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Ethyl 5-(2,6-dichlorophenyl)-7-methyl-2-(1-naphthylmethylene)-3-oxo-2,3-dihydro-5*H*-thiazolo[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
Disorder in main residue
R factor = 0.040
wR factor = 0.126
Data-to-parameter ratio = 12.5

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

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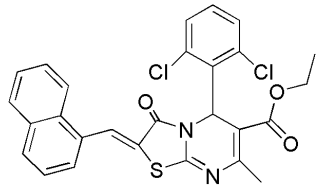
The title compound, $C_{27}H_{20}Cl_2N_2O_3S$, was synthesized by the reaction of ethyl 2-mercapto-4-methyl-6-(2,6-dichlorophenyl)-1,6-dihydro-pyrimidine-5-carboxylate, ethyl chloroacetate and aldehyde in acetic acid and acetic anhydride. In the molecule, the thiazolo[3,2-*a*]pyrimidine and naphthalene systems are essentially coplanar, with a dihedral angle between the combined plane and the mean plane of the benzene ring of the 2,6-dichlorophenyl substituent of 94.7 (4)°.

Comment

Dihydropyrimidine and its derivatives exhibit a wide variety of therapeutic and pharmacological properties. For example, dihydropyrimidines are novel calcium antagonists with potent and long-lasting vasodilative effects (Cho *et al.*, 1989). They have also been shown to have antiviral, antihypertensive (Wipf & Cunningham, 1995) and calcium channel blocking activity (Rovnyak *et al.*, 1992; Atwal *et al.*, 1990). In addition, dihydropyrimidines have also been found in some novel alkaloids, isolated from marine organisms, that exhibit biological activity (Fu *et al.*, 2003). Thus there has been considerable interest in the chemistry of dihydropyrimidines and their derivatives in recent years. We report here the crystal structure of the title compound, (I) (Fig. 1).



(I)

The bond lengths and angles in (I) have similar values to those of ethyl 5″-(2,6-dichlorophenyl)-1′-methyl-4-(1-naphthyl)-2,3″-dioxo-2,3,2″,3″-tetrahydro-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-2″-thiazolo[3″,2″-a]pyrimidine-6″-carboxylate ethyl acetate hemisolvate (MSDP; Li *et al.*, 2003), except for those of the thiazole ring. The C14—S1 bond length of 1.744 (3) Å is sightly longer than that in MSDP, and the C14—S1—C15 angle of 91.77 (2)° is sightly smaller than that in MSDP. The Cl—C bond lengths are in the range 1.732 (6)–1.746 (7) Å, in agreement with values reported in the literature (Busetti *et al.*, 1980; Sutherland & Ali-Adib, 1987).

The thiazolo[3,2-*a*]pyrimidine and naphthalene systems are coplanar to within 0.0395 (2) Å, and the dihedral angle between their mean plane and that of the C1–C6 benzene ring is 94.7 (4)°.

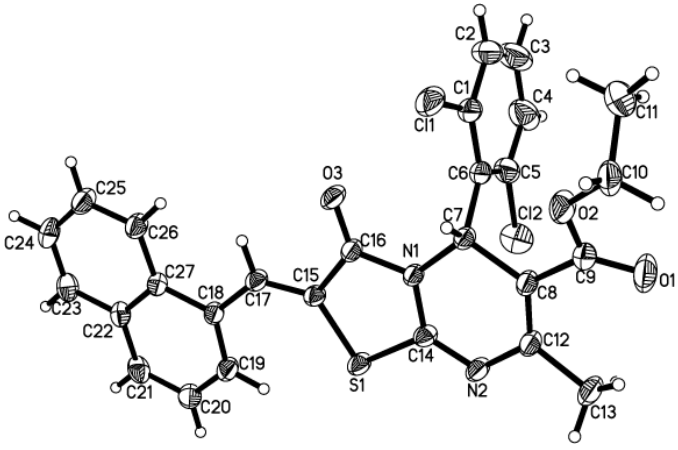


Figure 1
The molecular structure of (I), drawn with 30% displacement ellipsoids. The minor component of the disordered ethyl group has been omitted for clarity.

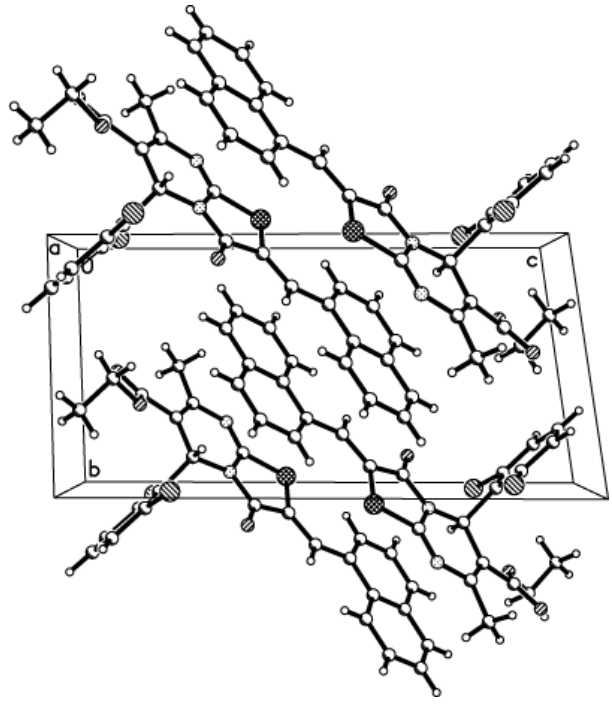


Figure 2
The crystal structure of (I), viewed along the *a* axis.

Experimental

A mixture of ethyl 2-mercapto-4-methyl-6-(2,6-dichlorophenyl)-1,6-dihydro-pyrimidine-5-carboxylate (0.02 mol), chloroacetic acid (0.02 mol) and an equimolar amount naphthylaldehyde in 20 ml of acetic acid and 10 ml of acetic anhydride was refluxed for 3 h (Mohamed & Abou-Elfotooh, 1978). The reaction mixture was poured into cold water, and the resulting precipitate was collected and recrystallized from acetic acid. ^{1}H NMR: δ 1.18 (*m*, 3H, –CH_3), 2.55 (*s*, 3H, –CH_3), 4.08 (*m*, 2H, –CH_2), 6.11 (*s*, 1H, –CH), 7.24–7.55 (*m*, 10H, ArH), 7.68 (*s*, 1H,–CH); m.p. 514–515 K. 20 mg of (I) was dissolved in 15 ml chloroform. The solution was kept at room temperature for 10 d and natural evaporation gave colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{27}H_{20}Cl_2N_2O_3S$	$Z = 2$
$M_r = 523.41$	$D_x = 1.455$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.974$ (3) Å	Cell parameters from 826 reflections
$b = 9.126$ (3) Å	$\theta = 2.8$–$26.3°$
$c = 16.463$ (6) Å	$\mu = 0.39$ mm^{-1}
$\alpha = 84.194$ (6)°	$T = 293$ (2) K
$\beta = 87.085$ (6)°	Block, colorless
$\gamma = 62.970$ (5)°	0.18 × 0.16 × 0.16 mm
$V = 1194.8$ (8) Å^3	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	4182 independent reflections
	2795 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.025$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$\theta_{max} = 25.0°$
	$h = -10 \rightarrow 9$
$T_{min} = 0.867$, $T_{max} = 1.000$	$k = -10 \rightarrow 10$
6116 measured reflections	$l = -17 \rightarrow 19$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.065P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.126$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.25$ e Å^{-3}
4182 reflections	$\Delta\rho_{min} = -0.23$ e Å^{-3}
335 parameters	

H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and refined in a riding model, with U_{iso}(H) = 1.2U_{eq}(carrier). Atoms C10 and C11 are disordered; the C10—C11 distance is 1.54 (4) Å and the O2—C10 distance is 1.45 (3) Å.

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*.

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