Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 2,2,4,8-tetramethyl-6-oxo-2*H*,6*H*-pyrimidino[2,1-*b*]thiazine-7-carboxylate

Michel Evain,[a]* Cyrille Landreau,[b] David Deniaud,[b] Alain Reliquet[b] and Jean Claude Meslin[b]

[a]Institut des Matériaux Jean Rouxel, 2 rue de la Houssinière, BP 32229, 44322 Nantes Cedex 3, France, and [b]Laboratoire de Synthèse Organique, UMR CNRS 6513, Faculté des Sciences et des Techniques, 2 rue de la Houssinière, BP 92208, 44322 Nantes Cedex 3, France

Correspondence e-mail: evain@cnrs-imn.fr

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.102
Data-to-parameter ratio = 23.8

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

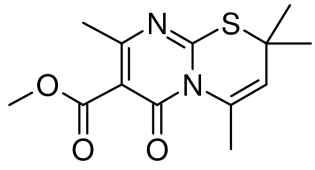
© 2002 International Union of Crystallography
Printed in Great Britain – all rights reserved

Received 13 March 2002
Accepted 19 March 2002
Online 28 March 2002

The title compound, $C_{13}H_{16}N_2O_3S$, contains a non-planar thiazine ring fused to a planar pyrimidine ring.

Comment

Despite the fact that pyrimido[2,1-*b*][1,3]thiazines are rather uncommon heterocycles, these compounds may be regarded as potentially biologically active candidates. A series of new 3,4-dihydro-2*H*,6*H*-pyrimido[2,1-*b*][1,3]thiazines was therefore evaluated by Bózsing *et al.* (1996) and these molecules proved to be promising anti-inflammatory and antipyretic agents.



(I)

The main route to these bicyclic compounds involves the condensation of a 2-thioxopyrimidine with an appropriate dielectrophile, which entails, in certain cases, the formation of two regioisomers (Kappe & Roschger, 1989). Since then, several alternative routes have been proposed (*e.g.* Fülöp *et al.*, 1991; Hanefeld *et al.*, 1996), most of these suffering from low versatility.

Our continuing program in heterocyclic synthesis has led us to develop a regioselective methodology allowing the preparation of various 2*H*,6*H*-pyrimido[2,1-*b*][1,3]thiazine and 2*H*,6*H*-pyrimido[2,1-*b*][1,3]thiazin-6-one derivatives, such as the title compound, (I) (Landreau *et al.*, 2002). As part of these results, we report here the molecular structure of (I).

All the atoms of the pyrimidine ring and some of the atoms of the thiazine ring are almost coplanar (with a maximum deviation of 0.136 Å for C6). Atoms C2 and C4 of the thiazine ring lie on opposite sides of this plane, at distances of 0.729 (2) and 0.350 (2) Å, respectively. The methyl ester group is tilted by 55.32 (11)° out of the plane.

Experimental

A solution of *N*,*N*-dimethyl-*N*′-(4,6,6-trimethyl-6*H*-1,3-thiazin-2-yl)-acetamidine (2 mmol) and methyl malonyl chloride (2.4 mmol) in dichloromethane (10 ml) was stirred at room temperature for 6 h. After cooling to 273 K, triethylamine (4.8 mmol) was added and stirring was continued at room temperature for 16 h. After removal of the solvent, the residue was chromatographed using CH_2Cl_2/EtOAc (9:1) as eluent. Crystallization from a solution in diethyl ether

gave (I) as white crystals (95%); m.p. 403 K. Single crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature from diethyl ether.

Crystal data

$C_{13}H_{16}N_2O_3S$	D_x = 1.313 Mg m^{-3}
M_r = 280.3	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 12.399 (5) Å	θ = 9.2–16.8°
b = 12.8080 (9) Å	μ = 0.23 mm^{-1}
c = 13.348 (5) Å	T = 293 K
β = 138.032 (18)°	Block, colourless
V = 1417.5 (9) Å^3	0.30 × 0.15 × 0.15 mm
Z = 4	

Data collection

Nonius CAD-4 and Stoe IPDS diffractometers	2258 reflections with $I > 2\sigma(I)$
$\theta/2\theta$ and ω scans	R_{int} = 0.079
Absorption correction: Gaussian (*JANA*2000; Petricek & Dusek, 2000)	θ_{max} = 30.1°
T_{min} = 0.941, T_{max} = 0.966	$h = -17 \rightarrow 16$
28 762 measured reflections	$k = -17 \rightarrow 17$
4119 independent reflections	$l = -17 \rightarrow 18$
	3 standard reflections
	frequency: 60 min
	intensity decay: 0.7%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$wR(F^2)$ = 0.102	$\Delta\rho_{min}$ = −0.42 e Å^{-3}
S = 1.07	Extinction correction: B—C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
4119 reflections	Extinction coefficient: 0.23 (4)
173 parameters	
H-atom parameters constrained	
$w = 1/[\sigma^2(I) + 0.0016I^2]$	

CAD-4 and IPDS data sets (8371 and 20391 reflections, respectively) were scaled on the basis of 3274 common reflections with $I > 10\sigma(I)$ [scale factor: 0.1710 (3)]. The orientation of the CH_3 groups were determined from difference Fourier syntheses and initially refined using rigid bodies. All H atoms were then fixed at calculated and/or refined positions. Riding isotropic displacement parameters were used for all H atoms.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1993) and *EXPOSE* (Stoe & Cie, 1997); cell refinement: *CELDIM* (Enraf–Nonius, 1993); data reduction: *JANA*2000 (Petricek & Dusek, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995); program(s) used to refine structure: *JANA*2000; molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *JANA*2000.

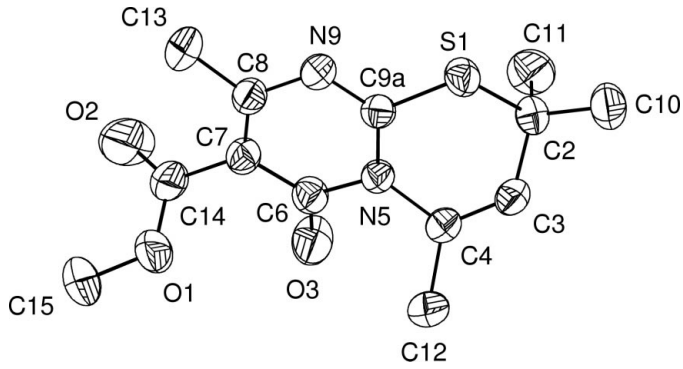


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

The authors gratefully acknowledge financial support by the French Ministry of Education and CNRS.

References

Becker, P. J. & Coppens, P. (1974). *Acta Cryst.* A**30**, 129–153.
Bózsing, D., Sohár, P., Gigler, G. & Kovács, G. (1996). *Eur. J. Med. Chem.* **31**, 663–668.
Brandenburg, K. & Berndt, M. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Enraf–Nonius (1993). *CAD*-4-*PC*. Enraf–Nonius, Delft, The Netherlands.
Fülöp, F., Huber, I., Szabó, Á., Bernáth, G. & Sohár, P. (1991). *Tetrahedron*, **47**, 7673–7676.
Hanefeld, W., Naeeni, M. & Schlitzer, M. (1996). *J. Heterocycl. Chem.* **33**, 1903–1907.
Kappe, C. O. & Roschger, P. (1989). *J. Heterocycl. Chem.* **26**, 55–64.
Landreau, C., Deniaud, D., Reliquet, A. & Meslin, J. C. (2002). *Synthesis*, **3**, 403–408.
Petricek, V. & Dusek, M. (2000). *JANA*2000. Institute of Physics, Praha, Czech Republic.
Sheldrick, G. M. (1995). *SHELXTL*. Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Stoe & Cie (1997). *EXPOSE* and *CELL* in *Stoe IPDS*. Stoe & Cie GmbH, Darmstadt, Germany.