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(2*Z*)-Ethyl 2-(4-chlorobenzylidene)-7-methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-1,3thiazolo-[3,2-*a*]pyrimidine-6-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.065
wR factor = 0.145
Data-to-parameter ratio = 13.6

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

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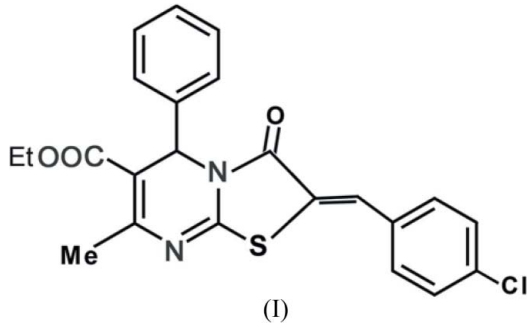
In the title compound, $C_{23}H_{19}ClN_2O_3S$, the central pyrimidine ring is significantly puckered, assuming a distorted chair conformation. Intermolecular C—H···O hydrogen-bond and π–π stacking interactions contribute to the stability of the structure.

Comment

The title compound, (I), belongs to a fused thiazolopyrimidine family. It possesses anticancer and anti-inflammatory activity. The anticancer drug screen was carried out using a diverse panel of cultured human tumor cell lines (Monks *et al.*, 1991). The anti-inflammatory activity is determined by inhibition in the Carageena-induced rat-paw edema method (Winter *et al.*, 1962). In view of these properties, the crystal structure of (I) has been determined.



(I)

Fig. 1 shows the molecular structure of (I) with the atom-numbering scheme. The pyrimidine is in a distorted chair form, as indicated by the puckering analysis [q_2 = 0.181 (3), q_3 = 0.066 (3) Å, θ = 69.9 (9) and φ = 35.5 (10)°; Cremer & Pople, 1975]. The thiazole ring makes dihedral angles of 82.8 (2) and 9.6 (2)° with benzene rings C11–C16 and C18–C23, respectively. The geometry of the thiazole ring is unremarkable. All bond lengths and angles in the pyrimidine ring have normal values, with the exception of N1—C1 and N1—C4; in (I), these are 1.274 (4) and 1.423 (4) Å, respectively. The corresponding values in the Cambridge Structural Database (2006 release; Allen, 2002) differ slightly, *viz*. 1.31 and 1.39 Å, respectively. The short C9—C10 bond distance [1.478 (6) Å] can probably be attributed to unresolved disorder of the terminal methyl group, as indicated by the displacement parameters of atoms C9 and C10. The C3—C8—O2—C9 and C8—O2—C9—C10 torsion angles of 175.0 (3) and −168.9 (4)°, respectively, describe the *trans* conformation of the ethoxy group.

The crystal structure of (I) is stabilized by an intermolecular C—H···O hydrogen bond (Fig. 2 and Table 1) and π–π

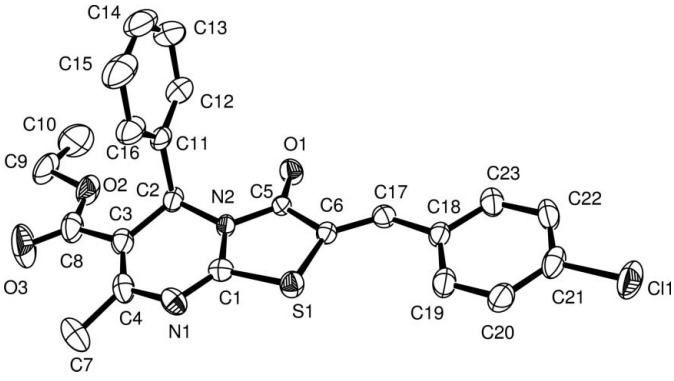


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids.

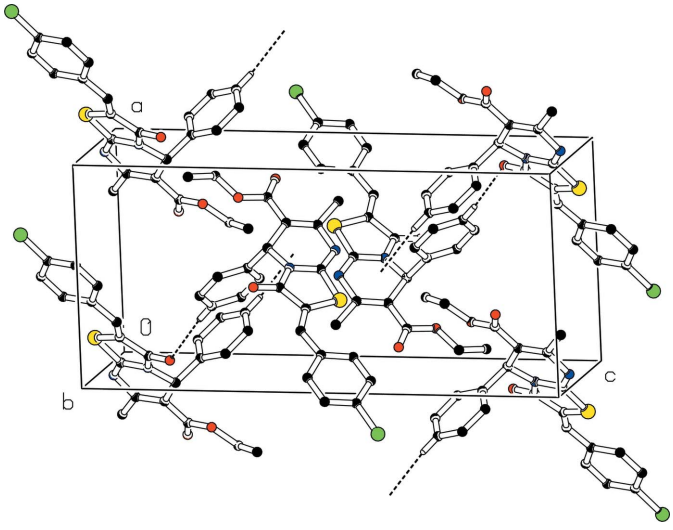


Figure 2
PLATON (Spek, 2003) plot of (I), showing intermolecular C—H···O interactions as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

stacking interactions. A *PLATON* analysis (Spek, 2003) of (I) indicated that short intramolecular C—H···O and C—H···S hydrogen bonds may also help to consolidate the crystal packing. There is a comparatively weak π–π stacking interaction between the C18–C23 benzene rings at (x, y, z) and ($1-x, 1-y, -z$); their centroids are separated by 3.731 (3) Å and the rings have a slippage of 1.318 Å (Fig. 3).

Experimental

A mixture of ethyl 6-methyl-4-phenyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (0.01 mol), chloroacetic acid (0.01 mol), fused sodium acetate (6 g) in glacial acetic acid (25 ml), acetic anhydride (10 ml) and benzaldehyde (0.01 mol) was refluxed for 3 h. The reaction mixture was cooled and poured into cold water. The resulting solid was collected and crystallized from methanol to obtain the final product (85% yield; m.p. 419 K). The compound was recrystallized by slow evaporation of an ethanol solution, yielding yellow needle-shaped single crystals suitable for X-ray diffraction.

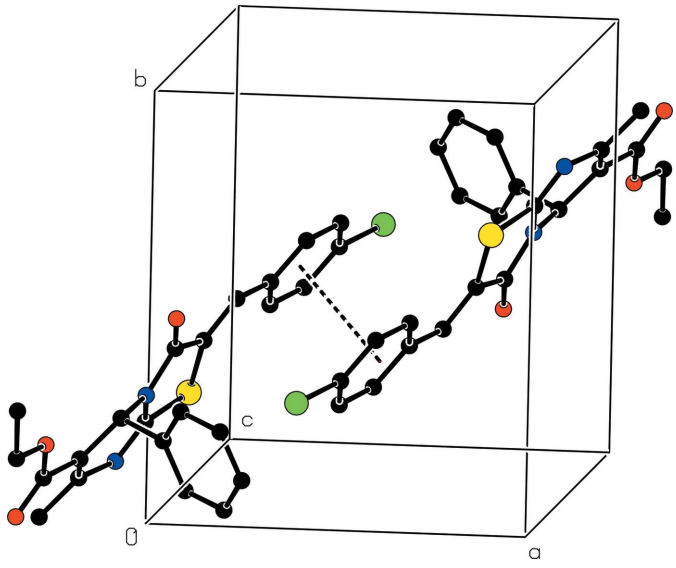


Figure 3
A view of the π–π stacking interaction (dashed line) in the crystal structure of (I). H atoms have been omitted for clarity.

Crystal data

$C_{23}H_{19}ClN_2O_3S$
M_r = 438.91
Monoclinic, $P2_1/n$
a = 9.597 (5) Å
b = 10.907 (5) Å
c = 20.607 (5) Å
β = 91.970 (5)°
V = 2155.8 (16) Å^3
Z = 4
D_x = 1.352 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 293 (2) K
Needle, yellow
0.4 × 0.2 × 0.1 mm

Data collection

Bruker SMART CCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.93, T_{max} = 0.978
10312 measured reflections
3698 independent reflections
2615 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.145
S = 1.06
3698 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.0325P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C19—H19···S1	0.93	2.63	3.311 (4)	131
C7—H7*C*···O3	0.96	2.21	2.915 (6)	129
C14—H14···O1^{i}	0.93	2.53	3.450 (5)	172

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

H atoms were placed in idealized positions (C—H = 0.93–0.98 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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