Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2-Diisopropylamino-3-phenylbenzo[4,5]furo[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) = 0.004 Å
R factor = 0.059
wR factor = 0.139
Data-to-parameter ratio = 15.2

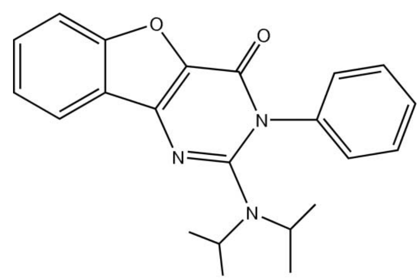
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 April 2005
Accepted 18 July 2005
Online 23 July 2005

In the title compound, $C_{22}H_{23}N_3O_2$, the three fused rings of the benzofuro[3,2-*d*]pyrimidine system are almost coplanar. The packing of the molecules in the crystal structure is mainly due to π–π and intermolecular C—H···O hydrogen-bonding interactions.

Comment

Derivatives of benzofuropyrimidines are of great importance because of their remarkable biological properties (Bodke *et al.*, 2003). In recent years, we have been engaged in the preparation of derivatives of heterocycles *via* the aza-Wittig reaction. The title compound, (I), was synthesized and structurally characterized in this context.



(I)

As shown in Fig. 1, all ring atoms in the benzofuro[3,2-*d*]pyrimidine system are essentially coplanar. Selected bond lengths and angles are listed in Table 1. A packing diagram is shown in Fig. 2. There exist some weak intermolecular C—H···O hydrogen-bonding interactions. Atom O2 and the H atom on C15 of the neighboring phenyl ring form a weak C—H···O interaction (Table 2). There are also intermolecular π–π interactions (Fig. 3). In the benzofuro[3,2-*d*]pyrimidine system, the maximum and minimum interplanar perpendicular distances between the nearly parallel and adjacent rings are 3.339 (3) and 3.388 (3) Å, respectively, the center-to-center distances are 3.496 (3) and 3.719 (3) Å, dihedral angles are 0.00 (2) and 0.91 (2)°, and the displacement angles are 23.93 (2) and 14.27 (2)°, showing that π–π stacking interactions exist (Janiak, 2000). However, the interplanar perpendicular distances between adjacent phenyl rings at the 3-position are 3.146 (3) and 3.229 (3) Å, the dihedral angle is 1.23 (2)°, and the center-to-center distance is 5.041 (3) Å, indicating that no π–π stacking interactions exist.

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 reaction, the mixture was

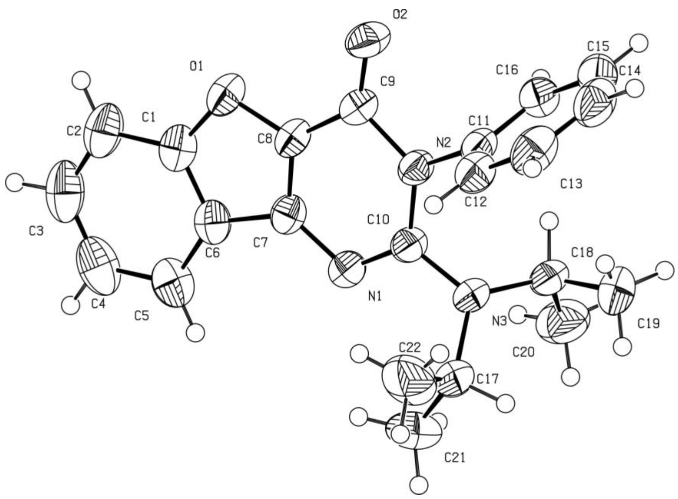


Figure 1
A view of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

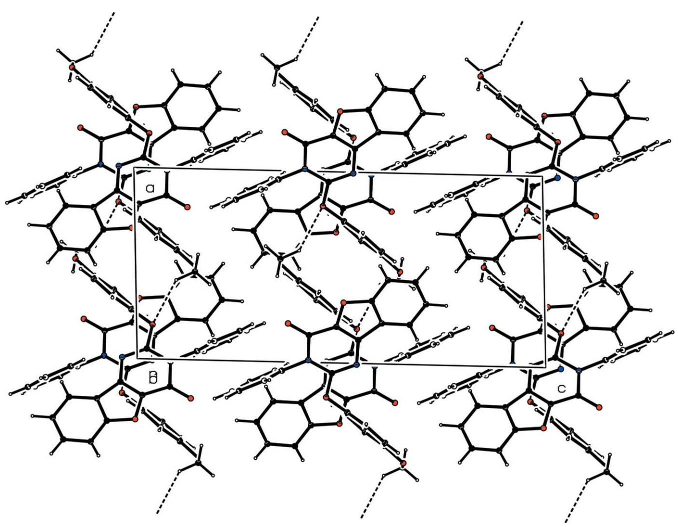


Figure 2
The crystal structure of (I), showing the formation of C—H···O hydrogen bonds (dashed lines).

allowed to stand for 10 h at 273–278 K, the solvent was removed under reduced pressure and diethyl ether/petroleum ether (1:2 *v*/*v*, 20 ml) was added to precipitate triphenylphosphine oxide. After filtration, the solvent was removed to give the carbodiimide, which was used directly without further purification. To the solution of the carbodiimide (15 ml) was added diisopropylamine (3 mmol). After reaction, the mixture was allowed to stand for 0.5 h, the solvent was removed and anhydrous ethanol (10 ml) containing 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 82% (m.p. 414 K). Suitable crystals were obtained by vapor diffusion of ethanol and dichloromethane at room temperature. Elemental analysis calculated for $C_{22}H_{23}N_3O_2$: C 73.11, H 6.41, N 11.63%; found: C 73.20, H 6.38, N 11.71%.

Crystal data

$C_{22}H_{23}N_3O_2$
M_r = 361.43
Monoclinic, $P2_1/c$
a = 9.5089 (10) Å
b = 7.7663 (8) Å
c = 26.204 (3) Å
β = 91.825 (2)°
V = 1934.2 (4) Å^3
Z = 4
D_x = 1.241 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2063 reflections
θ = 2.7–21.9°
μ = 0.08 mm^{-1}
T = 292 (2) K
Block, colorless
0.34 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
10238 measured reflections
3760 independent reflections
2727 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 26.0°
$h = -11 \rightarrow 10$
$k = -9 \rightarrow 9$
$l = -30 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.139
S = 1.09
3760 reflections
248 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.3195P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.381 (3)	C10—N1	1.297 (3)
C1—C6	1.387 (3)	C10—N3	1.383 (2)
C3—C4	1.386 (4)	C11—C12	1.379 (3)
C6—C7	1.450 (3)	C11—N2	1.453 (2)
C7—N1	1.369 (2)	C15—C16	1.374 (3)
C8—O1	1.382 (2)	C17—C21	1.524 (4)
C9—O2	1.219 (2)		
O1—C1—C2	124.6 (2)	C16—C11—C12	120.5 (2)
O1—C1—C6	112.23 (18)	C12—C11—N2	119.27 (19)
C2—C3—C4	122.4 (2)	C14—C15—C16	120.1 (2)
C5—C4—C3	121.1 (3)	N3—C17—C21	117.6 (2)
C1—C6—C5	119.9 (2)	C22—C17—C21	111.0 (2)
C8—C7—N1	124.31 (19)	N3—C18—C19	111.85 (19)
C7—C8—O1	112.56 (18)	C10—N1—C7	115.35 (17)
O2—C9—C8	128.19 (19)	C10—N2—C9	123.65 (17)
N1—C10—N3	120.82 (18)	C9—N2—C11	114.46 (16)
N1—C10—N2	123.15 (18)	C10—N3—C17	118.99 (17)
O1—C1—C2—C3	179.9 (2)	C6—C7—N1—C10	179.4 (2)
C3—C4—C5—C6	−0.1 (4)	N3—C10—N2—C9	178.11 (17)
C2—C1—C6—C7	−179.0 (2)	O2—C9—N2—C10	−177.48 (19)
C5—C6—C7—N1	1.3 (4)	C8—C9—N2—C11	−168.65 (17)
N1—C7—C8—O1	178.84 (17)	N1—C10—N3—C18	116.4 (2)
C7—C8—C9—O2	−179.4 (2)	N1—C10—N3—C17	−38.6 (3)
O1—C8—C9—O2	0.1 (3)	N2—C10—N3—C17	138.8 (2)
N2—C11—C12—C13	−178.16 (18)	C20—C18—N3—C10	−94.5 (2)
C13—C14—C15—C16	−0.5 (4)	C22—C17—N3—C10	−63.8 (3)
N2—C11—C16—C15	178.27 (18)	C22—C17—N3—C18	141.2 (2)
N3—C10—N1—C7	177.99 (18)	C6—C1—O1—C8	−0.4 (2)
N2—C10—N1—C7	0.9 (3)	C9—C8—O1—C1	179.96 (19)

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15···O2^{i}	0.93	2.48	3.253 (3)	140

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

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: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge financial support of this work by the National Basic Research Program of China (No. 2003CB114400) and the National Natural Science Foundation of China (Nos. 20372023 and 20102001).

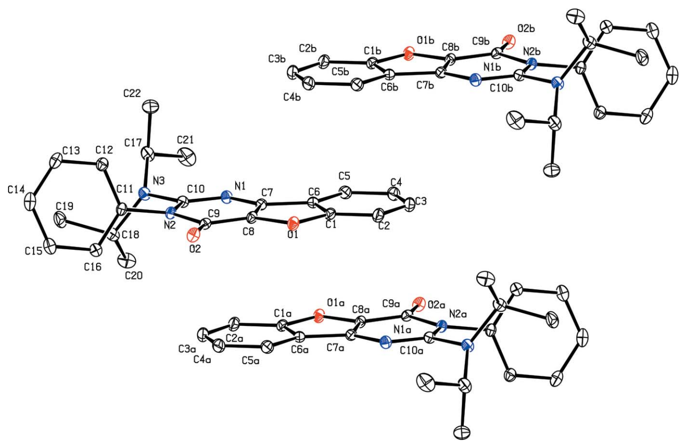


Figure 3
Part of the crystal structure of (I), showing the π–π stacking interactions. H atoms bonded to C atoms have been omitted for clarity. The top and bottom molecules are at the symmetry positions $(1 - x, 2 - y, -z)$ and $(1 - x, 1 - y, -z)$, respectively.

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