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Ethyl 2-(2,4-dichlorobenzylidene)-7-methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidine-6-carboxylate dichloromethane solvate

Xin-Gang Liu,* Ya-Qing Feng, Xiao-Fang Li and Zu-Pei Liang

School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: xingangl2002@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.059
wR factor = 0.179
Data-to-parameter ratio = 14.5

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

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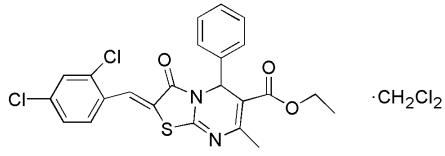
The title compound, $C_{23}H_{18}Cl_2N_2O_3S\cdot CH_2Cl_2$, was synthesized by heating ethyl 2-mercapto-4-methyl-6-phenyl-1,6-dihydropyrimidine-5-carboxylate, ethyl chloroacetate and 2,4-dichlorobenzaldehyde in ethanol. In the molecule, the nearly planar thiazole ring is fused with a dihydropyrimidine ring in a boat conformation.

Comment

1,4-Dihydropyrimidines show a very similar pharmacological profile to classical dihydropyridine calcium channel modulators (Lu *et al.*, 2002). We report here the synthesis and structure of the product of a dihydropyrimidine fused catalysis, namely ethyl 2-(2,4-dichlorobenzylidene)-7-methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidine-6-carboxylate dichloromethane solvate, (I). The molecular structure of (I) is illustrated in Fig. 1.



(I)

In the molecule, the thiazole ring is fused with a dihydropyrimidine ring in a boat conformation. The thiazoline ring (C8/C9/C10/N1/S1) is essentially planar, with a mean deviation of 0.006 (2) Å. The dihedral angle between the C8/C9/C10/N1/S1 plane and the C1–C6 benzene ring is 5.3 (2)°, and that between the C8/C9/C10/N1/S1 plane and the C15–C20 phenyl ring is 84.9 (3)°.

Experimental

A mixture of 2-mercapto-4-methyl-6-phenyl-1,6-dihydropyrimidine-5-carboxylic acid ethyl ester (0.02 mol), ethyl chloroacetate

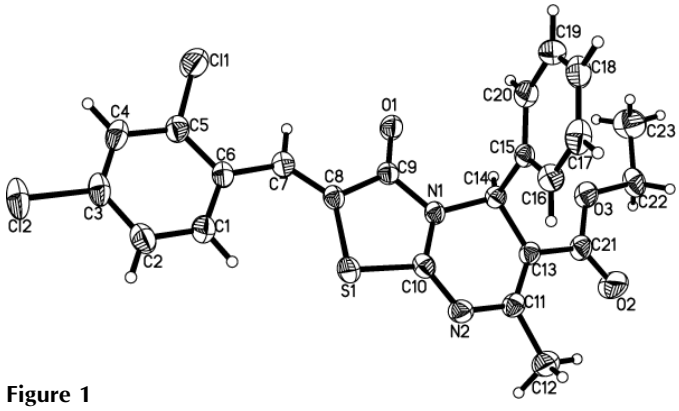


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids.

(0.02 mol) and pyridine (0.02 mol) was stirred under reflux in ethanol (40 ml) for 4 h. 2,4-Dichlorobenzaldehyde (0.02 mol) and piperidine (0.02 mol) were added and the mixture was refluxed for 4 h. After cooling and filtration, the title compound was recrystallized from acetic acid. ^{1}H NMR (p.p.m.): 1.19 (*m*, 3H, CH_3), 2.52 (*s*, 3H, CH_3), 4.12 (*m*, 2H, CH_2), 6.20 (*s*, 1H, CH), 7.27–7.47 (*m*, 8H, ArH), 7.74 (*s*, 1H,CH). M.p. 455–456 K. A portion (20 mg) of (I) was dissolved in dichloromethane (15 ml); the solution was kept at room temperature for 8 d and natural evaporation gave colorless single crystals of (I), suitable for X-ray analysis.

Crystal data

$C_{23}H_{18}Cl_2N_2O_3S\cdot CH_2Cl_2$
M_r = 558.28
Triclinic, $P\overline{1}$
a = 9.747 (4) Å
b = 11.291 (4) Å
c = 12.297 (5) Å
α = 84.546 (7)°
β = 74.655 (6)°
γ = 78.378 (6)°
V = 1277.1 (9) Å^3
Z = 2
D_x = 1.452 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1016 reflections
θ = 2.2–26.1°
μ = 0.58 mm^{-1}
T = 293 (2) K
Block, colorless
0.22 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.777, T_{max} = 0.933
6583 measured reflections
4478 independent reflections
2711 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.0°
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 8$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.180
S = 1.05
4478 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0864P)^2 + 0.2487P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

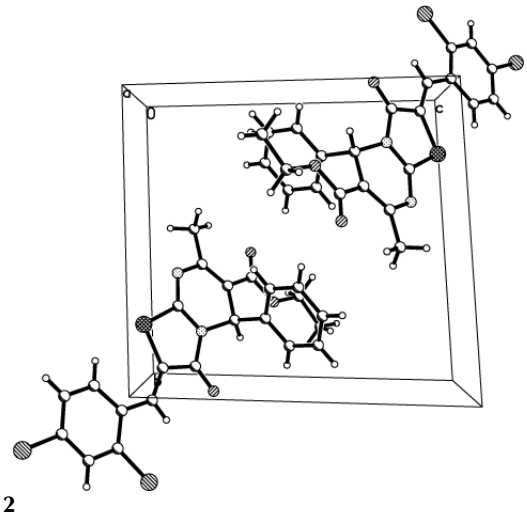


Figure 2
The crystal structure of (I), viewed along the *a* axis.

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and treated as riding [U_{iso} (H) = 1.2 U_{eq} (C)].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

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