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Methyl 2-oxo-2,3-dihydro-1,3-benzothiazole-3-acetate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.109
Data-to-parameter ratio = 18.0

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

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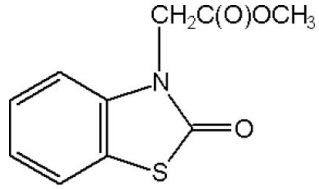
The title compound, $C_{10}H_9NO_3S$, crystallizes with two molecules in the asymmetric unit. The crystal packing is stabilized by van der Waals forces.

Comment

A number of benzothiazolone derivatives have been shown to exhibit biological activity as anticonvulsants (Ucor *et al.*, 1998), psychotropics (Taverne *et al.*, 1998), ligands to the serotoninergic 5-HT1A receptors (Diouf *et al.*, 1995) and plant growth regulators (Loos *et al.*, 1999). The title compound, (I), forms when 2-benzoyl-3-oxo-1,4-benzothiazine undergoes a ring transformation after treatment of 2-benzoyl-3-oxo-1,4-benzothiazine (Keita *et al.*, 2000) in the presence of tetra-*n*-butylammonium bromide (TBAB) with methyl chloroacetate and potassium carbonate in dimethylformamide.



(I)

The structure of (I) is shown in Fig. 1. All bond lengths and angles (Table 1) are similar to those found for 1-(1,3-benzothiazol-2-yl)propan-2-ol (Akkurt *et al.*, 2005). For the first molecule in the asymmetric unit, the S1/C1–C6/N1/C7/O1/C8 fragment of the benzothiazole moiety is essentially planar, with maximum deviations of −0.031 (2) and 0.028 (2) Å for atoms O1 and N1 respectively. In the second molecule, atoms O4 and N2 are displaced from the S2/C11–C16/N2/C17/O4/C18 plane by 0.035 (4) and −0.042 (3) Å, respectively.

In the crystal structure, there are no classical hydrogen bonds. The structure is stabilized by van der Waals forces.

Experimental

To a stirred solution containing 2-benzoyl-3-oxo-1,4-benzothiazine (1 g, 3.7 mmol), potassium carbonate (1 g, 7.43 mmol) and TBAB (20 mg) in dimethylformamide (30 ml) was added methyl chloroacetate (0.6 g, 5.57 mmol) in one portion. The reaction mixture was stirred for 48 h at room temperature and then filtered, and the solvent was allowed to evaporate. Water (100 ml) was added and the mixture was extracted with dichloromethane (10 ml × 3). The organic layer was dried over Na_2SO_2 and evaporated to dryness in a vacuum to give a viscous liquid product. This was further purified by silica gel column chromatography using dichloromethane and diethyl ether (9:1) as eluant to obtain a solid product, which on recrystallization

from ethanol gave yellow single crystals of (I) (yield 0.5 g, 60%; m.p. 389–391 K). IR (ATR, cm^{-1}): ν 2922–2851, 1733, 1667. ^1H NMR (300 MHz, $CDCl_3$, p.p.m.): δ 3.79 (*s*, 3H, CH_3), 4.72 (*s*, 2H, CH_2), 7.2–7.5 (*m*, Harm). ^{13}C NMR (75 MHz, $CDCl_3$, p.p.m.): δ 43.4, 52.9, 122.5, 123.7, 124.0, 136.0, 153.0, 167.5, 203.0.

Crystal data

$C_{10}H_9NO_3S$
M_r = 223.25
Monoclinic, $P2_1$
a = 4.7858 (3) Å
b = 12.4010 (7) Å
c = 17.5319 (10) Å
β = 95.679 (5)°
V = 1035.39 (11) Å^3
Z = 4
D_x = 1.432 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1967 reflections
θ = 2.0–28.0°
μ = 0.30 mm^{-1}
T = 296 K
Prism, pale yellow
0.53 × 0.35 × 0.17 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.858, T_{max} = 0.951
17159 measured reflections
4931 independent reflections
3875 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 27.9°
$h = -6 \rightarrow 6$
$k = -16 \rightarrow 16$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.109
S = 1.03
4931 reflections
274 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.0745P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Absolute structure: Flack (1983), 2328 Friedel pairs
Flack parameter: 0.000 (1)

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.741 (2)	O5—C19	1.184 (4)
S1—C7	1.769 (3)	O6—C19	1.310 (3)
S2—C11	1.737 (3)	O6—C20	1.437 (4)
S2—C17	1.750 (5)	N1—C8	1.442 (3)
O1—C7	1.209 (3)	N1—C6	1.386 (3)
O2—C9	1.191 (3)	N1—C7	1.379 (3)
O3—C10	1.458 (4)	N2—C16	1.384 (4)
O3—C9	1.320 (3)	N2—C17	1.395 (5)
O4—C17	1.227 (5)	N2—C18	1.444 (4)
C1—S1—C7	91.71 (11)	S1—C7—O1	124.7 (2)
C11—S2—C17	90.90 (16)	N1—C8—C9	112.55 (19)
C9—O3—C10	116.3 (2)	O2—C9—C8	124.7 (2)
C19—O6—C20	117.1 (3)	O3—C9—C8	110.39 (19)
C6—N1—C7	115.51 (19)	O2—C9—O3	124.9 (2)
C7—N1—C8	120.4 (2)	S2—C11—C12	127.9 (2)
C6—N1—C8	124.0 (2)	S2—C11—C16	111.55 (19)
C16—N2—C18	123.4 (3)	N2—C16—C11	113.3 (2)
C16—N2—C17	113.2 (3)	N2—C16—C15	126.7 (2)
C17—N2—C18	123.1 (3)	S2—C17—O4	127.4 (4)
S1—C1—C2	128.41 (19)	S2—C17—N2	110.9 (3)
S1—C1—C6	110.89 (17)	O4—C17—N2	121.7 (4)
N1—C6—C1	112.54 (19)	N2—C18—C19	111.7 (3)
N1—C6—C5	126.6 (2)	O5—C19—O6	125.0 (3)
O1—C7—N1	126.0 (2)	O5—C19—C18	124.7 (3)
S1—C7—N1	109.31 (17)	O6—C19—C18	110.3 (3)

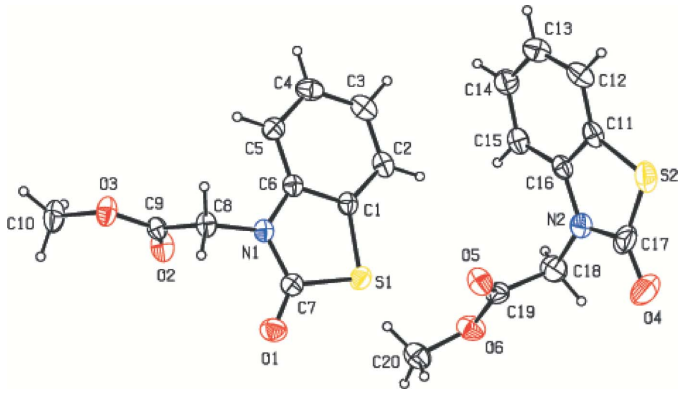


Figure 1
The structure of the asymmetric unit of (I), with the atom-numbering scheme and 20% probability displacement ellipsoids.

H atoms were introduced at calculated positions and treated as riding [U_{iso}(H) = 1.5U_{eq}(C) for methyl groups and 1.2U_{eq}(C) for other atoms, and C—H = 0.93, 0.96 and 0.97 Å]. In the absence of significant anomalous dispersion effects, Friedel pairs were merged prior to refinement. The large anisotropic displacement parameters of atoms S2, C17 and O4 in the benzothiazole ring system indicate either high thermal motion or possibly an unresolved disorder.

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).

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