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Ethyl 3-methyl-1-oxo-4*H*-1,4-benzothiazine-2-carboxylate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.082
Data-to-parameter ratio = 15.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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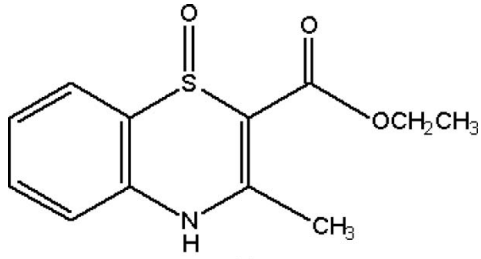
The crystal structure of the title compound, $C_{12}H_{13}NO_3S$, is stabilized by intermolecular N—H···O hydrogen bonds, which are formed between the NH groups and the sulfoxide O atoms.

Comment

1,4-Benzothiazine and its derivatives possess excellent biological and pharmacological activities, such as anti-inflammatory (Krapcho & Turk, 1973), antimicrobial (Sastry *et al.*, 1990), antifungal (Chaffman & Brogden, 1985), calcium antagonist (Aotsuka *et al.*, 1994) and antihypertensive (Kando & Hashimoto, 1993; Keita *et al.*, 2000). The reaction of 2,2′-dithiodianiline with ethyl acetoacetat gave the title compound, (I), besides other products.



(I)

The molecular structure of (I) is shown in Fig. 1, and selected geometric parameters are given in Table 1. The bond lengths and angles are normal when compared with similar structures in the Cambridge Structural Database (Version 5.26; Allen, 2002). The puckering parameters are Q_T = 0.290 (2) Å, θ = 113.7 (4)° and φ = 166.6 (5)° (Cremer & Pople, 1975). The S1═O1 double bond [1.5083 (19) Å] makes angles of 79.36 (9) and 78.60 (11)° with the least-squares planes of the S1/C6/C1/N1/C8/C7 and C1–C6 rings, respectively.

The crystal structure of (I) is stabilized by intermolecular N—H···O hydrogen bonds that form between the NH groups and the sulfoxide O atoms of symmetry-related molecules (Fig. 2 and Table 2).

Experimental

A mixture of dithiodianiline (5 g, 0.02 mol) and ethyl acetoacetate (5.3 g, 0.04 mol) was refluxed with stirring for 3 h. Absolute ethanol (10 ml) was added and stirring was continued at reflux for 1 h. On cooling, (I) (0.5 g, 10% yield) was collected by filtration (m.p. 447–449 K). Recrystallization from ethanol gave yellow crystals. IR (KBr, cm^{-1}): ν 2983–2955, 2900, 1696; ^{1}H NMR (300 MHz, $CDCl_3$, p.p.m.): δ 1.38 (*t*, *J* = 7 Hz, 3H, CH_3), 1.98 (*s*, 3H, CH_3), 4.34 (*m*, 2H, CH_2), 7.04–7.86 (*m*, Harm), 11.25 (*s*, 1H, NH); ^{13}C NMR (75 MHz, $CDCl_3$,

p.p.m.): δ 14.6, 21.0, 61.1, 118.7, 123.4, 125.3, 130.0, 132.1, 134.2, 153.4, 165.5.

Crystal data

$C_{12}H_{13}NO_3S$
M_r = 251.30
Orthorhombic, *Fdd*2
a = 12.9935 (13) Å
b = 33.042 (4) Å
c = 11.1555 (13) Å
V = 4789.4 (9) Å^3
Z = 16
D_x = 1.394 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 9568 reflections
θ = 2.5–27.3°
μ = 0.27 mm^{-1}
T = 296 K
Prism, pale yellow
0.58 × 0.32 × 0.10 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.861, T_{max} = 0.974
6798 measured reflections
2369 independent reflections
2037 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.2°
h = −16 → 16
k = −41 → 40
l = −12 → 14

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.082
S = 1.00
2369 reflections
157 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0541P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0009 (2)
Absolute structure: Flack (1983), 977 Friedel pairs
Flack parameter: 0.001 (3)

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.5083 (19)	O3—C10	1.343 (3)
S1—C6	1.760 (2)	O3—C11	1.444 (3)
S1—C7	1.750 (2)	N1—C1	1.384 (3)
O2—C10	1.208 (3)	N1—C8	1.348 (3)
O1—S1—C6	105.70 (10)	S1—C7—C8	123.31 (17)
O1—S1—C7	107.63 (10)	S1—C7—C10	114.57 (16)
C6—S1—C7	98.03 (10)	N1—C8—C7	121.85 (19)
C10—O3—C11	117.42 (19)	N1—C8—C9	113.71 (19)
C1—N1—C8	125.23 (19)	O2—C10—O3	122.6 (2)
N1—C1—C6	121.43 (19)	O2—C10—C7	126.3 (2)
N1—C1—C2	119.2 (2)	O3—C10—C7	111.06 (19)
S1—C6—C5	116.28 (16)	O3—C11—C12	111.4 (3)
S1—C6—C1	123.65 (17)		
O1—S1—C6—C5	85.45 (18)	C10—C7—C8—C9	9.1 (3)
O1—S1—C7—C8	82.2 (2)	S1—C7—C8—N1	17.5 (3)
O1—S1—C7—C10	−89.56 (17)	S1—C7—C8—C9	−162.10 (18)
O1—S1—C6—C1	−89.75 (19)	C8—C7—C10—O2	13.1 (4)
S1—C7—C10—O2	−175.0 (2)	C8—C7—C10—O3	−167.3 (2)
C10—C7—C8—N1	−171.3 (2)	S1—C7—C10—O3	4.6 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	1.96	2.821 (3)	175

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

All H atoms were included in calculated positions and refined using a riding model, with N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N), and with C—H = 0.93, 0.96 and 0.97 Å, and U_{iso}(H) = 1.5U_{eq}(C) for methyl groups and 1.2U_{eq}(C) for other C atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

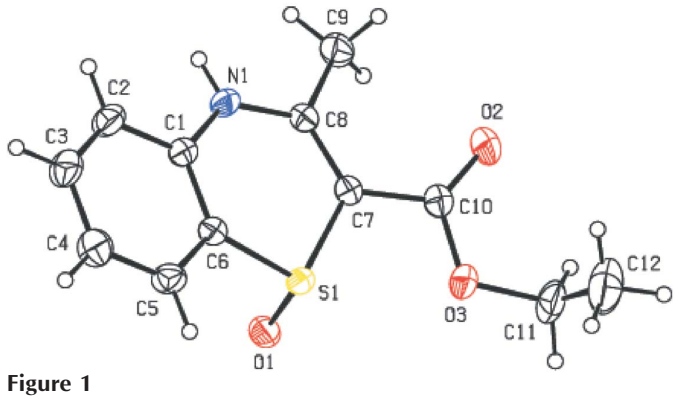


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids.

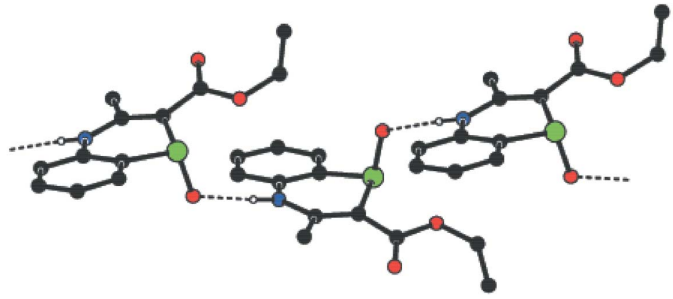


Figure 2
A view of the hydrogen bonding (dashed lines) in compound (I). H atoms not involved in hydrogen bonding have been omitted.

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