Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

3-Isopropyl-2-phenoxy-5,6,7,8-tetrahydro-1-benzothieno[2,3-*d*]pyrimidin-4(3*H*)-one

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Received 12 December 2005
Accepted 22 December 2005
Online 11 January 2006

In the title compound, $C_{19}H_{20}N_2O_2S$, the central thienopyrimidine ring system is essentially planar. The molecular structure is stabilized by C—H···O hydrogen bonds. In the crystal packing, the molecules are linked only by van der Waals forces.

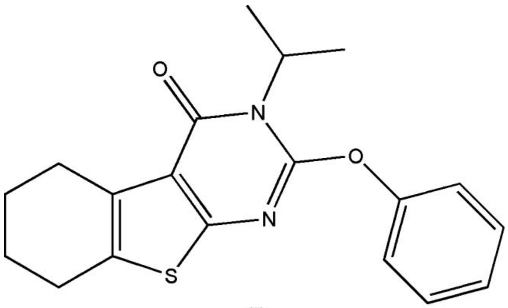
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.060
wR factor = 0.169
Data-to-parameter ratio = 15.0

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

Comment

Pyrimidine derivatives are attracting the 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), was synthesized and structurally characterized in this context.



(I)

In the title molecule (Fig. 1), the central thienopyrimidine ring system is essentially planar, with a maximum displacement of 0.077 (3) Å for atom N1. Selected bond lengths and angles are listed in Table 1. The C14–C19 phenyl ring is twisted by an angle of 75.90 (13)° with respect to the mean plane of the thienopyrimidine ring system.

There are three intramolecular C—H···O hydrogen bonds which stabilize the molecular structure (Table 2). The crystal packing is determined by van der Waals forces, neither intermolecular hydrogen-bonding interactions nor π-π stacking interactions being 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 and then the solvent was removed under reduced pressure and diethyl ether/petroleum ether (1:3 *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). PhOH (3.1 mmol) and excess K_2CO_3 were then added to the solution of the carbodiimide. After stirring the mixture for 18 h at room temperature, the solution was evapo-

doi:10.1107/S1600536805042881

rated and the residue was recrystallized from EtOH to give the title compound, (I), in a yield of 70% (m.p. 422 K). ^{1}H NMR ($CDCl_3$, 400 MHz): 7.46–7.17 (*m*, 5H, Ar-H), 5.63 (*m*, 1H, CH), 2.99–2.66 (*m*, 4H, $2CH_2$), 1.83–1.82 (*m*, 4H, $2CH_2$), 1.59–1.58 (*d*, *J* = 6.8 Hz, 6H, $2CH_3$); MS (EI, 70 eV) *m*/*z* (%): 340 (M^+, 100), 298 (53), 270 (45), 255 (76), 227 (42). Elemental analysis calculated for $C_{19}H_{20}N_2O_2S$: C 67.03, H 5.92, N 8.23%; found: C 66.85, H 5.85, N 8.17%. Crystals suitable for single-crystal X-ray diffraction were obtained from a hexane/dichloromethane solution (1:3 *v*/*v*) at room temperature.

Crystal data

$C_{19}H_{20}N_2O_2S$
M_r = 340.43
Monoclinic, $P2_1$
a = 12.4411 (15) Å
b = 5.8030 (7) Å
c = 13.2870 (16) Å
β = 113.994 (2)°
V = 876.37 (18) Å^3
Z = 2
D_x = 1.290 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1462 reflections
θ = 2.9–21.1°
μ = 0.20 mm^{-1}
T = 292 (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.943, $T_{\max}$ = 0.962
5473 measured reflections
3473 independent reflections
2596 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.025
$\theta_{\max}$ = 27.0°
$h = -13 \rightarrow 15$
$k = -7 \rightarrow 7$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.169
S = 1.05
3473 reflections
231 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0921P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.27 e Å^{-3}
$\Delta\rho_{\min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 1386 Friedel pairs
Flack parameter: −0.01 (12)

Table 1
Selected geometric parameters (Å, °).

C5—C6	1.367 (5)	C8—N2	1.370 (4)
C5—S1	1.745 (4)	C8—S1	1.707 (4)
C6—C7	1.415 (5)	C9—O1	1.220 (4)
C7—C8	1.391 (4)	C9—N1	1.416 (4)
C7—C9	1.444 (5)		
C8—S1—C5	91.48 (17)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O1	0.98 (4)	2.21 (4)	2.733 (5)	112 (3)
C11—H11*C*···O2	0.96	2.27	2.846 (6)	118
C12—H12*A*···O2	0.96	2.43	2.981 (5)	116

The positional parameters of H10, bonded to C10, were refined freely. All other H atoms were placed at calculated positions and treated as riding atoms, with C—H = 0.93–0.97 Å, and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for aromatic and methylene H atoms or 1.5$U_{\rm eq}$(C) for methyl H atoms. The terminal cyclohexene ring is disordered. There are two possible conformations, C1–C6 and C1/C2′/C3′/C4–C6, with an occupancy ratio of 0.690 (12):0.310 (12).

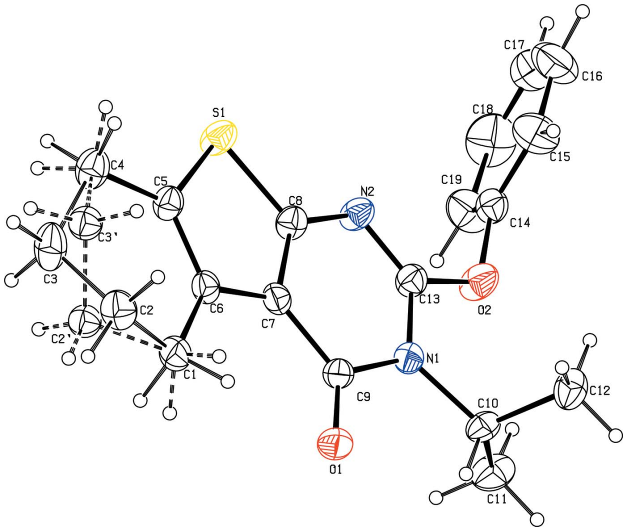


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size. Both disorder components are shown.

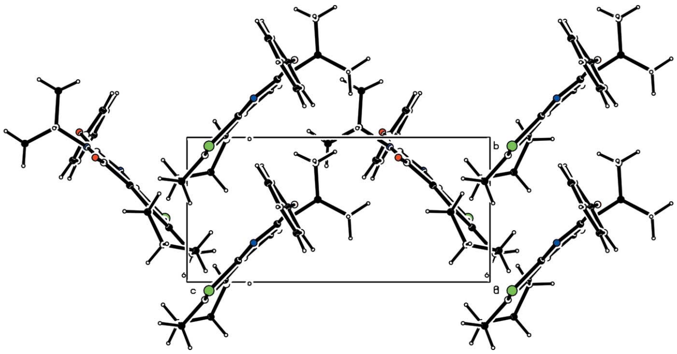


Figure 2
Packing diagram of the title compound, viewed along the *a* axis. Only the major conformation of the disordered cyclohexene ring is shown.

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).

We gratefully acknowledge financial support of this work by the National Basic Research Programme of China (2003CB114400) and the National Natural Science Foundation of China (Nos. 20372023 and 20102001).

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