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3-*n*-Butyl-2-methoxy-1-benzothieno[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) = 0.007 Å
Disorder in main residue
R factor = 0.067
wR factor = 0.175
Data-to-parameter ratio = 13.6

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

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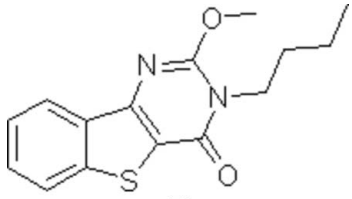
The title compound, $C_{15}H_{16}N_2O_2S$, contains a five-membered thiophene ring fused to a benzene ring and a substituted pyrimidinone ring. All three rings in each of the independent molecules of the asymmetric unit lie in approximately the same plane. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonding and π–π stacking interactions.

Comment

The derivatives of heterocycles containing a thienopyrimidine system, which are well known bioisosteres of quinazolines, are of great importance because of their remarkable biological properties. We have recently focused on the synthesis of fused heterocyclic systems containing thienopyrimidine *via* an aza-Wittig reaction at room temperature (Ding *et al.*, 2004). We present here the structure of one such thienopyrimidine derivative, the title compound, (I) (Fig. 1).



(I)

Compound (I) has two unique molecules in the asymmetric unit of the monoclinic unit cell. Molecule 1 is numbered with atoms C1–C15, S1, O1, O2, N1 and N2, while molecule 2 has atoms C16–C30, S2, O3, O4, N3 and N4. Compound (I) contains a five-membered thiophene ring fused to a benzene ring and a substituted pyrimidinone ring. All three rings lie in approximately the same plane, with a mean deviation of 0.0061 Å for molecule 1 and 0.0124 Å for molecule 2.

The structure of (I) is stabilized by intermolecular C—H···O hydrogen bonding (Table 1). For molecule 1, the distances between the centroids of the thiophene and pyrimidine rings, and those of the pyrimidine and benzene rings, related in each case by the symmetry operator $(1-x, 2-y, 2-z)$, are 3.795 (2) and 3.727 (2) Å, respectively. The dihedral angles between these pairs of rings in adjacent molecules are 0.52 (1) and 0.18 (1)°, respectively. For molecule 2, the distance between the pyrimidine and benzene ring centroids related by symmetry operator $(-x, 1-y, -z)$ is 3.692 (2) Å, while the dihedral angle between these rings is 0.85 (1)°. Hence, significant π–π stacking interactions occur between pairs of inversion-related molecules in the crystal.

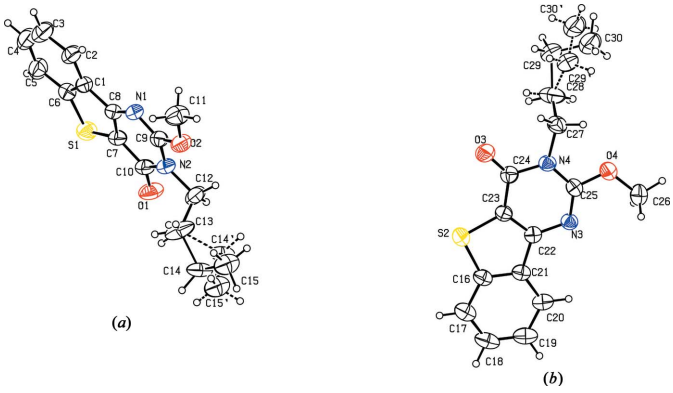


Figure 1
Views of the two unique molecules of (I), showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. (*a*) Molecule 1. (*b*) Molecule 2. Both disorder components are shown.

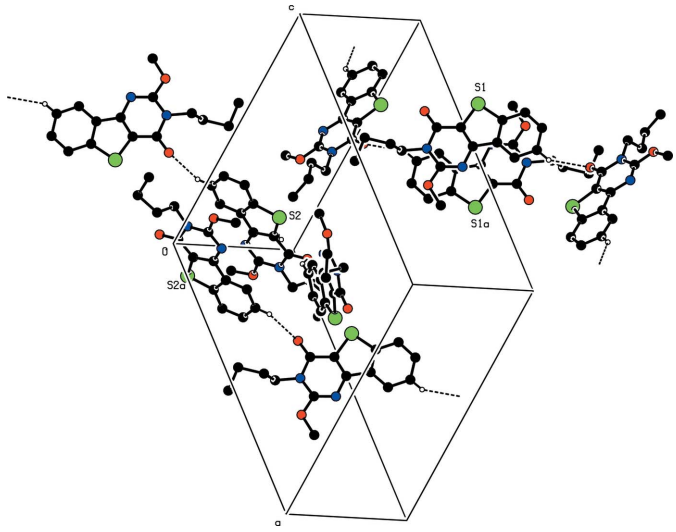


Figure 2
Part of the crystal structure of (I), showing the π–π stacking interactions. H atoms bound to C atoms have been omitted for clarity. [Symmetry codes: (*a*) $1-x, 2-y, 2-z$ for molecule 1 and $-x, 1-y, -z$ for molecule 2.] Dashed lines indicate hydrogen bonds. Only one disorder component is shown.

Experimental

To a solution of ethyl 3-triphenylphosphoranylideneamino-benzo[*b*]thiophen-2-carboxylate (3 mmol) in dry dichloromethane (5 ml) was added *n*-butyl isocyanate (3 mmol) under nitrogen at room temperature. After allowing the reaction mixture to stand for 8–12 h at 273–278k, the solvent was removed under reduced pressure and ether–petroleum ether (1:2, 12 ml) added in order to precipitate triphenylphosphine oxide. After filtration, anhydrous methanol (10 ml) was added with several drops of CH_3ONa in CH_3OH. The mixture was stirred for 6 h at room temperature. The solution was then concentrated under reduced pressure and the residue recrystallized from methanol to afford compound (I) (yield 66%; m.p. 368 K). Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz, δ, p.p.m.): 8.23–7.47 (*m*, 4H, Ar-H), 4.19–4.12 (*m*, 2H, N—CH_2), 4.18 (*s*, 3H, O—CH_3), 1.72–1.66 (*m*, 2H, CH_2), 1.44–1.38 (*m*, 2H, CH_2), 0.98–0.95 (*s*, 3H, 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
Monoclinic, $P2_1/c$
a = 18.1842 (17) Å
b = 11.0507 (11) Å
c = 16.2682 (16) Å
β = 115.905 (2)°
V = 2940.6 (5) Å^3
Z = 8
D_x = 1.303 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1656 reflections
θ = 2.2–18.2°
μ = 0.22 mm^{-1}
T = 292 (2) K
Plate, colourless
0.30 × 0.30 × 0.08 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
14542 measured reflections
5177 independent reflections
2878 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 25.0°
$h = -12 \rightarrow 21$
$k = -13 \rightarrow 13$
$l = -19 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.175
S = 1.02
5177 reflections
381 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 0.6565P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C19—H19···O1^{i}	0.93	2.43	3.205 (5)	141
C3—H3···O3^{ii}	0.93	2.57	3.310 (5)	137

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

H atoms were positioned geometrically and treated as riding atoms, with C—H distances of 0.93 (aromatic), 0.96 (CH_3), 0.97 (CH_2) and 0.98 Å (CH), and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for the methyl groups. The *n*-butyl groups in both molecules showed positional disorder. The final occupancies for the major and minor disorder components are 0.53 (1) and 0.47 (1), respectively, in molecule 1, and 0.72 (1) and 0.28 (1), respectively, in molecule 2.

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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