Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Ming-Guo Liu,[a]* Ju-Zhen Yuan,[b] Yang-Gen Hu[c] and Sheng-Zhen Xu[b]

[a]College of Chemistry and Life Science, China Three Gorges University, YiChang 443002, People's Republic of China, [b]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 [c]Department of Medicinal Chemistry, Yunyang Medical College, Shiyan 442000, People's Republic of China

Correspondence e-mail: mgliu0427@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
R factor = 0.060
wR factor = 0.145
Data-to-parameter ratio = 15.1

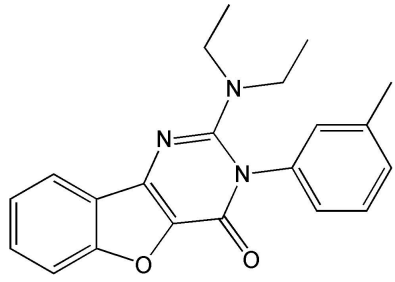
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 28 November 2005
Accepted 5 December 2005
Online 10 December 2005

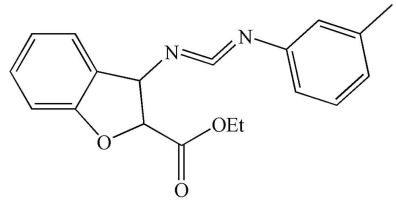
In the title compound, $C_{21}H_{21}N_3O_2$, the three fused rings of the 1-benzofuro[3,2-*d*]pyrimidine system are almost coplanar. The packing of the molecules in the crystal structure is mainly governed by π–π interactions.

Comment

The derivatives of benzofuropyrimidines are of great importance because of their remarkable biological properties (Bodke & Sangapure, 2003). In recent years, we have been engaged in the preparation of derivatives of heterocycles *via* an aza-Wittig reaction (Ding *et al.*, 2004*a*,*b*). The heterocyclic title compound, (I), may be used as a new precursor for obtaining bioactive molecules and its structure is presented here (Fig. 1). The three fused rings of the benzofuro[3,2-*d*]pyrimidine system are almost coplanar, with a maximum deviation of 0.079 (2) Å for C9. This plane is at an angle of 63.79 (11)° to the substituted benzene ring. Bond lengths and angles (Table 1) are in agreement with reported literature values (Allen *et al.*,1987).



(I)



(II)

The centroid-to-centroid distances are 3.5173 (2) for rings *A* (O1, C1 C6, C7, C8) and B^{i} (C1, C2, C3, C4, C5, C6) [symmetry code: (i) = $2-x, 2-y, 2-z$] and 3.6726 (3) Å for rings *B* and A^{ii} [symmetry code: (ii) = $2-x, 1-y, 2-z$]. The corresponding dihedral angles are 0.03 (2) and 1.26 (2)°, respectively. The contribution of π–π stacking interactions to the stability of the crystal structure is further demonstrated by the angles between the ring-centroid vectors [7.84 (2) for *A* to B^{i}

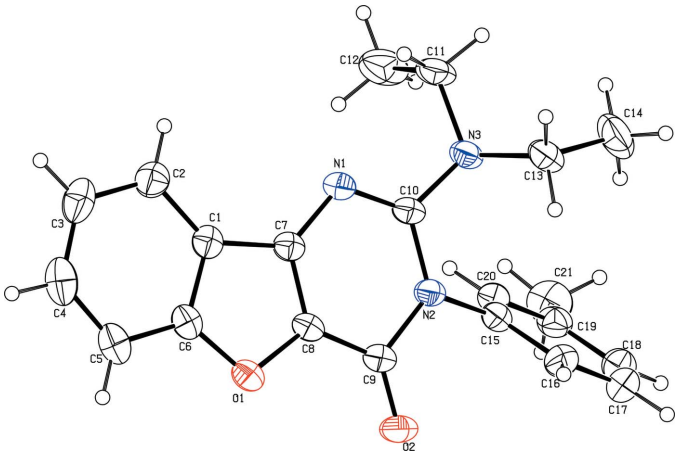


Figure 1
View of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H-atoms are represented by circles of arbitrary size.

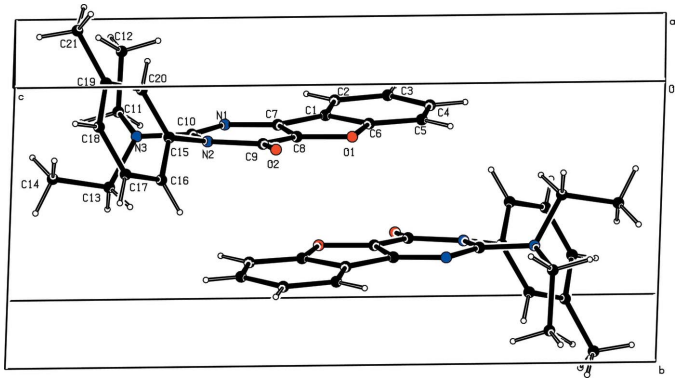


Figure 2
Packing diagram for (I), showing the π–π stacking interactions.

and 20.42 (3)° for *B* to A^{ii}] and the angles between the ring normals [8.58 (3) and 20.87 (2)° (Janiak, 2000)].

Experimental

To a solution of iminophosphorane (1.40 g, 3 mmol) in dry dichloromethane (15 ml) was added phenyl isocyanate (3 mmol) under nitrogen at room temperature. After standing for 10 h at 273–278 K, the solvent was removed under reduced pressure and diethyl ether/petroleum ether (1:2, 20 ml) was added to precipitate triphenylphosphine oxide. After filtration the solvent was removed to give the carbodiimide, (II), which was used directly without further purification. To the solution of (II) prepared above in dichloromethane (15 ml) was added diethylamine (3 mmol). After the reaction mixture was allowed to stand for 0.5 h, the solvent was removed and anhydrous ethanol (10 ml) and several drops of EtONa in EtOH were added. The mixture was stirred for 3 h at room temperature, concentrated under reduced pressure and the residue recrystallized from ethanol to give the title compound (I) (yield 0.81 g, 78%, m.p. 420 K). Suitable crystals were obtained by vapor diffusion of ethanol and dichloromethane at room temperature. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz): 0.84–0.88(t, 6H, CH3, *J*=7.2 Hz), 2.42 (*s*, 3H, CH3), 3.12–3.17 (*q*, 4H, CH2, *J* = 6.8 Hz), 7.14–8.03 (*m*, 8H, Ar—H). MS (EI 70 eV) *m*/*z*(%): 347 (M^+, 62), 318 (86), 275 (65), 130 (84), 91 (100). Elemental analysis: calculated for $C_{21}H_{21}N_3O_2$: C 72.60, H 6.09, N 12.10%; found: C 72.52, H 6.16, N 12.07%.

Crystal data

$C_{21}H_{21}N_3O_2$
M_r = 347.41
Monoclinic, $P2_1/n$
a = 16.164 (2) Å
b = 7.0063 (9) Å
c = 17.627 (2) Å
β = 113.685 (2)°
V = 1828.1 (4) Å^3
Z = 4
D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1397 reflections
θ = 2.5–21.6°
μ = 0.08 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
11487 measured reflections
3590 independent reflections
1941 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 26.0°
$h = -19 \rightarrow 19$
$k = -8 \rightarrow 8$
$l = -19 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.145
S = 0.94
3590 reflections
238 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.392 (3)	C10—N3	1.389 (3)
C1—C6	1.393 (3)	C10—N2	1.399 (3)
C1—C7	1.455 (3)	C11—N3	1.470 (3)
C5—C6	1.373 (3)	C13—N3	1.469 (3)
C6—O1	1.378 (3)	C15—C16	1.374 (3)
C7—N1	1.359 (3)	C15—C20	1.380 (3)
C7—C8	1.363 (3)	C15—N2	1.449 (3)
C8—O1	1.378 (3)	C16—C17	1.382 (4)
C8—C9	1.407 (3)	C17—C18	1.363 (4)
C9—O2	1.215 (3)	C18—C19	1.386 (4)
C9—N2	1.434 (3)	C19—C20	1.388 (3)
C10—N1	1.297 (3)	C19—C21	1.500 (4)
C2—C1—C6	119.5 (2)	C7—C8—C9	123.1 (2)
C2—C1—C7	135.2 (3)	O1—C8—C9	123.7 (2)
C6—C1—C7	105.4 (2)	O2—C9—C8	128.1 (2)
C3—C2—C1	117.7 (3)	O2—C9—N2	121.7 (2)
C2—C3—C4	120.7 (3)	C8—C9—N2	110.2 (2)
C5—C4—C3	122.7 (3)	N1—C10—N3	120.2 (2)
C5—C6—O1	124.9 (3)	N1—C10—N2	124.2 (2)
C5—C6—C1	123.6 (3)	N3—C10—N2	115.5 (2)
O1—C6—C1	111.5 (2)	C16—C15—N2	120.8 (2)
N1—C7—C8	124.6 (2)	C20—C15—N2	119.3 (2)
N1—C7—C1	130.0 (2)	C10—N3—C13	116.9 (2)
C8—C7—C1	105.3 (2)	C10—N3—C11	115.8 (2)
C7—C8—O1	113.1 (2)	C8—O1—C6	104.81 (19)
C6—C1—C2—C3	1.0 (4)	C2—C1—C7—C8	−178.0 (3)
C7—C1—C2—C3	−179.9 (3)	C6—C1—C7—C8	1.2 (3)
C4—C5—C6—O1	−179.0 (2)	C1—C7—C8—O1	−1.0 (3)
C4—C5—C6—C1	1.5 (4)	O1—C8—C9—O2	2.2 (4)
C2—C1—C6—C5	−2.0 (4)	O1—C8—C9—N2	179.6 (2)
C7—C1—C6—C5	178.7 (2)	C1—C7—N1—C10	−179.6 (2)
C2—C1—C6—O1	178.4 (2)	O2—C9—N2—C10	179.4 (2)
C7—C1—C6—O1	−1.0 (3)	C8—C9—N2—C10	1.8 (3)
C6—C1—C7—N1	−176.6 (2)	C8—C9—N2—C15	−168.1 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···N2	0.97	2.59	2.971 (3)	104

The H atoms were positioned geometrically [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) U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L. & Orpen, A. G. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Bodke, Y. & Sangapure, S. S. (2003). *J. Indian Chem. Soc.* **80**, 187–189.
Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ding, M. W.; Chen, Y. F.; Huang, N. Y. (2004*b*). *Eur. J. Org. Chem.* 3872–3878.
Ding, M. W.; Xu, S. Z.; Zhao, J. F. (2004*a*). *J. Org. Chem.* **69**, 8366–8371.
Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* 3885–3896.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.