

3-(4-Chlorophenyl)-5-ethyl-6-methyl-2-morpholinothieno[2,3-*d*]pyrimidin-4(3*H*)-oneZheng-Dong Fang^{a,b*} and
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Key indicators

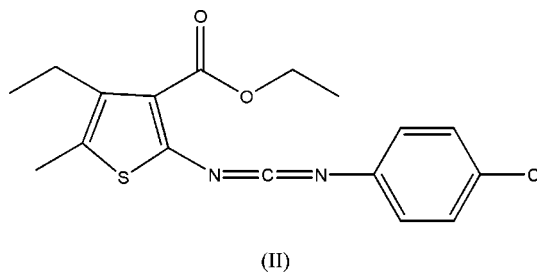
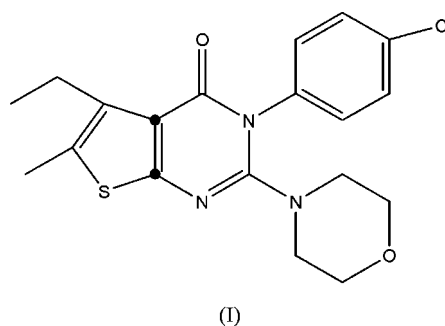
Single-crystal X-ray study
T = 292 K
Mean σ (C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.153
Data-to-parameter ratio = 18.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, C₁₉H₂₀ClN₃O₂S, 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 mainly governed by intermolecular C–H···O hydrogen-bonding and π – π interactions.

Received 25 April 2006
Accepted 1 June 2006

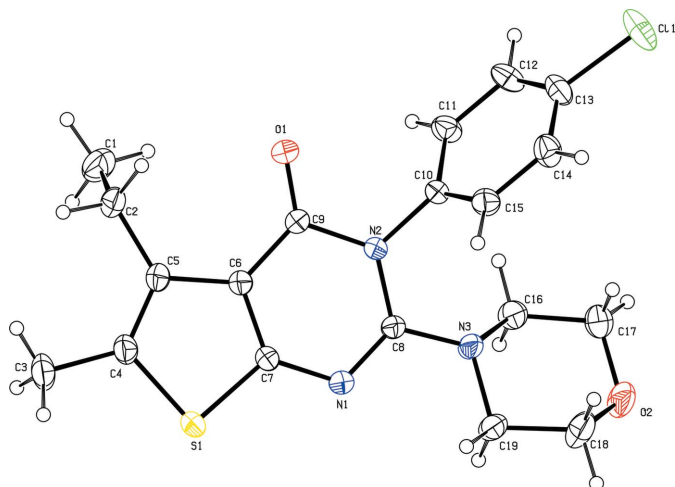
Comment

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 we report its structure here (Fig. 1).

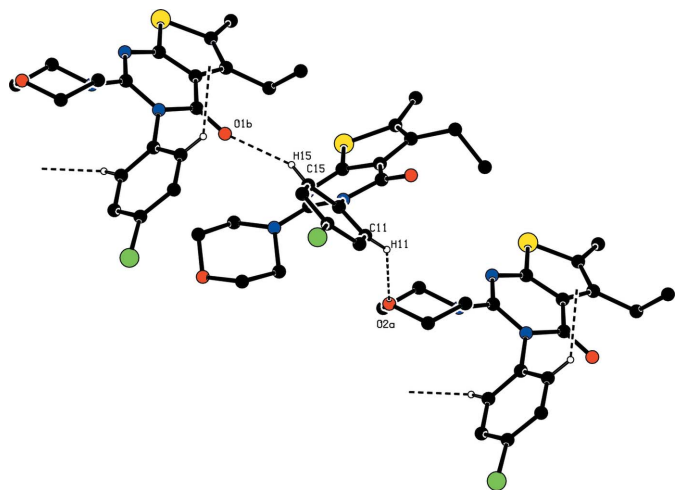


The two fused rings of the thieno[2,3-*d*]pyrimidin-4(3*H*)-one system of (I) are almost coplanar, with a maximum deviation of 0.045 (2) Å for atom C9. This plane is at an angle of 119.7 (2)° to the substituted benzene ring. Bond lengths and angles (Table 1) are in agreement with reported literature values (Tashkhodzhaev *et al.*, 2002; Vasu *et al.*, 2004). The morpholine ring has a half-chair conformation.

The crystal structure of (I) is stabilized by intermolecular C–H···O hydrogen bonding and π – π interactions (Fig. 2 and Table 2).


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.


Figure 2

A packing diagram for (I), showing the intermolecular C—H...O hydrogen-bonding interactions (dashed lines). [Symmetry codes: (a) $-x + \frac{1}{2}, y - \frac{1}{2}, z$; (b) $-x + \frac{3}{2}, y + \frac{1}{2}, z$.]

Experimental

To a solution of ethyl 4-ethyl-5-methyl-3-carboxylate thiophene iminophosphoraneiminophosphorane (0.95 g, 2 mmol) in dry dichloromethane (15 ml) was added 4-chlorophenyl isocyanate (2 mmol) under nitrogen at room temperature. After standing for 10 h at 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) in dichloromethane (15 ml) was added morpholine (2 mmol). After allowing the reaction mixture to stand for 4.5 h, 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, concentrated under reduced pressure and the residue recrystallized from ethanol to give the title compound, (I) (yield 0.61 g, 80.3%; m.p. 479 K). Suitable

crystals were obtained by vapour diffusion of ethanol into dichloromethane at room temperature.

Crystal data

$C_{19}H_{20}ClN_3O_2S$
 $M_r = 389.89$
 Orthorhombic, $Pbca$
 $a = 8.0455$ (6) Å
 $b = 11.7674$ (9) Å
 $c = 39.887$ (3) Å
 $V = 3776.2$ (5) Å³

$Z = 8$
 $D_x = 1.372$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.33$ mm⁻¹
 $T = 292$ (2) K
 Block, colourless
 $0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 22565 measured reflections

4490 independent reflections
 3621 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.061$
 $\theta_{max} = 28.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.153$
 $S = 1.04$
 4490 reflections
 237 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2 + 1.2775P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.002$
 $\Delta\rho_{max} = 0.54$ e Å⁻³
 $\Delta\rho_{min} = -0.40$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C1—C2	1.510 (3)	C10—N2	1.449 (2)
C3—C4	1.501 (3)	C13—Cl1	1.737 (2)
C5—C6	1.446 (3)	C17—O2	1.416 (3)
C7—N1	1.360 (2)	C18—H18B	0.9700
C7—S1	1.7214 (19)	C19—N3	1.459 (2)
C9—O1	1.218 (2)		
C2—C1—H1A	109.5	C10—C15—C14	119.42 (19)
C5—C2—C1	113.73 (19)	O2—C17—C16	111.45 (19)
C4—C3—H3A	109.5	O2—C18—C19	111.7 (2)
C5—C4—S1	112.66 (15)	N3—C19—H19B	110.1
C7—C6—C5	113.19 (16)	C8—N1—C7	115.40 (16)
N1—C8—N2	123.34 (16)	C19—N3—C16	111.02 (16)
C12—C13—C14	120.72 (19)	C7—S1—C4	91.55 (9)
C3—C4—C5—C6	178.2 (2)	N3—C8—N2—C9	177.51 (18)
C4—C5—C6—C7	1.3 (3)	O1—C9—N2—C8	-178.5 (2)
C7—C6—C9—O1	-175.3 (2)	C18—C19—N3—C8	-160.18 (19)
C11—C12—C13—C11	174.94 (19)	C19—C18—O2—C17	59.2 (3)
N2—C10—C15—C14	176.16 (19)	C6—C7—S1—C4	-0.28 (16)

Table 2

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C11—H11...O2 ⁱ	0.93	2.51	3.240 (3)	135
C15—H15...O1 ⁱⁱ	0.93	2.35	3.192 (2)	151

Symmetry codes: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, z$; (ii) $-x + \frac{3}{2}, y + \frac{1}{2}, z$.

All H atoms were positioned geometrically (C—H = 0.96–0.97 Å) and refined as riding, allowing for free rotation of the methyl groups; $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$ (methyl 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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