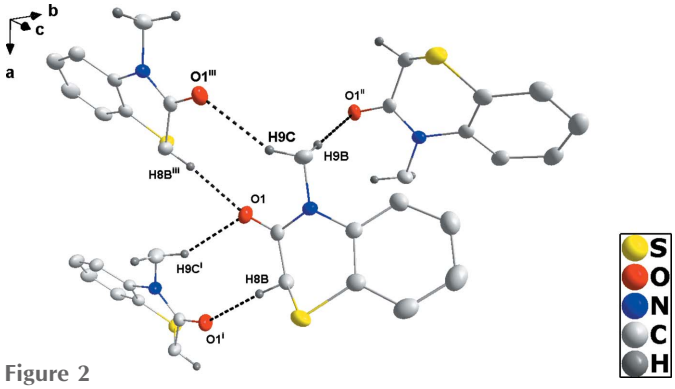


Figure 2
Detail of the intermolecular C—H···O hydrogen bonding [symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, z$; (iii) $-\frac{1}{2}+x, \frac{1}{2}-y, 1-z$].

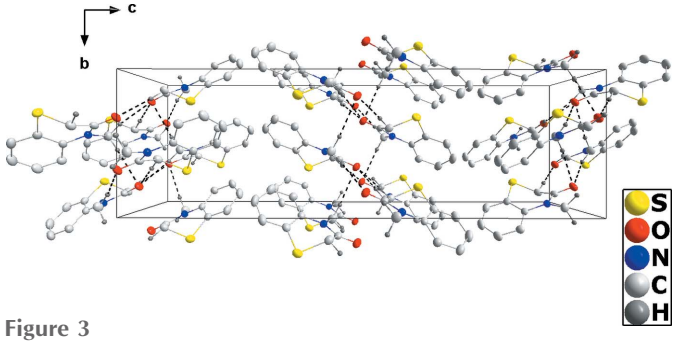


Figure 3
Packing viewed along the *a* axis with C—H···O hydrogen bonds shown as dotted lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	C_9H_9NOS
M_r	179.23
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	150
a, *b*, *c* (Å)	7.3148 (6), 8.4030 (6), 27.670 (2)
V (Å^3)	1700.8 (2)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.95
Crystal size (mm)	0.33 × 0.31 × 0.04
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.63, 0.88
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16139, 1674, 1628
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.076, 1.04
No. of reflections	1674
No. of parameters	146
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.24

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Refinement

Crystal and refinement data are presented in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170097 [https://doi.org/10.1107/S2414314617000979]

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Crystal data

C_9H_9NOS
M_r = 179.23
Orthorhombic, *Pbca*
a = 7.3148 (6) Å
b = 8.4030 (6) Å
c = 27.670 (2) Å
V = 1700.8 (2) Å^3
Z = 8
F(000) = 752
D_x = 1.400 Mg m^{-3}
Cu *Kα* radiation, λ = 1.54178 Å
Cell parameters from 9930 reflections
θ = 3.2–72.4°
μ = 2.95 mm^{-1}
T = 150 K
Plate, colourless
0.33 × 0.31 × 0.04 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC I*μ*S micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.63, T_{max} = 0.88
16139 measured reflections
1674 independent reflections
1628 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 72.2°, θ_{min} = 3.2°
h = −8→9
k = −10→10
l = −34→30

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.076
S = 1.04
1674 reflections
146 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.7672P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0029 (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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.70736 (4)	0.30751 (4)	0.62414 (2)	0.02827 (15)
O1	0.44820 (14)	0.32356 (11)	0.50834 (3)	0.0293 (2)
N1	0.36431 (14)	0.44168 (12)	0.57877 (3)	0.0207 (2)
C1	0.55962 (17)	0.44702 (14)	0.65097 (4)	0.0227 (3)
C2	0.59106 (19)	0.50090 (17)	0.69783 (5)	0.0302 (3)
H2	0.693 (2)	0.458 (2)	0.7141 (6)	0.039 (5)*
C3	0.4778 (2)	0.61424 (18)	0.71846 (5)	0.0347 (3)
H3	0.499 (3)	0.652 (2)	0.7512 (7)	0.046 (5)*
C4	0.3350 (2)	0.67709 (18)	0.69178 (5)	0.0320 (3)
H4	0.261 (3)	0.759 (2)	0.7048 (6)	0.047 (5)*
C5	0.29965 (17)	0.62271 (16)	0.64548 (5)	0.0257 (3)
H5	0.195 (2)	0.666 (2)	0.6276 (5)	0.030 (4)*
C6	0.40958 (16)	0.50524 (14)	0.62472 (4)	0.0200 (3)
C7	0.49111 (17)	0.38099 (14)	0.54745 (4)	0.0220 (3)
C8	0.68710 (17)	0.38743 (16)	0.56376 (5)	0.0254 (3)
H8A	0.731 (2)	0.492 (2)	0.5634 (6)	0.033 (4)*
H8B	0.759 (3)	0.319 (2)	0.5421 (6)	0.034 (4)*
C9	0.17297 (18)	0.44689 (17)	0.56278 (5)	0.0280 (3)
H9A	0.095 (2)	0.435 (2)	0.5922 (6)	0.034 (4)*
H9B	0.148 (3)	0.549 (2)	0.5456 (6)	0.043 (5)*
H9C	0.152 (2)	0.359 (2)	0.5394 (6)	0.033 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0234 (2)	0.0272 (2)	0.0342 (2)	0.00595 (11)	−0.00356 (11)	0.00232 (12)
O1	0.0317 (5)	0.0301 (5)	0.0261 (5)	−0.0038 (4)	−0.0010 (4)	−0.0083 (4)
N1	0.0175 (5)	0.0229 (5)	0.0217 (5)	0.0001 (4)	−0.0023 (4)	−0.0015 (4)
C1	0.0213 (6)	0.0234 (6)	0.0233 (6)	−0.0027 (5)	−0.0004 (4)	0.0032 (5)
C2	0.0280 (7)	0.0383 (7)	0.0242 (6)	−0.0054 (6)	−0.0050 (5)	0.0052 (5)
C3	0.0367 (8)	0.0469 (8)	0.0206 (6)	−0.0080 (6)	0.0020 (5)	−0.0050 (6)
C4	0.0322 (7)	0.0364 (7)	0.0275 (7)	−0.0012 (6)	0.0074 (6)	−0.0069 (5)
C5	0.0239 (6)	0.0270 (6)	0.0262 (6)	0.0010 (5)	0.0023 (5)	−0.0006 (5)
C6	0.0194 (6)	0.0214 (6)	0.0192 (6)	−0.0030 (4)	0.0008 (4)	0.0011 (4)
C7	0.0242 (6)	0.0189 (5)	0.0229 (6)	−0.0016 (5)	0.0007 (5)	−0.0012 (4)
C8	0.0209 (6)	0.0269 (6)	0.0283 (6)	0.0000 (5)	0.0018 (5)	−0.0044 (5)
C9	0.0202 (6)	0.0345 (7)	0.0294 (7)	0.0014 (5)	−0.0059 (5)	−0.0028 (6)

Geometric parameters (Å, º)

S1—C1	1.7589 (13)	C3—H3	0.97 (2)
S1—C8	1.8067 (14)	C4—C5	1.3846 (19)
O1—C7	1.2257 (15)	C4—H4	0.94 (2)
N1—C7	1.3680 (16)	C5—C6	1.3967 (17)
N1—C6	1.4183 (15)	C5—H5	0.979 (17)
N1—C9	1.4684 (16)	C7—C8	1.5040 (18)
C1—C2	1.3924 (18)	C8—H8A	0.937 (19)
C1—C6	1.4042 (17)	C8—H8B	0.983 (18)
C2—C3	1.385 (2)	C9—H9A	0.998 (17)
C2—H2	0.943 (18)	C9—H9B	0.998 (19)
C3—C4	1.384 (2)	C9—H9C	0.995 (17)
C1—S1—C8	95.30 (6)	C5—C6—C1	118.91 (11)
C7—N1—C6	123.36 (10)	C5—C6—N1	120.02 (11)
C7—N1—C9	117.81 (10)	C1—C6—N1	121.00 (11)
C6—N1—C9	118.77 (10)	O1—C7—N1	122.17 (12)
C2—C1—C6	119.84 (12)	O1—C7—C8	121.54 (11)
C2—C1—S1	120.56 (10)	N1—C7—C8	116.29 (10)
C6—C1—S1	119.61 (9)	C7—C8—S1	110.02 (9)
C3—C2—C1	120.56 (13)	C7—C8—H8A	110.9 (10)
C3—C2—H2	122.5 (11)	S1—C8—H8A	109.3 (10)
C1—C2—H2	116.9 (11)	C7—C8—H8B	108.0 (11)
C4—C3—C2	119.59 (13)	S1—C8—H8B	107.6 (10)
C4—C3—H3	119.6 (12)	H8A—C8—H8B	110.9 (14)
C2—C3—H3	120.8 (12)	N1—C9—H9A	107.2 (10)
C3—C4—C5	120.59 (13)	N1—C9—H9B	110.2 (11)
C3—C4—H4	120.4 (12)	H9A—C9—H9B	111.7 (14)
C5—C4—H4	119.0 (12)	N1—C9—H9C	108.8 (10)
C4—C5—C6	120.40 (12)	H9A—C9—H9C	111.4 (14)
C4—C5—H5	119.5 (9)	H9B—C9—H9C	107.6 (13)
C6—C5—H5	120.1 (9)		
C8—S1—C1—C2	−145.51 (11)	S1—C1—C6—N1	6.45 (16)
C8—S1—C1—C6	34.01 (11)	C7—N1—C6—C5	152.11 (12)
C6—C1—C2—C3	−1.42 (19)	C9—N1—C6—C5	−25.19 (16)
S1—C1—C2—C3	178.10 (11)	C7—N1—C6—C1	−30.72 (17)
C1—C2—C3—C4	−1.6 (2)	C9—N1—C6—C1	151.98 (12)
C2—C3—C4—C5	2.8 (2)	C6—N1—C7—O1	178.39 (11)
C3—C4—C5—C6	−1.0 (2)	C9—N1—C7—O1	−4.28 (17)
C4—C5—C6—C1	−1.97 (19)	C6—N1—C7—C8	−1.19 (16)
C4—C5—C6—N1	175.26 (11)	C9—N1—C7—C8	176.13 (11)
C2—C1—C6—C5	3.18 (18)	O1—C7—C8—S1	−129.74 (11)
S1—C1—C6—C5	−176.35 (9)	N1—C7—C8—S1	49.85 (13)
C2—C1—C6—N1	−174.02 (11)	C1—S1—C8—C7	−60.08 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*B*···O1^{i}	0.983 (18)	2.301 (18)	3.2819 (16)	175.0 (15)
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