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2-Ethoxy-3-isopropylbenzo[4,5]thieno[3,2-*d*]-pyrimidin-4(3*H*)-one

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.122
Data-to-parameter ratio = 14.7

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

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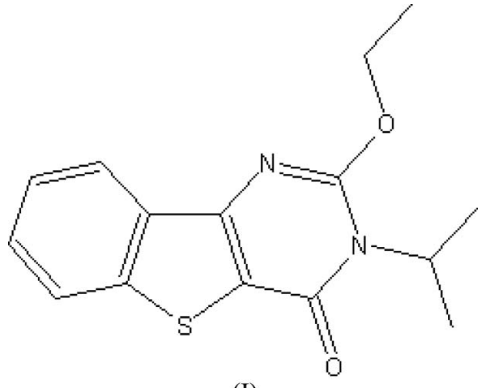
The molecule of the title compound, $C_{15}H_{16}N_2O_2S$, has crystallographic mirror symmetry. The crystal structure is stabilized by inter- and intramolecular C—H···O hydrogen-bonding interactions.

Comment

Thienopyrimidine derivatives are of interest as possible antiviral agents, and because of their other biological properties, including antibacterial, antifungal, antiallergic and anti-inflammatory. We have recently focused on the synthesis of fused heterocyclic systems containing thienopyrimidine *via* the aza-Wittig reaction at room temperature (Ding *et al.*, 2004). We present the X-ray crystallographic analysis of the title compound, (I), in this paper.



(I)

As shown in Fig. 1, compound (I) contains three fused rings. All the ring atoms in the benzothieno[3,2-*d*]pyrimidine system are essentially in the same plane. Selected bond lengths and

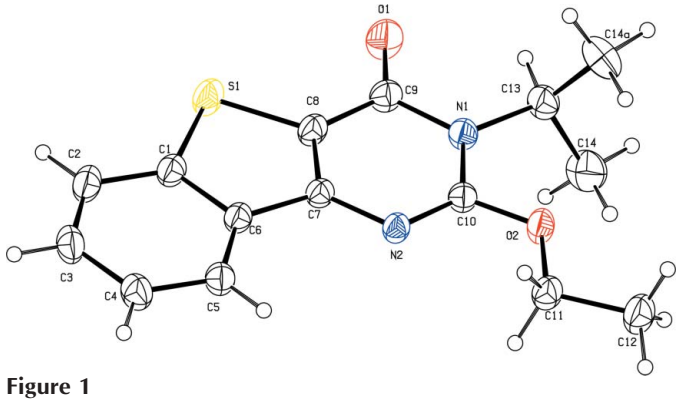


Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size [symmetry code: (*a*) $x, \frac{1}{2}-y, z$].

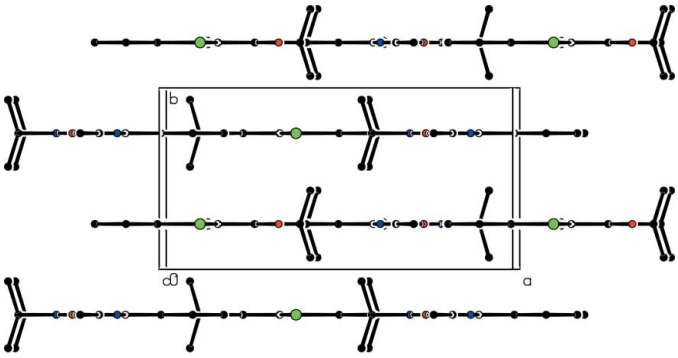


Figure 2
The crystal packing, viewed along the *c* axis, showing the planar sheets of molecules parallel to (010).

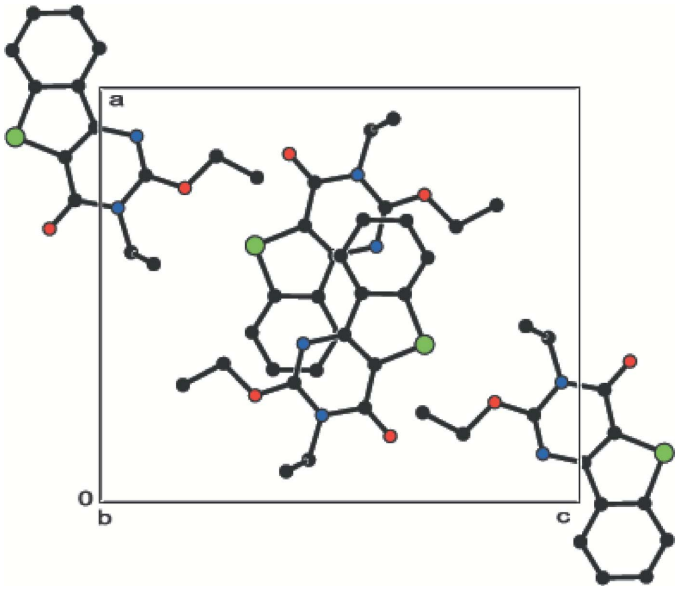


Figure 3
View of the packing of (I) along the *b* axis. H atoms bonded to C atoms have been omitted for clarity.

torsion angles are presented in Table 1. Some C—H···O inter- and intramolecular hydrogen bonds are present in the crystal structure (Table 1). The molecules are packed as layers parallel to the *ac* plane (Figs. 2 and 3).

Experimental

To a solution of ethyl 3-triphenylphosphoranylideneaminobenzo[*b*]-thiophene-2-carboxylate (3 mmol) in dry dichloromethane (5 ml) was added isopropyl isocyanate (3 mmol) under nitrogen at room temperature. After the reaction mixture was left to stand for 8–12 h at 273–278 K, the solvent was removed under reduced pressure and diethyl ether/petroleum ether (1:2 *v*/*v*, 12 ml) was added to precipitate triphenylphosphine oxide. After filtration, anhydrous ethanol (10 ml) was added with several drops of EtONa in EtOH. The mixture was stirred for 1–6 h at room temperature. The solution was then concentrated under reduced pressure and the residue was recrystallized from ethanol to afford compound (I) (yield: 65%, m.p. 446 K). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.19–7.45 (4H, Ar—H), 5.58 (*m*, 1H, N—CH), 4.68–4.62 (*m*, 2H, O—CH_2) 1.54–1.50 (*m*, 9H, CH_3). Crystals suitable for single-crystal X-ray diffraction were grown from dichloromethane at 300 K.

Crystal data

$C_{15}H_{16}N_2O_2S$
M_r = 288.36
Orthorhombic, *Pnma*
a = 13.3930 (16) Å
b = 6.8922 (8) Å
c = 15.4970 (18) Å
V = 1430.5 (3) Å^3
Z = 4
D_x = 1.339 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 1811 reflections
θ = 2.6–24.4°
μ = 0.23 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
8299 measured reflections
1768 independent reflections
1339 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.5°
h = −17 → 16
k = −8 → 8
l = −20 → 15

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.122
S = 1.06
1768 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 0.1581P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···O2	0.96	2.40	2.941 (2)	115
C13—H13···O1	0.98	2.22	2.744 (3)	112
C2—H2···O1^{i}	0.93	2.53	3.407 (3)	157

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{1}{2}$.

H atoms were positioned geometrically and treated as riding, with C—H = 0.93–0.98 Å and U_{iso}(H) values set equal to xU_{eq}(carrier atom), with *x* = 1.2 for C*sp*^{2} and *x* = 1.5 for methyl C parent atoms.

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: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

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