

3-Benzyl-2-butylamino-5,6,7,8-tetrahydro-
benzothieno[2,3-*d*]pyrimidin-4(3*H*)-oneXiao-Hua Zeng, Hong-Mei
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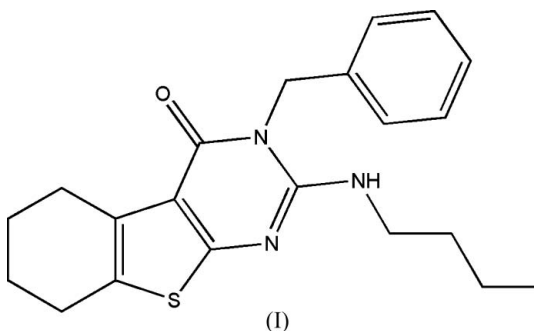
Key indicators

Single-crystal X-ray study
 $T = 292\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.058
 wR factor = 0.149
Data-to-parameter ratio = 17.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

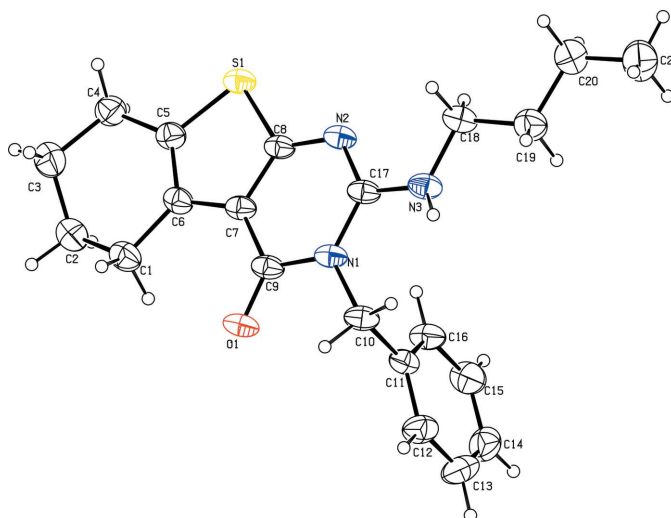
In the title molecule, $\text{C}_{21}\text{H}_{25}\text{N}_3\text{OS}$, the fused thiophene and pyrimidine rings are essentially coplanar. In the crystal structure, $\text{N}-\text{H}\cdots\text{O}$ and weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules, forming one-dimensional chains along [010].

Comment

Pyrimidine derivatives are currently attracting attention because of the important role played by such systems in many natural products and antibiotics (Ding *et al.*, 2004). We have been interested in the preparation of derivatives of heterocycles *via* the aza-Wittig reaction. We report here the synthesis and structure of the title compound, (I).



In the title molecule (Fig. 1), all ring atoms in the thieno[2,3-*d*]pyrimidine system are essentially coplanar, indicating that the thieno[2,3-*d*]pyrimidine system is conjugated. There exists

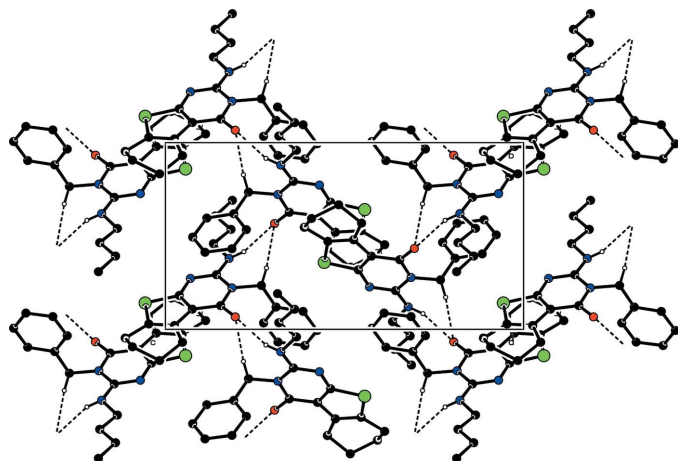
**Figure 1**

View of the molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are represented as spheres of arbitrary radii.

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**Figure 2**

Packing diagram of (I), showing intermolecular hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

a crossed angle between the benzene ring and the above mentioned plane, and no disorder of the molecule. In the crystal structure, N—H...O and weak C—H...O hydrogen bonds link the molecules, forming one-dimensional chains along [010] (Fig. 2 and Table 2).

Experimental

To a solution of iminophosphorane (1.45 g, 3 mmol) in anhydrous dichloromethane (15 ml), butyl isocyanate (3 mmol) was added under dry N₂ at room temperature. After the reaction mixture was left unstirred for 48 h at room temperature, 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 solvent was removed to give the carbodiimide, which was used directly without further purification. To a solution of the carbodiimide (15 ml), benzylamine (3 mmol) was added. After the mixture was stirred for 6 h, the solvent was removed and anhydrous ethanol (10 ml) containing several drops of EtONa in EtOH was added. The mixture was stirred for 12 h at room temperature. The solution was concentrated and the residue was recrystallized from ethanol to give the title compound (I), in a yield of 47% (m.p. 379 K). ¹H NMR (CDCl₃, 400 MHz): δ 7.36–7.24 (m, 5H, Ar—H), 5.25 (s, 2H, Ph—CH₂), 4.35 (br, 1H, NH), 3.32–3.30 (m, 2H, NCH₂), 2.99–2.69 (m, 4H, 2CH₂), 1.86–1.84 (m, 4H, 2CH₂), 1.39–1.35 (m, 2H, CH₂), 1.15–1.05 (m, 2H, CH₂), 0.82–0.78 (t, *J* = 7.4 Hz, 3H, CH₃); MS (*m/z*, %), 367 (*M*⁺, 7), 277 (2), 104 (3), 91 (100), 77 (3), 65 (17), 57 (7). Elemental analysis calculated for C₂₁H₂₅N₃OS: C 68.63, H 6.86, N 11.43%; found: C 68.71, H 7.93, N 11.34%. Crystals suitable for single-crystal X-ray diffraction were obtained from hexane and dichloromethane at room temperature.

Crystal data

C₂₁H₂₅N₃OS
M_r = 367.50
 Monoclinic, *P*2₁/*c*
a = 11.7759 (11) Å
b = 9.2747 (9) Å
c = 18.6871 (18) Å
 β = 107.760 (2)°
V = 1943.7 (3) Å³
Z = 4

D_x = 1.256 Mg m^{−3}
 Mo *K*α radiation
 Cell parameters from 1789 reflections
 θ = 2.3–22.4°
 μ = 0.18 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} = 0.948, *T_{max}* = 0.982
 11061 measured reflections

4192 independent reflections
 2809 reflections with *I* > 2σ(*I*)
R_{int} = 0.039
 θ_{\max} = 27.0°
h = −13 → 15
k = −11 → 11
l = −23 → 21

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.058
wR (*F*²) = 0.149
S = 1.04
 4192 reflections
 240 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2 + 0.0324P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C5—C6	1.351 (3)	C8—S1	1.734 (2)
C5—S1	1.742 (2)	C9—O1	1.233 (2)
C6—C7	1.436 (3)	C9—N1	1.406 (3)
C7—C8	1.390 (3)	C17—N2	1.314 (3)
C7—C9	1.422 (3)	C17—N1	1.392 (3)
C8—N2	1.348 (3)		
C8—S1—C5	91.56 (10)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C10—H10B...O1 ⁱ	0.97	2.40	3.370 (3)	174
N3—H3...O1 ⁱ	0.84 (1)	2.12 (1)	2.928 (2)	162 (2)

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

The H atom bonded to N3 was refined isotropically. All other H atoms were placed in calculated positions and treated as riding atoms, with C—H = 0.93–0.97 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) or 1.5*U*_{eq}(methyl C).

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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