Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(*tert*-Butylamino)-3-phenylbenzo[4,5]thieno[3,2-*d*]pyrimidin-4(3*H*)-one

Sheng-Zhen Xu[a] and Yang-Gen Hu[b,a]*

[a]Key Laboratory of Pesticides and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China, and [b]Department of Medicinal Chemistry, Yunyang Medical College, Shiyan 442000, People's Republic of China

Correspondence e-mail: huyangg111@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
R factor = 0.054
wR factor = 0.149
Data-to-parameter ratio = 13.4

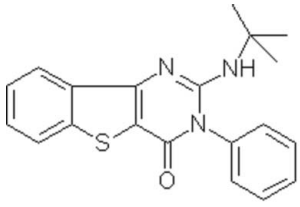
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 October 2006
Accepted 12 November 2006

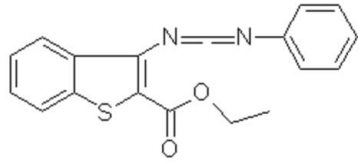
In the title compound, $C_{20}H_{19}N_3OS$, the three fused rings of the benzo[4,5]thieno[3,2-*d*]pyrimidone system are essentially coplanar. The crystal packing is mainly stabilized by C—H···π and π–π interactions.

Comment

Thienopyrimidine derivatives are of great importance because of their remarkable biological properties (Ding *et al.*, 2004). We have recently been engaged in the preparation of heterocyclic derivatives containing a fused pyrimidone unit using the aza-Wittig reaction (Cao *et al.*, 2006; Hu, Li *et al.*, 2005; Hu, Xu *et al.*, 2005; Hu *et al.*, 2006). We present here the structure of one such thienopyrimidine derivative, (I) (Fig. 1).



(I)



(II)

The three fused rings of (I) are essentially coplanar, the maximum deviation being 0.050 (3) Å for atom C8. The phenyl ring C11–C16 is twisted with respect to the benzo[4,5]thieno[3,2-*e*]pyrimidinone ring system, making a dihedral angle of 70.5 (1)°.

A π–π interaction (Janiak, 2000) between the pyrimidine ring and the benzene ring C1–C6 at (1 − *x*, −*y*, 2 − *z*) [centroid-to-centroid distance of 3.682 (2) Å] and an intermolecular C—H···π interaction (Table 1; *Cg* is the centroid of the pyrimidine ring) are effective in stabilizing the crystal structure of (I). There are also weak intramolecular C—H···N hydrogen bonds (Table 1).

Experimental

To a solution of ethyl 3-triphenylphosphoranylideneamino-benzo[4,5]thiophene-2-carboxylate (3 mmol) in dry dichloromethane (5 ml) was added phenyl isocyanate (3 mmol) under nitrogen at room temperature. After allowing the reaction mixture to stand for 10 h at 273–278 K, the solvent was removed under reduced pressure and ether–petroleum ether (1:2 *v*/*v*, 12 ml) was added to precipitate triphenylphosphine oxide. After filtration, the solvent was removed to give ethyl 3-(phenyliminomethyleneamino)benzo[*b*]thiophene-2-carboxylate, (II), which was used directly without further purification. To a solution of (II) (15 ml) in dichloromethane (15 ml) was added *tert*-butylamine (3 mmol). After allowing the reaction mixture to

stand for 4 h, the solvent was removed and anhydrous ethanol (10 ml) with several drops of EtONa in EtOH was added. The mixture was stirred for 3 h at room temperature. The solution was concentrated under reduced pressure and the residue was recrystallized from ethanol to give the title compound, (I), in a yield of 63%. Suitable crystals were obtained by vapour diffusion of ethanol into dichloromethane at room temperature.

Crystal data

$C_{20}H_{19}N_3OS$
M_r = 349.44
Triclinic, $P\overline{1}$
a = 9.7106 (15) Å
b = 10.1139 (15) Å
c = 10.5568 (16) Å
α = 104.928 (3)°
β = 115.988 (2)°
γ = 91.557 (3)°
V = 888.7 (2) Å^3
Z = 2
D_x = 1.306 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 292 (2) K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.962, T_{max} = 0.981
4495 measured reflections
3058 independent reflections
2179 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.149
S = 1.04
3058 reflections
229 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0723P)^2 + 0.077P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 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—H19C···N1	0.96	2.57	3.163 (4)	120
C18—H18A···N1	0.96	2.43	2.986 (4)	117
C18—H18A···Cg^i	0.96	2.71	3.454 (4)	135

Symmetry code: (i) $-x+1, -y+1, -z+2$. Cg is the centroid of the pyrimidine ring.

H atoms were located in a difference Fourier map and then treated as riding, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{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* (Bruker, 2001).

The authors gratefully acknowledge financial support of this work by the Science Research Project of Yunyang Medical College (grant No. 2006QDJ16).

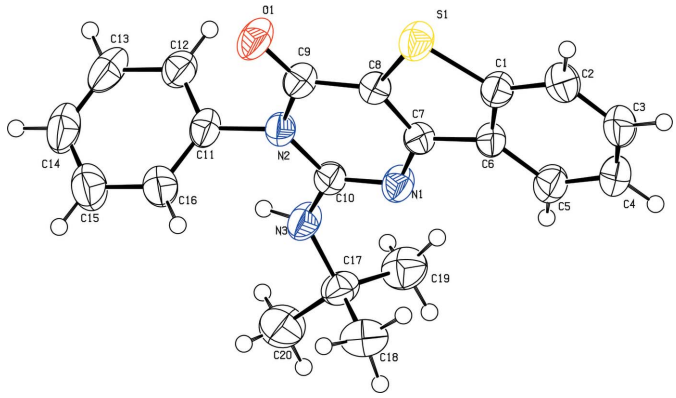


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

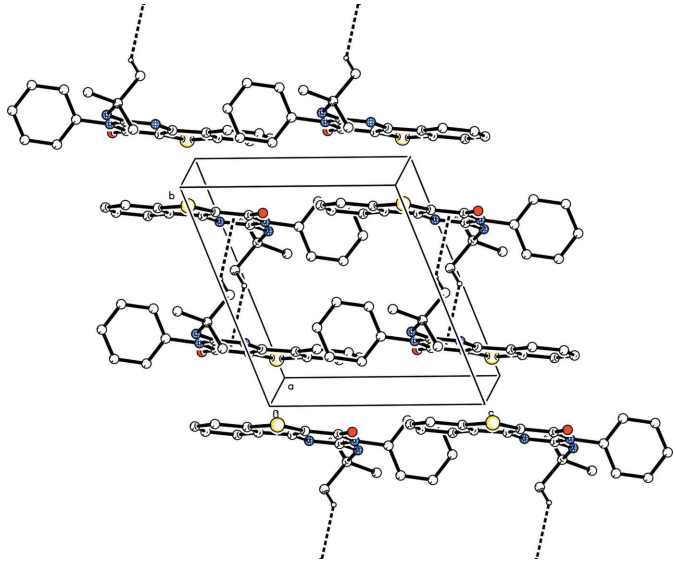


Figure 2
A partial packing diagram of (I), showing the π–π stacking and C—H···π interactions. The C—H···π interactions are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted.

References

Bruker (2001). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cao, M.-H., Xu, S.-Z. & Hu, Y.-G. (2006). *Acta Cryst.* E**62**, o1319–o1320.
Ding, M.-W., Xu, S.-Z. & Zhao, J.-F. (2004). *J. Org. Chem.* **69**, 8366–8371.
Hu, Y.-G., Li, G.-H., Tian, J.-H., Ding, M.-W. & He, H.-W. (2005). *Acta Cryst.* E**61**, o3266–o3268.
Hu, Y.-G., Xu, S.-Z., Yuan, J.-Z., Ding, M.-W. & He, H.-W. (2005). *Acta Cryst.* E**61**, o2649–o2651.
Hu, Y.-G., Zheng, A.-H. & Li, G.-H. (2006). *Acta Cryst.* E**62**, o1457–o1459.
Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3885–3896.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.