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Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.054
 wR factor = 0.137
Data-to-parameter ratio = 18.9

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

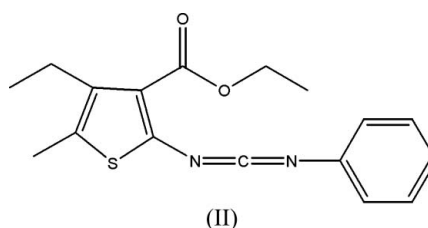
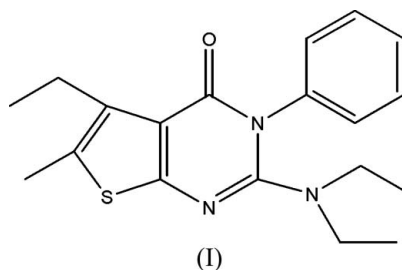
2-Diethylamino-6-ethyl-6-methyl-3-phenyl-thieno[2,3-*d*]pyrimidin-4(3*H*)-one

In the title compound, $\text{C}_{19}\text{H}_{23}\text{N}_3\text{OS}$, the two fused rings of the thieno[2,3-*d*]pyrimidin-4(3*H*)-one system are almost coplanar. The packing of the molecules in the crystal structure is determined by van der Waals forces. No intermolecular hydrogen-bonding interactions or π - π stacking interactions are present in the crystal structure.

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Comment

The derivatives of thieno[2,3-*d*]pyrimidin-4(3*H*)-ones are of great importance because of their remarkable biological properties (Santagati *et al.*, 2002; Wang *et al.*, 2005). In recent years, we have been engaged in the preparation of derivatives of heterocycles *via* an aza-Wittig reaction (Ding *et al.*, 2004). The heterocyclic title compound, (I), may be used as a new precursor for obtaining bioactive molecules and its structure is presented here (Fig. 1).



The two fused rings of the thieno[2,3-*d*]pyrimidin-4(3*H*)-one system of (I) are almost coplanar, with a maximum deviation of 0.046 (1) Å for atom C9. This plane is at an angle of 65.89 (8)° to that of the benzene ring. Bond lengths and angles (Table 1) are in agreement with reported literature values (Tashkhodzhaev *et al.*, 2002; Vasu *et al.*, 2004).

The crystal packing of (I) is determined by van der Waals forces. Neither intermolecular hydrogen-bonding interactions nor π - π stacking interactions are observed in the crystal structure.

Experimental

To a solution of ethyl 4-ethyl-5-methyl-3-oxo-1-phenyl-1H-thieno[2,3-*d*]pyrimidin-4(3*H*)-one-6-carboxylate (0.95 g, 2 mmol) in dry dichloromethane (15 ml)

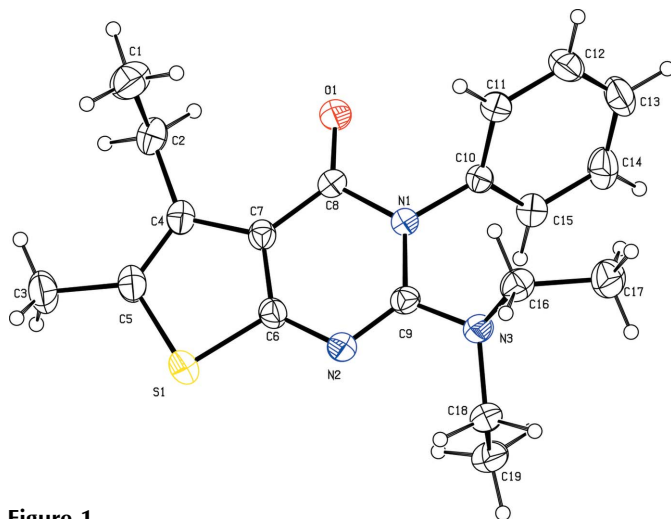


Figure 1

A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

was added phenyl isocyanate (2 mmol) under nitrogen at room temperature. After standing for 10 h at 273–278 K, the solvent was removed under reduced pressure and diethyl ether–petroleum ether (1:2, 20 ml) was added to precipitate triphenylphosphine oxide. After filtration, the solvent was removed to give the carbodiimide, (II), which was used directly without further purification. To a solution of (II), prepared as above, in dichloromethane (15 ml) was added diethylamine (2 mmol). After the reaction mixture had been allowed to stand for 30 min, the solvent was removed and anhydrous ethanol (10 ml) and several drops of EtONa in EtOH were added. The mixture was stirred for 3 h at room temperature and concentrated under reduced pressure; the residue was recrystallized from ethanol to give the title compound, (I) (yield 0.65 g, 95.0%, m.p. 418 K). Suitable crystals were obtained by vapour diffusion of ethanol into a solution in dichloromethane at room temperature. IR (KBr, ν , cm^{-1}): 1681 (C=O), 1540 (C=N), 2965 (C–H, alkyl).

Crystal data

$\text{C}_{19}\text{H}_{23}\text{N}_3\text{OS}$
 $M_r = 341.46$
 Monoclinic, $P2_1/c$
 $a = 8.8085$ (10) Å
 $b = 9.5503$ (11) Å
 $c = 22.037$ (3) Å
 $\beta = 97.269$ (2)°
 $V = 1839.0$ (4) Å³
 $Z = 4$

$D_x = 1.233$ Mg m⁻³
 $D_m = 1.233$ Mg m⁻³
 D_m measured by flotation
 Mo $K\alpha$ radiation
 $\mu = 0.19$ mm⁻¹
 $T = 292$ (2) K
 Block, yellow
 $0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 11310 measured reflections

4174 independent reflections
 3015 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.137$
 $S = 1.02$
 4174 reflections
 221 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0682P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C5–S1	1.739 (2)	C9–N3	1.376 (2)
C6–N2	1.361 (2)	C10–N1	1.453 (2)
C6–S1	1.7240 (18)	C16–N3	1.476 (2)
C8–O1	1.212 (2)	C18–N3	1.476 (2)
C9–N2	1.303 (2)		
C4–C5–S1	112.28 (14)	C15–C10–N1	118.68 (16)
N2–C6–C7	127.41 (16)	N3–C16–C17	112.47 (16)
C7–C6–S1	110.97 (13)	C9–N1–C8	122.48 (14)
O1–C8–N1	119.51 (16)	C9–N2–C6	115.40 (15)
N1–C8–C7	113.44 (15)	C9–N3–C18	115.77 (14)
N2–C9–N3	119.98 (16)	C18–N3–C16	113.93 (14)
N3–C9–N1	116.69 (15)	C6–S1–C5	91.72 (9)
C11–C10–N1	120.08 (15)		
N2–C6–C7–C4	−178.44 (17)	N1–C10–C15–C14	178.91 (16)
S1–C6–C7–C4	0.5 (2)	O1–C8–N1–C9	179.79 (16)
N2–C6–C7–C8	5.3 (3)	N3–C9–N2–C6	−179.92 (15)
S1–C6–C7–C8	−175.78 (13)	N2–C6–S1–C5	178.76 (15)
N1–C10–C11–C12	−178.37 (17)		

The H atoms were positioned geometrically [C–H = 0.93 (CH), 0.97 (CH₂) and 0.96 Å (CH₃)] and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2$ (1.5 for methyl) times $U_{\text{eq}}(\text{C})$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2001); software used to prepare material for publication: SHELXTL.

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