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2-Diethylamino-3-(4-methylphenyl)-1-benzothieno[3,2-*d*]pyrimidin-4(3*H*)-one

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Key indicators

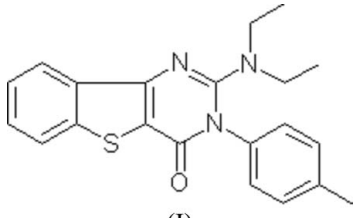
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.130
Data-to-parameter ratio = 18.1

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

In the title compound, $C_{21}H_{21}N_3OS$, the three fused rings of the 1-benzothieno[3,2-*d*]pyrimidine system are almost coplanar. The crystal packing is stabilized by π–π stacking interactions and intermolecular C—H···O hydrogen bonds.

Comment

Heterocyclic compounds containing a fused pyrimidinone system have various applications in agriculture and exhibit remarkable biological activity (Ding *et al.*, 2004). We have recently focused on the synthesis of heterocyclic compounds containing a fused pyrimidinone system using an aza-Wittig reaction. The X-ray crystal structures of some thieno[3,2-*d*]pyrimidine derivatives have been reported (Xu *et al.*, 2005; Xu, Hu, Liu *et al.*, 2006; Xu, Hu, Wang *et al.*, 2006; Zheng *et al.*, 2006). We present here the crystal structure of the title compound, (I) (Fig. 1), which can be used as a precursor for obtaining bioactive molecules.



(I)

In (I), the bond lengths and angles are unexceptional. The mean planes of the benzothienopyrimidine ring system [maximum deviation of 0.074 (2) Å for atom C9] and benzene ring C10–C15 make a dihedral angle of 82.16 (6)°. The crystal structure (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1) and by π–π stacking interactions with centroid–centroid separations of 3.801 (1), 3.787 (1), 3.600 (1) and 3.824 (1) Å for $Cg1\cdots Cg1^{i}$, $Cg2\cdots Cg1^{i}$, $Cg1\cdots Cg3^{i}$ and $Cg3\cdots Cg3^{i}$, respectively, where *Cg*1, *Cg*2 and *Cg*3 are the centroids of rings S1/C1/C6–C8, N1/C7–C9/N2/C17 and C1–C6, respectively [symmetry code: (i) $x, \frac{3}{2}-y, \frac{1}{2}+z$].

Experimental

3-[(4-Methylphenyl)iminomethyleneamino]benzothiophene-2-carboxylic acid, (II), was prepared according to Xu, Hu, Liu *et al.* (2006) and Xu, Hu, Wang *et al.* (2006). To a solution of (II) (3 mmol) in dichloromethane (15 ml) diethylamine (3 mmol) was added. After the reaction mixture was allowed to stand for 1 h, the solvent was removed and anhydrous ethanol (10 ml) and several drops of EtONa in EtOH were added. The mixture was stirred for 2 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 76%. Crystals suitable for X-ray diffraction were obtained by evaporation of a solution in ethanol and dichloromethane (1:2 *v*/*v*) at room temperature.

Crystal data

$C_{21}H_{21}N_3OS$
M_r = 363.47
Monoclinic, $P2_1/c$
a = 19.718 (3) Å
b = 12.7157 (17) Å
c = 7.5952 (10) Å
β = 99.419 (2)°
V = 1878.7 (4) Å^3
Z = 4
D_x = 1.285 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 292 (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.946, T_{max} = 0.964
16349 measured reflections
4306 independent reflections
3218 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.130
S = 0.96
4306 reflections
238 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O1^{i}	0.93	2.35	3.276 (2)	172
C15—H15···O1^{ii}	0.93	2.49	3.335 (2)	150

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

The H atoms were positioned geometrically [C—H = 0.93 (CH), 0.97 (CH_2) and 0.96 Å (CH_3)] and constrained to ride on their parent atoms, with U_{iso}(H)= 1.2 (1.5 for methyl) times U_{eq}(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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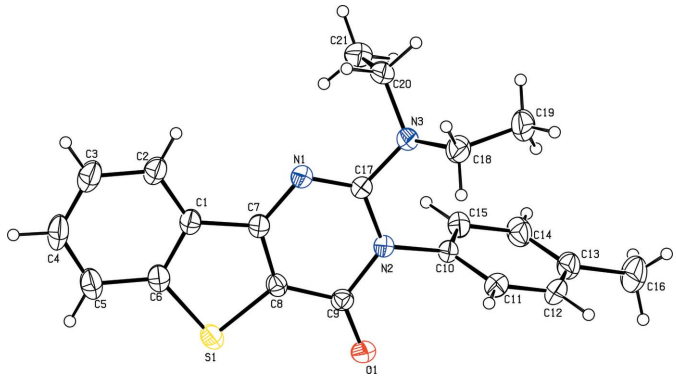

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

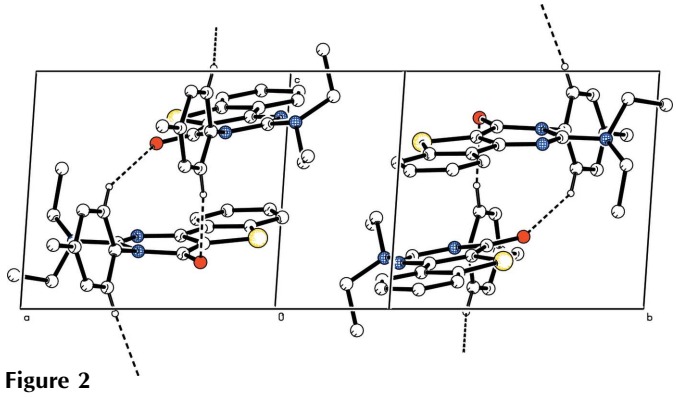

Figure 2
Part of the crystal structure of (I), showing the C—H···O hydrogen-bonding interactions as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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