Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 2-amino-4-methyl-6-phenyl-6*H*-1,3-thiazine-5-carboxylate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.106
Data-to-parameter ratio = 17.6

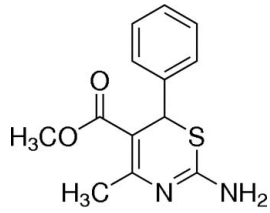
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{13}H_{14}N_2O_2S$, intermolecular N—H···O(N) hydrogen bonds link the molecules in the crystal structure into sheets parallel to the *bc* plane.

Received 10 June 2005
Accepted 15 June 2005
Online 24 June 2005

Comment

The molecular structure of the title compound, (I), is shown in Fig. 1. Hydrogen-bond geometry details are listed in Table 1. Two molecules related by an inversion center are linked by paired N—H···N hydrogen bonds *via* the nitrogen acceptor of the thiazine ring. A further N—H···O hydrogen bond with a carboxylate oxygen acceptor results in the formation of sheets parallel to the *bc* plane (Fig. 2)



(I)

Experimental

A mixture of methyl acetoacetate (4.0 mmol), benzaldehyde (4.0 mmol) and piperidinium acetate (14 mg) was stirred at 383 K for 60 min. Thiourea (4.0 mmol), polyphosphate acid (600 mg) and methanol (10 ml) were then added. The reaction mixture was refluxed for 4 h, cooled and poured into saturated $NaHCO_3$ solution (30 ml), and then extracted with ethyl acetate (30 ml). The organic phase was separated, dried with anhydrous Na_2SO_4 and evaporated *in vacuo*. The residue was purified by flash column chromatography on silica gel (eluting with hexane/ethyl acetate, 3:1) to give the desired product (yield 80%). Colorless crystals were obtained from EtOH solution after allowing it to stand for 4 d (m.p. 458–459 K). IR (KBr): 3362, 3000, 2951, 1690, 1654, 1527, 1206, 1062 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ 7.26 (*d*, J = 7.1 Hz, 2H), 7.23 (*m*, 1H), 7.17 (*d*, J = 7.1 Hz, 2H), 5.20–5.50 (*br s*, 2H), 5.31 (*s*, 1H), 3.68 (*s*, 3H), 2.49 (*s*, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.5, 158.8, 155.3, 141.8, 128.9, 127.9, 127.0, 103.0, 51.8, 42.8, 23.7. MS (EI): *m*/*z* 262 $[M]^+$. HRMS: *m*/*z* $[M]^+$ calculated for $C_{13}H_{14}N_2O_2S$: 262.0776; found: 262.0784.

Crystal data

$C_{13}H_{14}N_2O_2S$
M_r = 262.33
Monoclinic, $P2_1/c$
a = 11.3798 (4) Å
b = 8.7584 (3) Å
c = 13.3900 (6) Å
β = 99.178 (1)°
V = 1317.48 (9) Å^3
Z = 4
D_x = 1.322 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7566 reflections
θ = 3.0–27.4°
μ = 0.24 mm^{-1}
T = 295 (1) K
Block, colorless
0.32 × 0.30 × 0.27 mm

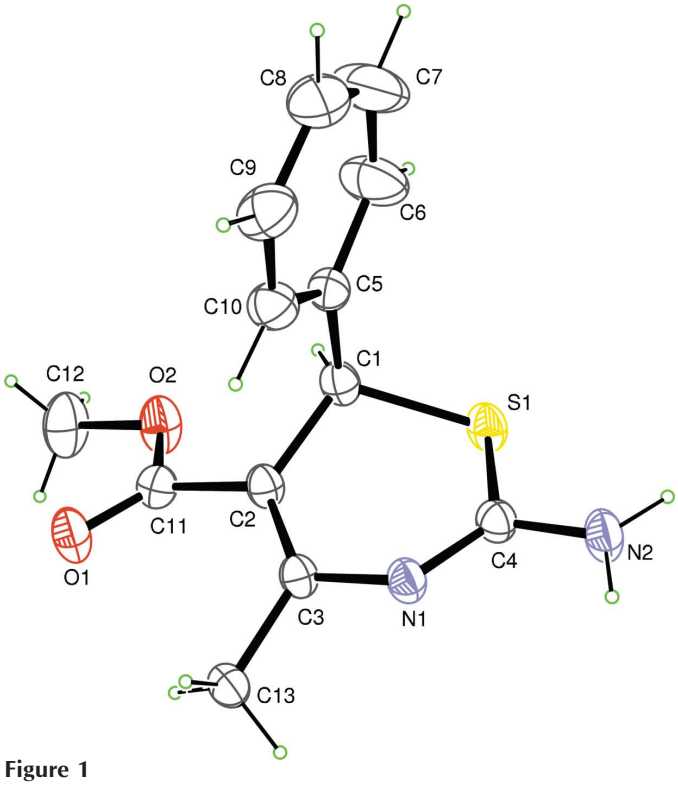


Figure 1
The molecule of compound (I) in the crystal structure. Displacement ellipsoids are drawn at the 40% probability level.

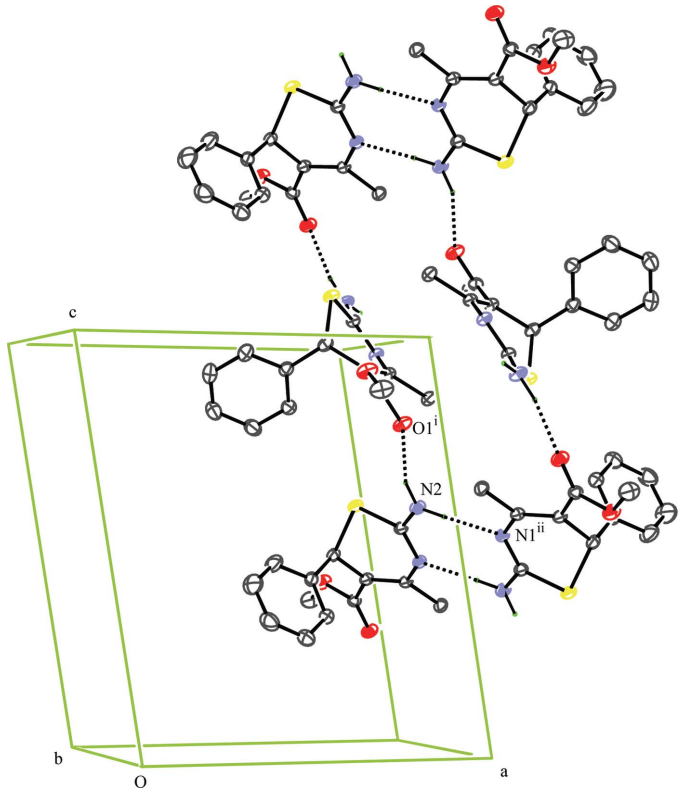


Figure 2
The molecular packing of (I), viewed approximately along the *b* axis. Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in the hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $2-x, -y, 1-z$.]

Data collection

Rigaku R-AXIS RAPID diffractometer	3005 independent reflections
ω scans	2078 reflections with $F^2 > 2\sigma(F^2)$
Absorption correction: multi-scan (*ABSCOR*: Higashi, 1995)	$R_{int} = 0.031$
$T_{min} = 0.911$, $T_{max} = 0.937$	$\theta_{max} = 27.5°$
12745 measured reflections	$h = -14 \rightarrow 14$
	$k = -11 \rightarrow 11$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[0.0011F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.106$	$\Delta\rho_{max} = 0.34$ e Å^{-3}
$S = 1.00$	$\Delta\rho_{min} = -0.31$ e Å^{-3}
2884 reflections	Extinction correction: Larson (1970), equation 22
164 parameters	Extinction coefficient: 70 (21)
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H201···O1^{i}	0.92	2.08	2.9425 (18)	157
N2—H202···N1^{ii}	0.90	2.05	2.9515 (18)	175

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

Atoms H201 and H202 were found in a difference Fourier map and fixed in position. The other H atoms were placed in calculated positions, with C—H = 0.96 or 0.98 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atoms. The scope of the 2θ refinement was restricted.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC,2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 20036); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2003).

The author thanks the National Natural Science Foundation of China (No. 20272051) and the Teaching and Research Award Program for Outstanding Young Teachers in Higher Education Institutions of MOE, People's Republic of China.

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