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5-Benzylamino-6-phenyl-2-propylsulfanyl-thiazolo[5,4-*d*]pyrimidin-7(6*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 = 0.066
wR factor = 0.183
Data-to-parameter ratio = 13.9

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

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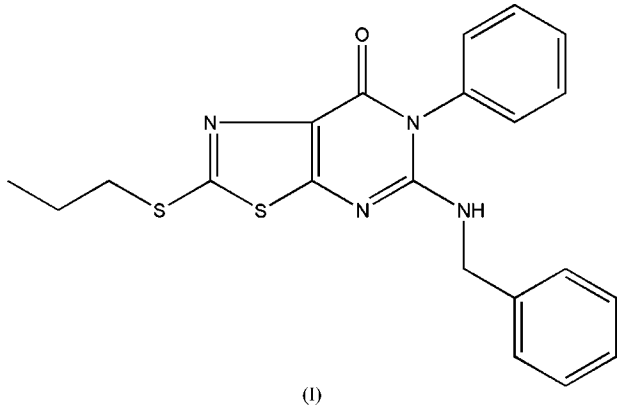
In the title compound, $C_{21}H_{20}N_4OS_2$, the two rings of the fused thiazolo[5,4-*d*]pyrimidine system are almost coplanar. In the crystal structure, hydrogen-bonded tetramers are formed *via* N—H···N hydrogen bonds.

Comment

Thiazolo[5,4-*d*]pyrimidines are purine analogue derivatives and they have potentially useful biological properties (El-Bayouki & Basyouni, 1988). In recent years, we have been engaged in the synthesis of derivatives of various heterocycles *via* the aza-Wittig reaction (Ding, Yang & Zhu, 2004; Ding, Chen & Huang, 2004). The title compound, (I), may be used as a new precursor for obtaining bioactive molecules and we report here the X-ray crystallographic analysis of this compound.



(I)

The molecular structure of the title compound is shown in Fig. 1, and selected bond distances and angles are given in Table 1. All the ring atoms in the thiazolo[5,4-*d*]pyrimidine system are essentially coplanar. The *n*-propylthio group is disordered [occupancies 0.583 (6)/0.417 (6); unprimed/primed atoms; Fig. 1].

In the crystal structure of (I), symmetry-related molecules are linked *via* N—H···N hydrogen bonds to form tetramers. Details are given in Table 2 and Fig. 2.

Experimental

Compound (I) was prepared by adding benzylamine (0.22 g) to a solution of ethyl 5-[(phenylimino)methyleneamino]-2-(propylthio)-thiazole-4-carboxylate (2 mmol) in dry dichloromethane (10 ml). The solution was stirred for 2 h at 298 K. The solvent was then removed and anhydrous ethanol (10 ml), containing several drops of EtONa in EtOH, was added. The mixture was stirred for 6 h at room temperature. The solution was then concentrated under reduced

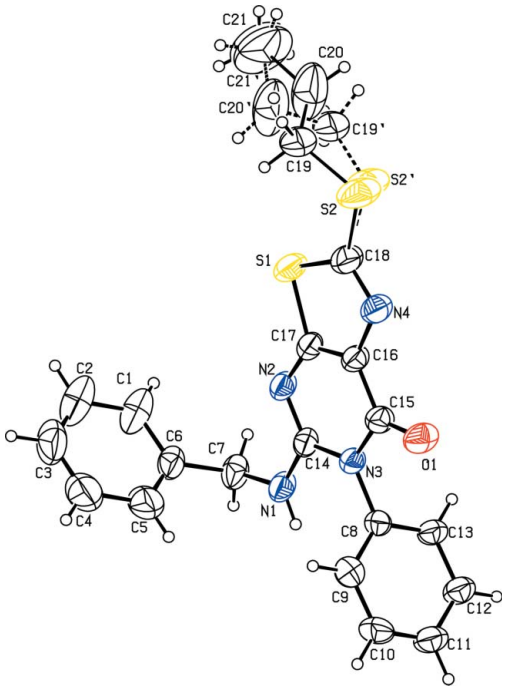


Figure 1
View of the molecular structure of (I), showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level. Both disorder components are shown.

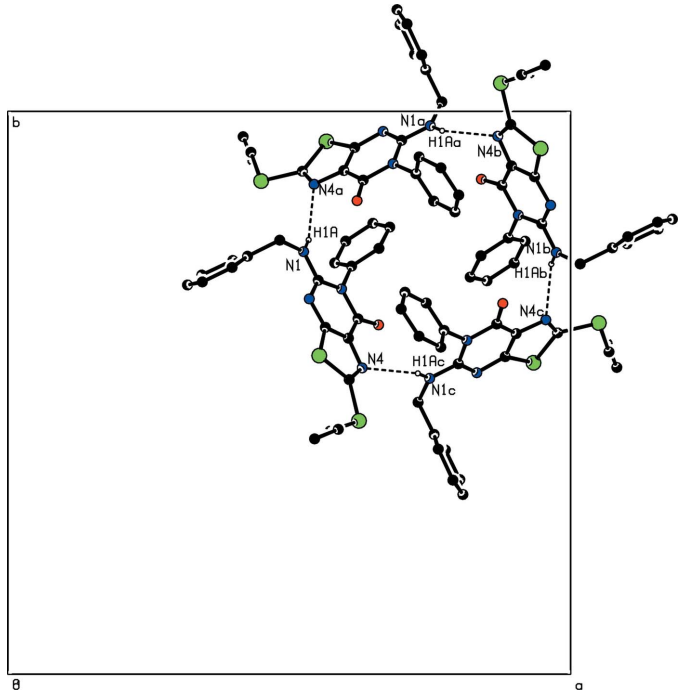


Figure 2
A partial view of the crystal packing of (I), showing the formation of an N—H···N hydrogen-bonded tetramer. Hydrogen bonds are shown as dashed lines and atoms labelled a, b and c correspond to symmetry operations ($y, \frac{3}{2} - x, \frac{3}{2} - z$), ($\frac{3}{2} - x, \frac{3}{2} - y, z$) and ($\frac{3}{2} - y, x, \frac{3}{2} - z$), respectively. H atoms not involved in hydrogen bonds have been omitted.

pressure and the residue was recrystallized from ethanol to give compound (I), in a yield of 85% (m.p. 460 K). Suitable crystals were obtained by vapour diffusion of ethanol and dichloromethane at room temperature. ^{1}HNMR ($CDCl_3$, 400 MHz): 7.17–7.58 (*m*, 10H, Ph—H), 4.57–4.59 (*m*, 3H, N1—H, C7—H), 3.26–3.3(*t*, 2H, C19—H), 1.78–1.82 (*m*, 2H, C20—H), 1.03–1.06 (*t*, 3H, C21—H).

Crystal data

$C_{21}H_{20}N_4OS_2$
M_r = 408.53
Tetragonal, $P4_2/n$
a = 23.3140 (6) Å
c = 7.7925 (4) Å
V = 4235.6 (3) Å^3
Z = 8
D_x = 1.281 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2882 reflections
θ = 2.5–16.9°
μ = 0.27 mm^{-1}
T = 292 (2) K
Plate, colourless
0.30 × 0.20 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
29639 measured reflections
3737 independent reflections
2138 reflections with $I > 2\sigma(I)$
R_{int} = 0.080
θ_{max} = 25.0°
$h = -27 \rightarrow 27$
$k = -27 \rightarrow 27$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.183
S = 1.04
3737 reflections
268 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 1.9388P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ <0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C17	1.739 (4)	N1—C14	1.345 (4)
S1—C18	1.758 (5)	N2—C17	1.353 (5)
S2—C18	1.798 (8)	N2—C14	1.309 (5)
S2—C19	1.776 (12)	N3—C15	1.413 (5)
S2′—C19′	1.714 (17)	N3—C8	1.447 (4)
S2′—C18	1.740 (11)	N3—C14	1.394 (5)
O1—C15	1.223 (5)	N4—C16	1.387 (5)
N1—C7	1.442 (5)	N4—C18	1.281 (6)
C17—S1—C18	88.2 (2)	O1—C15—N3	119.9 (3)
C18—S2—C19	102.8 (5)	O1—C15—C16	127.6 (3)
C18—S2′—C19′	115.0 (8)	N3—C15—C16	112.5 (3)
C7—N1—C14	121.1 (3)	N4—C16—C15	123.7 (3)
C14—N2—C17	113.6 (3)	N4—C16—C17	116.7 (3)
C8—N3—C14	120.3 (3)	S1—C17—C16	109.4 (3)
C8—N3—C15	116.9 (3)	N2—C17—C16	127.7 (4)
C14—N3—C15	122.8 (3)	S1—C17—N2	122.9 (3)
C16—N4—C18	109.9 (3)	S1—C18—S2′	117.0 (5)
N1—C7—C6	114.2 (3)	S1—C18—N4	115.9 (3)
N3—C8—C9	121.3 (3)	S2′—C18—N4	125.9 (5)
N3—C8—C13	118.6 (3)	S1—C18—S2	127.3 (4)
N2—C14—N3	123.8 (3)	S2—C18—N4	116.0 (4)
N1—C14—N3	116.9 (3)	S2—C19—C20	121.2 (8)
N1—C14—N2	119.3 (3)	S2′—C19′—C20′	112.7 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N4^{i}	0.86	2.38	3.069 (4)	138

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

The H atoms were placed in calculated positions and treated as riding atoms, with C—H = 0.93–0.97 Å, N—H = 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(parent C or N atom) and 1.5U_{eq}(methyl C atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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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