

Xiao-Hua Zeng, Ju-Zhen Yuan,
 Nian-Yu Huang, Ming-Wu Ding*
 and Hong-Wu He

Key Laboratory of Pesticide and Chemical
 Biology of the Ministry of Education, College of
 Chemistry, Central China Normal University,
 Wuhan 430079, People's Republic of China

Correspondence e-mail: zengken@126.com

Key indicators

Single-crystal X-ray study
 T = 292 K
 Mean $\sigma(C-C)$ = 0.003 Å
 R factor = 0.050
 wR factor = 0.126
 Data-to-parameter ratio = 18.0

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

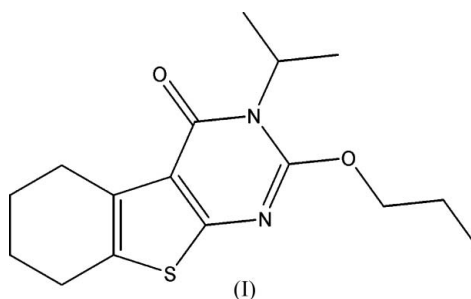
3-Isopropyl-2-propoxy-5,6,7,8-tetrahydro-1-benzothieno[2,3-d]pyrimidin-4(3H)-one

In the title compound, $C_{16}H_{22}N_2O_2S$, the fused thiophene and pyrimidine rings are almost coplanar. The packing of the molecules in the crystal structure is mainly due to intermolecular C—H...O hydrogen-bonding interactions, resulting in the formation of a zigzag chain.

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Comment

Derivatives of heterocycles containing the thienopyrimidine system, which are well known bioisosteres of quinazolines, are of great importance because of their remarkable biological properties (De Laszlo *et al.*, 1992; Taguchi *et al.*, 1993; Walter, 1999). The title compound, (I), may be used as a new precursor for obtaining bioactive molecules.



The structure of (I) is shown in Fig. 1. All atoms within the thieno[2,3-d]pyrimidine system are essentially coplanar. The bond lengths and angles are within experimental error, in the ranges of values in previously reported structures in the Cambridge Structural Database (Version 5.26; Allen, 2002).

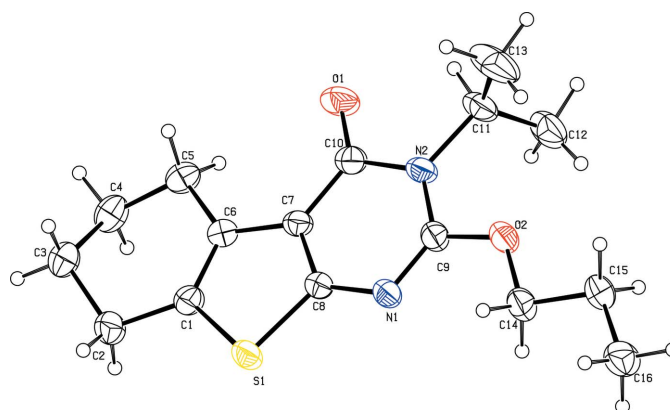


Figure 1
 A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

There is a weak intermolecular C—H···O hydrogen bond (Table 1), resulting in the formation of a zigzag-like chain parallel to the *b* axis (Fig. 2).

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 (1:2 *v/v*, 20 ml) was added to precipitate triphenylphosphine oxide. After filtration, the solution of carbodiimide was added to a newly prepared solution of Na/PrOH (0.1 g in 2 ml). After stirring the reaction mixture for 28 h, the solvent was removed under reduced pressure and the residue was recrystallized from EtOH to give the title compound, (I), in a yield of 55% (m.p. 370 K). Spectroscopic analysis: ¹H NMR (CDCl₃, 400 MHz, δ , p.p.m.): 5.45 (*m*, 1H, CH), 4.43–4.40 (*t*, *J* = 6.6 Hz, 2H, OCH₂), 2.96–2.70 (*m*, 4H, 2CH₂), 1.86–1.65 (*m*, 8H, 4CH₂), 1.46–1.45 (*d*, *J* = 6.8 Hz, 6H, 2CH₃), 1.02–0.98 (*t*, *J* = 7.4 Hz, 3H, CH₃); MS (*m/z*, %): 306 (*M*⁺, 100), 264 (57), 222 (99), 194 (27), 179 (88). Elemental analysis, calculated for C₁₆H₂₂N₂O₂S: C 62.71, H 7.24, N 9.14%; found: C 62.54, H 7.17, N 9.02%. Crystals of (I) suitable for X-ray diffraction were obtained from a solution in hexane and dichloromethane (1:3 *v/v*) at room temperature.

Crystal data

C ₁₆ H ₂₂ N ₂ O ₂ S	$D_x = 1.273 \text{ Mg m}^{-3}$
$M_r = 306.42$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2182 reflections
$a = 10.1586$ (9) Å	$\theta = 2.3\text{--}22.3^\circ$
$b = 14.4156$ (13) Å	$\mu = 0.21 \text{ mm}^{-1}$
$c = 11.9245$ (11) Å	$T = 292$ (2) K
$\beta = 113.675$ (2)°	Block, colourless
$V = 1599.3$ (3) Å ³	$0.30 \times 0.24 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2208 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.052$
Absorption correction: none	$\theta_{\text{max}} = 27.0^\circ$
9659 measured reflections	$h = -12 \rightarrow 12$
3476 independent reflections	$k = -15 \rightarrow 18$
	$l = -13 \rightarrow 15$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2]$
$wR(F^2) = 0.126$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.92$	$(\Delta/\sigma)_{\text{max}} = 0.001$
3476 reflections	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
193 parameters	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C16—H16C···O1	0.96	2.56	3.520 (3)	175

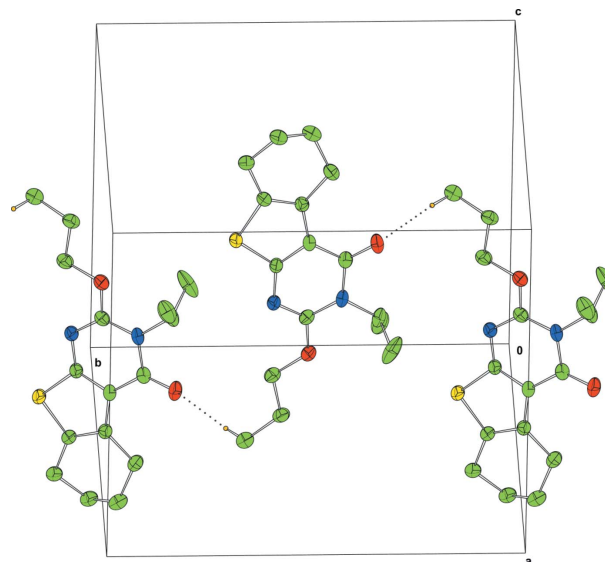


Figure 2

A partial packing view of the crystal structure of (I), showing the C—H···O hydrogen bonds (dotted lines) and the formation of the zigzag-like chain. H atoms not involved in hydrogen bonding have been omitted for clarity.

H atoms were placed in calculated positions and treated as riding on their parent C atoms, with C—H = 0.97 (C_{methylene}) or 0.98 Å (C_{methyl}) and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}_{\text{methylene}})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *SHELXTL*.

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