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2-(4-Chlorophenoxy)-3-isopropyl-5,6,7,8-tetrahydro-2-benzothieno[2,3-*d*]pyrimidin-4(3*H*)-one

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In the title compound, $C_{19}H_{19}ClN_2O_2S$, the thiophene and pyrimidine rings are almost coplanar. Intramolecular C—H···O hydrogen bonds stabilize the molecular structure. The crystal packing is determined by van der Waals forces. No intermolecular hydrogen bonding interactions or π–π stacking interactions are present in the crystal structure.

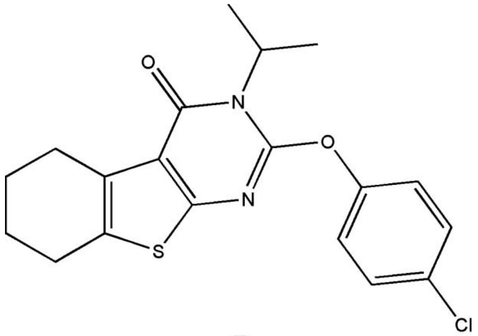
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.130
Data-to-parameter ratio = 18.1

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

Comment

Pyrimidine derivatives are the focus of increasing attention of the synthetic community because of the important role played by such systems in many natural products, antibiotics and drugs (Ding *et al.*, 2004). In recent years, we have been engaged in the preparation of derivatives of heterocycles *via* the aza-Wittig reaction. The title compound, (I), has been synthesized and structurally characterized in this context.



(I)

The molecular structure indicates that the thieno[2,3-*d*]pyrimidine system is conjugated (Fig. 1); all ring atoms in this system are essentially coplanar. The bond lengths and angles are, within experimental error, comparable to values in previously reported structures in the Cambridge Structural Database (Version 5.26; Allen, 2002).

Three intramolecular C—H···O hydrogen bonds stabilize the molecular structure (Table 1). The crystal packing is determined by van der Waals forces; neither intermolecular hydrogen bonding interactions nor π–π stacking interactions are observed.

Experimental

To a solution of iminophosphorane (1.45 g, 3 mmol) in anhydrous dichloromethane (15 ml) was added isopropyl isocyanate (3 mmol) under dry nitrogen at room temperature. The reaction mixture was left unstirred for 48 h at room temperature. The solvent was then removed under reduced pressure and diethyl ether/petroleum ether (1:2 *v*/*v*, 20 ml) was added to precipitate triphenylphosphine oxide.

After filtration, the solvent was removed, and the residue was dissolved in CH_3CN (15 ml). After adding 4-chlorophenol (3.1 mmol) and excess K_2CO_3 to the solution of the carbodiimide, the mixture was stirred for 24 h at room temperature, the solution was condensed and the residue was recrystallized from EtOH to give the title compound, (I), in a yield of 75% (m.p. 441 K). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.41–7.11 (*m*, 4H, Ar—H), 5.62 (*m*, 1H, CH), 2.99–2.67 (*m*, 4H, $2CH_2$), 1.84–1.82 (*m*, 4H, $2CH_2$), 1.58–1.56 (*d*, *J* = 6.4 Hz, 6H, $2CH_3$); MS (*m*/*z*, %), 377 (22), 374 (M^+, 91), 331 (36), 304 (29), 254 (100), 205 (33), 43 (26). Elemental analysis calculated (%) for $C_{19}H_{19}ClN_2O_2S$: C 60.87, H 5.11, N 7.47; found (%): C 60.56, H 5.17, N 7.32. Crystals suitable for single-crystal X-ray diffraction were obtained by vapor diffusion of hexane into a dichloromethane solution (1:3 *v*/*v*) at room temperature.

Crystal data

$C_{19}H_{19}ClN_2O_2S$
$M_r = 374.87$
Monoclinic, $P2_1$
$a = 13.2129$ (12) Å
$b = 5.7350$ (5) Å
$c = 13.2120$ (12) Å
$\beta = 115.13^\circ$
$V = 906.46$ (14) Å^3
$Z = 2$

$D_x = 1.373$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 3917 reflections
$\theta = 2.9$–26.7°
$\mu = 0.34$ mm^{-1}
$T = 292$ (2) K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.905$, $T_{\max} = 0.967$
10551 measured reflections

4119 independent reflections
3396 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.056$
$\theta_{\max} = 27.5^\circ$
$h = -17 \rightarrow 16$
$k = -7 \rightarrow 7$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.130$
$S = 1.00$
4119 reflections
228 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}
Absolute structure: Flack (1983), 1386 Friedel pairs
Flack parameter: 0.00 (8)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C10—H10$\cdots$O1	0.98	2.20	2.720 (3)	112
C11—H11$C\cdots$O2	0.96	2.35	2.876 (4)	114
C12—H12$A\cdots$O2	0.96	2.40	2.969 (4)	117

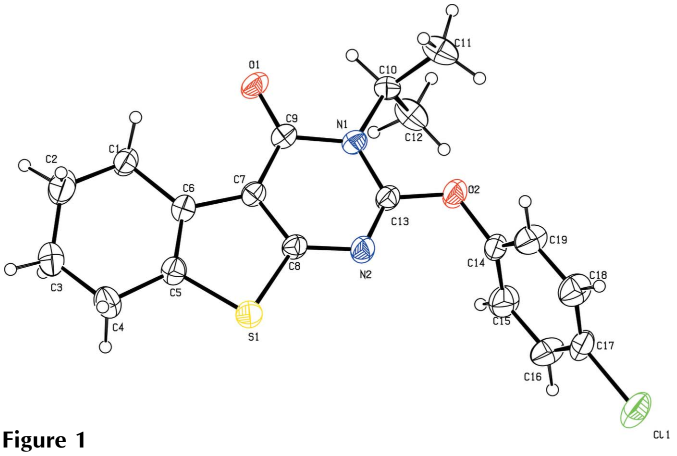


Figure 1
View of the molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

All H atoms were placed at calculated positions and treated as riding atoms, with C—H = 0.93–0.98 Å; $U_{\rm iso}({\rm H}) = xU_{\rm eq}({\rm C})$, where x = 1.5 for methyl H and x = 1.2 for other H atoms. It is noted that the chlorine atom has a very large value for U_{22}, *viz*. 0.1315 (9) Å^2, in a direction which is skewed with respect to the plane of the benzene ring.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

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