Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(2-Methylbenzylidene)-2,3-dihydro-1,5-benzothiazepin-4(5*H*)-one

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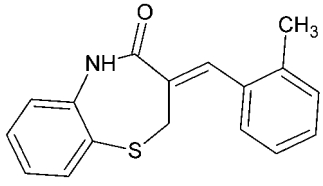
Received 26 November 2010; accepted 16 December 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.202; data-to-parameter ratio = 19.7.

In the crystal structure of the title compound, $C_{17}H_{15}NOS$, the molecules form centrosymmetric dimers through pairs of N—H···O hydrogen bonds. The seven-membered ring adopts a distorted half-chair conformation.

Related literature

Dibenzo[*c*,*e*]thiepine derivatives exhibit chiroptical properties (Tomascovic *et al.*, 2000) and dibenzo[*b*,*e*]thiepin-5,5-dioxide derivatives possess antihistaminic and antiallergenic activities (Rajsner *et al.*, 1971) while benzene thiepine derivatives have been identified as effective antihistaminic compounds (Metys *et al.*, 1965).



Experimental

Crystal data

$C_{17}H_{15}NOS$
M_r = 281.36
Monoclinic, $C2/c$
a = 19.1192 (5) Å
b = 13.0049 (3) Å
c = 14.8903 (4) Å
β = 128.591 (1)°
V = 2893.84 (13) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 293 K
0.22 × 0.18 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
13879 measured reflections
3560 independent reflections
2707 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.202
S = 0.83
3560 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0···O^{i}	0.86	2.01	2.8705 (18)	177

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); 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, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank Professor D. Velmurugan, Centre for Advanced Study in Crystallography and Biophysics, University of Madras, for providing data-collection and computing facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5422).

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supporting information

Acta Cryst. (2011). E**67**, o243 [doi:10.1107/S1600536810052888]

3-(2-Methylbenzylidene)-2,3-dihydro-1,5-benzothiazepin-4(5*H*)-one

D. Sridevi, Sundari Bhaskaran, G. Usha, G. Murugan and M. Bakthadoss

S1. Comment

The title compound is used as an intermediate for the synthesis of dosulepin, which is an antidepressant of the tricyclic family. Dosulepin prevents reabsorbing of serotonin and noradrenaline in the brain, helps to prolong the mood lightening effect of any released noradrenaline and serotonin, thus relieving depression. The dibenzo[c,e]thiepine derivatives exhibit chiroptical properties (Tomascovic *et al.*, 2000). Dibenzo[b,e]thiepin-5,5-dioxide derivatives possess antihistaminic and antiallergenic activities (Rajsner *et al.*, 1971). Benzene thiepine derivatives are identified as a new type of effective antihistaminic compounds (Metys *et al.*, 1965). Considering the wide range of biological activities of the thiepine derivatives, we determined the crystal structure of the title compound. The seven membered thiepin ring adopts a distorted half-chair conformation. The molecules form centrosymmetric dimers through N—H···O hydrogen bonds.

S2. Experimental

A mixture of(*Z*)-methyl 2-(bromomethyl)-3-*o*-tolylacrylate (2 mmol) and *o*-aminothiophenol(2 mmol) in the presence of potassium *tert*-butoxide (2.4 mmol) in dry THF (10 ml) was stirred at room temperature for 1 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated and the resulting crude mass was diluted with water (20 ml) and extracted with ethyl acetate (3 x 20ml). The organic layer was washed with brine (2 x 20ml) and dried over anhydrous sodium sulfate. The organic layer was concentrated, which provided a crude mass (*Z*)-methyl 2-((2-aminophenylthio)methyl)-3-*o*-tolylacrylate. The crude product was treated with a catalytic amount of *p*-toluene sulphonic acid (0.4 mmol), in p-xylene (10 ml), under reflux conditions for 12 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated under reduced pressure and worked up as mentioned previously, which successfully provide the crude final product. The final product was purified by column chromatography on silica gel to afford the title compound in good yield (67%).

S3. Refinement

H atoms were positioned geometrically and were treated as riding on their parent atoms, with C—H distances of 0.93–0.97Å, an N—H distance of 0.86Å and U_{iso}(H)=1.2U_{eq}(N, C).

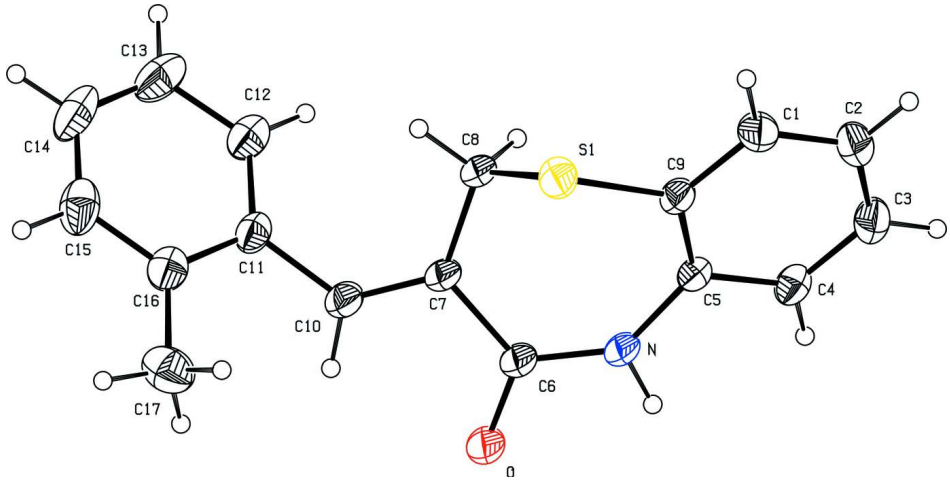

Figure 1

Perspective view of the title compound with 30% probability ellipsoids.

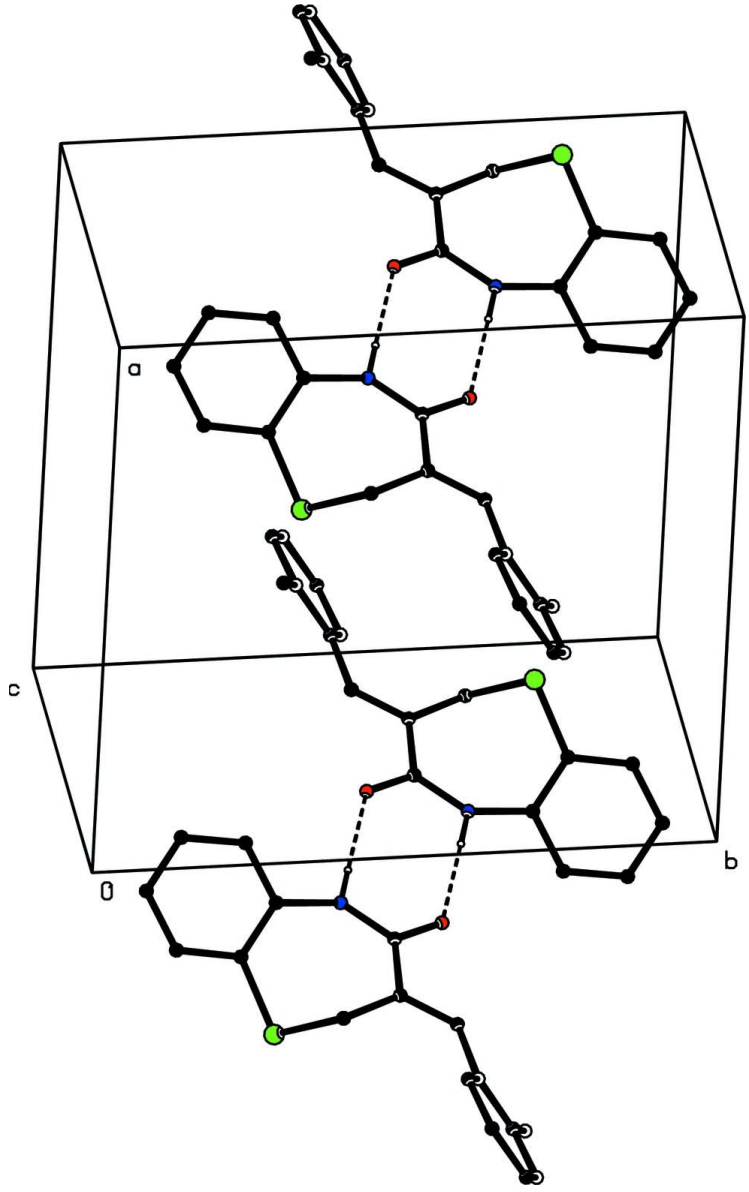

Figure 2

Partial packing diagram, viewed along the c axis.

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Crystal data

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$c = 14.8903\ (4)$ Å
$\beta = 128.591\ (1)°$
$V = 2893.84\ (13)$ Å^3
$Z = 8$
$F(000) = 1184$
$D_x = 1.292$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3560 reflections
$\theta = 2–28°$
$\mu = 0.22$ mm^{-1}
$T = 293$ K
Block, colourless
0.22 × 0.18 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
13879 measured reflections
3560 independent reflections
2707 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.1°$
$h = -25 \rightarrow 20$
$k = -15 \rightarrow 16$
$l = -15 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.202$
$S = 0.83$
3560 reflections
181 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ 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.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.07250 (13)	0.88179 (15)	0.21097 (16)	0.0540 (5)
H1	0.1146	0.9233	0.2730	0.065*
C2	−0.00643 (15)	0.92462 (17)	0.11794 (18)	0.0623 (5)
H2	−0.0169	0.9947	0.1164	0.075*
C3	−0.06956 (12)	0.86298 (15)	0.02748 (16)	0.0557 (5)
H3	−0.1233	0.8912	−0.0351	0.067*
C4	−0.05362 (11)	0.75966 (14)	0.02896 (14)	0.0448 (4)
H4	−0.0970	0.7185	−0.0326	0.054*
C5	0.02685 (10)	0.71567 (12)	0.12171 (12)	0.0365 (4)
C6	0.08537 (12)	0.52859 (14)	0.17690 (13)	0.0485 (4)
C7	0.14280 (11)	0.52918 (13)	0.30555 (12)	0.0424 (4)
C8	0.15581 (12)	0.62540 (13)	0.36935 (13)	0.0478 (4)
H8A	0.1988	0.6123	0.4512	0.057*
H8B	0.0995	0.6441	0.3523	0.057*
C9	0.09042 (10)	0.77806 (13)	0.21391 (13)	0.0398 (4)
C10	0.18328 (12)	0.44083 (13)	0.35720 (14)	0.0477 (4)
H10	0.1742	0.3876	0.3092	0.057*

C11	0.24053 (11)	0.41648 (13)	0.48045 (14)	0.0449 (4)
C12	0.21413 (14)	0.43985 (16)	0.54648 (16)	0.0573 (5)
H12	0.1612	0.4759	0.5135	0.069*
C13	0.26536 (17)	0.4103 (2)	0.66057 (18)	0.0723 (7)
H13	0.2472	0.4269	0.7040	0.087*
C14	0.34253 (16)	0.35663 (19)	0.70886 (18)	0.0750 (7)
H14	0.3770	0.3360	0.7853	0.090*
C15	0.36932 (13)	0.33310 (17)	0.64501 (18)	0.0676 (6)
H15	0.4222	0.2966	0.6792	0.081*
C16	0.31970 (11)	0.36225 (14)	0.52991 (15)	0.0504 (4)
C17	0.35129 (16)	0.33506 (19)	0.4639 (2)	0.0722 (6)
H17A	0.4068	0.2983	0.5126	0.108*
H17B	0.3601	0.3967	0.4366	0.108*
H17C	0.3074	0.2926	0.3997	0.108*
N	0.03793 (9)	0.61072 (12)	0.10949 (10)	0.0450 (4)
H0	0.0047	0.5939	0.0381	0.054*
O	0.07844 (12)	0.44836 (11)	0.12814 (11)	0.0766 (5)
S1	0.19459 (3)	0.73150 (4)	0.33323 (3)	0.0503 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0582 (10)	0.0419 (10)	0.0457 (9)	−0.0007 (8)	0.0246 (8)	−0.0069 (7)
C2	0.0737 (12)	0.0426 (10)	0.0558 (11)	0.0134 (8)	0.0332 (10)	0.0055 (8)
C3	0.0507 (9)	0.0560 (12)	0.0456 (10)	0.0109 (8)	0.0228 (8)	0.0110 (8)
C4	0.0400 (8)	0.0503 (10)	0.0332 (8)	−0.0013 (6)	0.0174 (7)	0.0027 (6)
C5	0.0373 (7)	0.0405 (8)	0.0284 (7)	−0.0002 (6)	0.0190 (6)	0.0005 (6)
C6	0.0570 (9)	0.0436 (10)	0.0281 (7)	0.0032 (7)	0.0183 (7)	−0.0047 (6)
C7	0.0501 (8)	0.0414 (9)	0.0262 (7)	0.0001 (7)	0.0192 (6)	−0.0041 (6)
C8	0.0600 (9)	0.0448 (10)	0.0258 (7)	0.0080 (7)	0.0205 (7)	−0.0024 (6)
C9	0.0391 (7)	0.0424 (9)	0.0305 (7)	0.0003 (6)	0.0180 (6)	0.0004 (6)
C10	0.0579 (9)	0.0426 (10)	0.0323 (8)	−0.0020 (7)	0.0231 (8)	−0.0030 (7)
C11	0.0497 (8)	0.0391 (9)	0.0345 (8)	−0.0061 (6)	0.0207 (7)	0.0027 (6)
C12	0.0610 (10)	0.0628 (13)	0.0444 (10)	−0.0029 (9)	0.0310 (9)	0.0050 (8)
C13	0.0867 (16)	0.0852 (17)	0.0483 (11)	−0.0180 (12)	0.0438 (12)	0.0024 (11)
C14	0.0811 (14)	0.0757 (16)	0.0366 (9)	−0.0188 (12)	0.0211 (10)	0.0107 (9)
C15	0.0500 (9)	0.0583 (13)	0.0547 (12)	−0.0067 (8)	0.0131 (9)	0.0079 (9)
C16	0.0489 (8)	0.0429 (10)	0.0442 (9)	−0.0116 (7)	0.0217 (8)	−0.0030 (7)
C17	0.0664 (12)	0.0654 (14)	0.0809 (16)	−0.0040 (10)	0.0441 (12)	−0.0149 (11)
N	0.0499 (7)	0.0433 (8)	0.0229 (6)	0.0019 (6)	0.0135 (6)	−0.0046 (5)
O	0.1042 (12)	0.0492 (9)	0.0302 (6)	0.0191 (8)	0.0193 (7)	−0.0072 (5)
S1	0.0394 (3)	0.0464 (3)	0.0371 (3)	−0.00100 (15)	0.0101 (2)	−0.00664 (16)

Geometric parameters (Å, º)

C1—C2	1.378 (3)	C9—S1	1.7538 (16)
C1—C9	1.386 (2)	C10—C11	1.470 (2)
C1—H1	0.9300	C10—H10	0.9300

C2—C3	1.373 (3)	C11—C16	1.393 (3)
C2—H2	0.9300	C11—C12	1.390 (3)
C3—C4	1.375 (3)	C12—C13	1.385 (3)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.397 (2)	C13—C14	1.363 (3)
C4—H4	0.9300	C13—H13	0.9300
C5—C9	1.392 (2)	C14—C15	1.367 (4)
C5—N	1.410 (2)	C14—H14	0.9300
C6—O	1.230 (2)	C15—C16	1.397 (3)
C6—N	1.355 (2)	C15—H15	0.9300
C6—C7	1.501 (2)	C16—C17	1.482 (3)
C7—C10	1.330 (2)	C17—H17A	0.9600
C7—C8	1.495 (2)	C17—H17B	0.9600
C8—S1	1.8001 (19)	C17—H17C	0.9600
C8—H8A	0.9700	N—H0	0.8600
C8—H8B	0.9700		

C2—C1—C9	121.29 (17)	C7—C10—H10	115.8
C2—C1—H1	119.4	C11—C10—H10	115.8
C9—C1—H1	119.4	C16—C11—C12	119.40 (16)
C3—C2—C1	119.44 (19)	C16—C11—C10	119.11 (17)
C3—C2—H2	120.3	C12—C11—C10	121.33 (17)
C1—C2—H2	120.3	C13—C12—C11	121.1 (2)
C2—C3—C4	120.29 (17)	C13—C12—H12	119.5
C2—C3—H3	119.9	C11—C12—H12	119.5
C4—C3—H3	119.9	C14—C13—C12	119.6 (2)
C3—C4—C5	120.83 (16)	C14—C13—H13	120.2
C3—C4—H4	119.6	C12—C13—H13	120.2
C5—C4—H4	119.6	C15—C14—C13	119.99 (19)
C9—C5—C4	118.81 (15)	C15—C14—H14	120.0
C9—C5—N	125.61 (14)	C13—C14—H14	120.0
C4—C5—N	115.48 (14)	C14—C15—C16	122.0 (2)
O—C6—N	117.10 (14)	C14—C15—H15	119.0
O—C6—C7	118.78 (15)	C16—C15—H15	119.0
N—C6—C7	124.09 (14)	C11—C16—C15	117.93 (19)
C10—C7—C8	123.34 (14)	C11—C16—C17	121.88 (18)
C10—C7—C6	115.63 (14)	C15—C16—C17	120.2 (2)
C8—C7—C6	120.97 (14)	C16—C17—H17A	109.5
C7—C8—S1	112.83 (12)	C16—C17—H17B	109.5
C7—C8—H8A	109.0	H17A—C17—H17B	109.5
S1—C8—H8A	109.0	C16—C17—H17C	109.5
C7—C8—H8B	109.0	H17A—C17—H17C	109.5
S1—C8—H8B	109.0	H17B—C17—H17C	109.5
H8A—C8—H8B	107.8	C6—N—C5	138.81 (13)
C1—C9—C5	119.31 (15)	C6—N—H0	110.6
C1—C9—S1	118.05 (13)	C5—N—H0	110.6
C5—C9—S1	122.61 (13)	C9—S1—C8	98.53 (8)
C7—C10—C11	128.30 (16)		

C9—C1—C2—C3	1.6 (3)	C7—C10—C11—C12	49.1 (3)
C1—C2—C3—C4	−0.9 (3)	C16—C11—C12—C13	0.1 (3)
C2—C3—C4—C5	−0.3 (3)	C10—C11—C12—C13	175.39 (19)
C3—C4—C5—C9	1.0 (2)	C11—C12—C13—C14	−0.6 (3)
C3—C4—C5—N	−175.59 (16)	C12—C13—C14—C15	0.5 (4)
O—C6—C7—C10	0.9 (3)	C13—C14—C15—C16	−0.1 (3)
N—C6—C7—C10	178.94 (17)	C12—C11—C16—C15	0.3 (3)
O—C6—C7—C8	178.10 (19)	C10—C11—C16—C15	−175.08 (17)
N—C6—C7—C8	−3.8 (3)	C12—C11—C16—C17	179.97 (18)
C10—C7—C8—S1	123.26 (16)	C10—C11—C16—C17	4.6 (3)
C6—C7—C8—S1	−53.7 (2)	C14—C15—C16—C11	−0.3 (3)
C2—C1—C9—C5	−0.9 (3)	C14—C15—C16—C17	−180.0 (2)
C2—C1—C9—S1	177.38 (15)	O—C6—N—C5	−175.66 (19)
C4—C5—C9—C1	−0.4 (2)	C7—C6—N—C5	6.3 (3)
N—C5—C9—C1	175.84 (16)	C9—C5—N—C6	26.7 (3)
C4—C5—C9—S1	−178.59 (12)	C4—C5—N—C6	−157.0 (2)
N—C5—C9—S1	−2.4 (2)	C1—C9—S1—C8	128.73 (15)
C8—C7—C10—C11	5.3 (3)	C5—C9—S1—C8	−53.03 (15)
C6—C7—C10—C11	−177.56 (18)	C7—C8—S1—C9	87.11 (13)
C7—C10—C11—C16	−135.7 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0···O^{i}	0.86	2.01	2.8705 (18)	177

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