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9-(2-Phenylethyl)-6-(2-thienyl)-9*H*-purine

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In the title compound [alternative name: 9-benzyl-6-(2-thienyl)-9*H*-purine], $C_{16}H_{12}N_4S$, the purine system is not strictly planar and the dihedral angle between the fused rings is 1.46°. The packing of the molecules is influenced by hydrophobic and hydrophilic interactions.

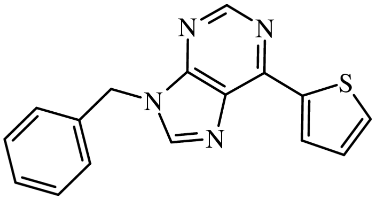
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.130
Data-to-parameter ratio = 14.4

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

Comment

Tuberculosis (TB) is the major cause of death, from a single infectious agent, among adults in developing countries and there has been an unfortunate revival of TB in the industrialized world. There are *ca* 8 million new cases of TB each year and it has been estimated that *ca* 30 million people will die from tuberculosis within about 10 years (Duncan, 1997). Thus, there is an urgent need for new antimycobacterial agents and various 9-benzylpurines, including the title compound, (I), have shown high inhibitory activity against *Mycobacterium tuberculosis* (Bakkestuen *et al.*, 2000). Hence, we undertook the present study to examine the effect of the thiophene substituent at the 6-position.



(I)

The molecular geometry is illustrated in Fig. 1 and the bond lengths and angles are listed in Table 1. The values are in agreement with those found in other structures containing the purine ring system (Stewart & Jensen, 1964; Lai & Marsh, 1972; Wilson *et al.*, 1986; Low *et al.*, 1987; Flensburg, 1994).

The benzene ring is inclined at an angle of 64.03 (5)° to the purine ring system and at an angle of 60.23 (6)° to the thiophene ring at the 6-position. The thiophene ring is nearly coplanar with the purine ring, the dihedral angle being only 6.39 (4)°. The base is not exactly planar; the two rings are rotated by 1.46 (6)° about the C4—C5 bond.

The bond distance N9—C10 [1.469 (2) Å] is longer than that found in the case of adenin-9-ylacetate [1.451 (1) Å] and the C8—N9—C10—C11 torsion angle of −94.6 (2)° also differs from the value of 104.65 (13)° reported by Flensburg (1994). The bond distances and angles in the thiophene ring are, by and large, normal. The bond angles C5—C6—C17 [123.89 (15)°] and N1—C6—C17 [118.13 (14)°] are compar-

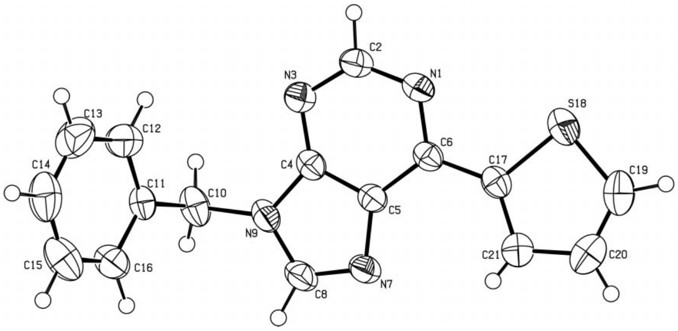


Figure 1
Plot showing the atomic numbering scheme for (I). Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

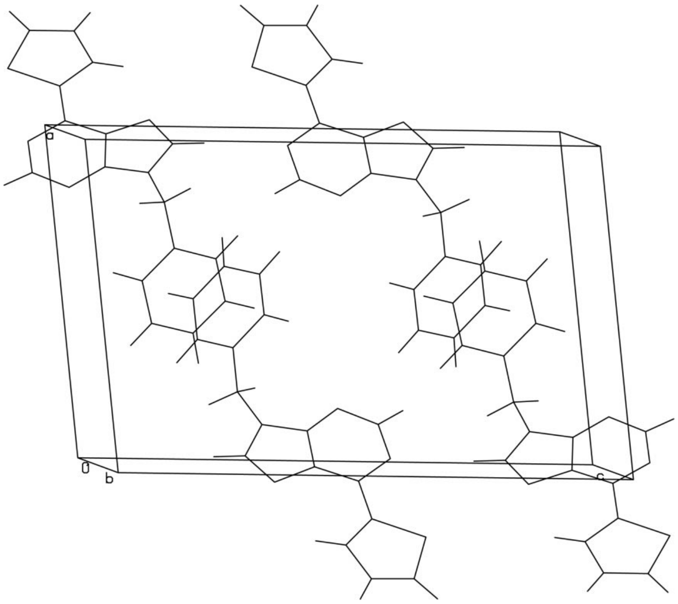


Figure 2
A view of the packing of the molecules in the unit cell.

able to corresponding angles of 124.21 (10)° and 118.29 (10)° in the case of NH_2 substitution at the 6-position, indicating that the thiophene ring has no great effect on the bond angles.

The N1—C6 bond distance of 1.352 (2) Å is slightly greater than a C═N distance of 1.282 (2) Å in 2-[(4-hydroxyphenyl)iminomethyl]thiophene (Kazak *et al.*, 2000) due to the aromaticity of the purine ring system. However, the C6—C17 bond distance of 1.449 (2) Å is not affected.

As is evident from Fig. 2, a hydrophobic region is created due to interactions between the benzene rings. The molecular packing is stabilized by segregation of alternate hydrophobic and hydrophilic zones.

Experimental

The title compound was synthesized as described by Gundersen *et al.* (1994). Crystals suitable for X-ray diffraction studies were obtained by slow evaporation at room temperature from a mixture of ethanol, acetone and chloroform (1:1:1).

Crystal data

$C_{16}H_{12}N_4S$
$M_r = 292.36$
Monoclinic, $P2_1/c$
$a = 10.1130$ (4) Å
$b = 8.9101$ (2) Å
$c = 15.6434$ (7) Å
$\beta = 96.8550$ (17)°
$V = 1399.52$ (9) Å^3
$Z = 4$

$D_x = 1.388$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7690 reflections
$\theta = 3.2$–28°
$\mu = 0.23$ mm^{-1}
$T = 293$ (2) K
Trigonal prism, pale yellow
0.48 × 0.43 × 0.12 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
7690 measured reflections
3364 independent reflections
2745 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.024$
$\theta_{\rm max} = 28.0°$
$h = -14 \rightarrow 14$
$k = -11 \rightarrow 12$
$l = 0 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.130$
$S = 1.09$
3364 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.536P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.26$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.45$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C6—C17	1.449 (2)	C17—S18	1.7164 (17)
N9—C10	1.469 (2)	S18—C19	1.700 (2)
C10—C11	1.504 (2)	C19—C20	1.339 (3)
C17—C21	1.391 (3)	C20—C21	1.411 (3)
N1—C6—C17	118.13 (14)	C21—C17—C6	128.41 (15)
C5—C6—C17	123.89 (15)	C21—C17—S18	110.91 (13)
N9—C10—C11	112.85 (14)	C6—C17—S18	120.67 (13)
N7—C5—C6—N1	−179.02 (16)	N9—C10—C11—C16	88.9 (2)
N7—C5—C6—C17	−0.4 (3)	N1—C6—C17—C21	−175.69 (16)
C4—C5—C6—C17	177.47 (14)	C5—C6—C17—C21	5.7 (3)
C8—N9—C10—C11	−94.6 (2)	N1—C6—C17—S18	5.0 (2)
C4—N9—C10—C11	88.3 (2)	C5—C6—C17—S18	−173.64 (12)
N9—C10—C11—C12	−91.0 (2)		

All H atoms, except for H21, were located from a difference Fourier synthesis and refined isotropically. H21, attached to C21, was positioned geometrically and not refined. Bond distances to H atoms range from 0.90 to 1.00 Å.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.

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