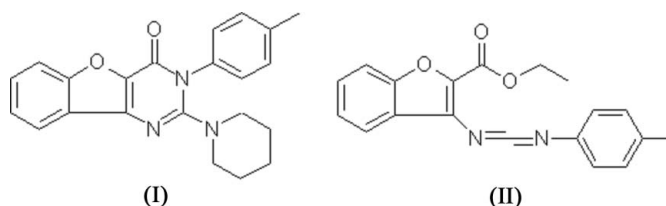


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Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.043
 wR factor = 0.117
Data-to-parameter ratio = 16.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3-(4-Methylphenyl)-2-(1-pyridyl)-3H-benzo-
[4,5]furo[3,2-d]pyrimidin-4-oneIn the crystal structure of the title compound, $\text{C}_{22}\text{H}_{21}\text{N}_3\text{O}_2$, the packing of the molecules is mainly governed by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and $\pi-\pi$ interactions.Received 1 March 2006
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Comment

In recent years, we have been engaged in the preparation of heterocyclic derivatives containing a fused pyrimidinone system using the aza-Wittig reaction (Ding, Chen & Huang, 2004; Ding, Xu & Zhao, 2004; Ding *et al.* 2005). Some X-ray crystal structure reports for benzofuro[3,2-*d*]pyrimidine derivatives have been published (Hu, Li *et al.*, 2005; Hu, Xu *et al.*, 2005; Sun & Hu, 2005). We describe here the structure of the title compound, (I), prepared using an iminophosphorane with 4-methylphenyl isocyanate and by a subsequent reaction with piperidine under mild conditions.

The three fused rings of the benzofuro[3,2-*d*]pyrimidine system are essentially coplanar (Fig. 1). The piperidine ring shows a distorted chair conformation [$\varphi = 203.84$ (2)° and $\theta = 175.54$ (2)°, puckering amplitude = 0.583 (2) Å; Cremer & Pople, 1975]. An intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond between the ketone group and the methyl-substituted benzene ring (Table 2 and Fig. 2) is effective in stabilizing the crystal structure. There are also intermolecular $\pi-\pi$ interactions (Janiak, 2000). In the benzofuro[3,2-*d*]pyrimidine system, the maximum and minimum interplanar perpendicular distances between adjacent rings related by an inversion centre are 3.400 (3) and 3.650 (3) Å, respectively, the centroid-to-centroid distances are 3.613 (3) and 3.461 (3) Å, and the dihedral angles are 0.00 (2) and 2.57 (2)° (Fig. 2).

Experimental

To a solution of iminophosphorane (1.40 g, 3 mmol) in dry dichloromethane (15 ml) was added 4-methylphenyl isocyanate (3 mmol) under nitrogen at room temperature. After reaction, the mixture was 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 3-*p*-tolyliminomethyleneaminobenzofuran-2-carboxylic acid ethyl ester, (II), which was used directly without further purification. To a solution of (II) in dichloromethane (15 ml) was added piperidine (3 mmol). After the reaction mixture was allowed to stand for 0.5 h, the solvent was removed and anhydrous ethanol (10 ml) with several drops of EtONa in EtOH was 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). The product was recrystallized from ethanol–dichloromethane (1:1 *v/v*, 20 ml) at room temperature to give crystals suitable for X-ray diffraction (m.p. 485 K, yield 82%).

Crystal data

$C_{22}H_{21}N_3O_2$ $D_x = 1.295 \text{ Mg m}^{-3}$
 $M_r = 359.42$ Mo $K\alpha$ radiation
 Monoclinic, $P2_1/n$ Cell parameters from 3667 reflections
 $a = 14.4774 (12) \text{ \AA}$ $\theta = 2.5\text{--}27.3^\circ$
 $b = 7.7044 (7) \text{ \AA}$ $\mu = 0.09 \text{ mm}^{-1}$
 $c = 16.6859 (15) \text{ \AA}$ $T = 292 (2) \text{ K}$
 $\beta = 97.754 (2)^\circ$ Block, colourless
 $V = 1844.1 (3) \text{ \AA}^3$ $0.30 \times 0.30 \times 0.30 \text{ mm}$
 $Z = 4$

Data collection

Bruker SMART 4K CCD area-detector diffractometer 2960 reflections with $I > 2\sigma(I)$
 φ and ω scans $R_{int} = 0.052$
 Absorption correction: none $\theta_{max} = 27.0^\circ$
 10894 measured reflections $h = -15 \rightarrow 18$
 3995 independent reflections $k = -9 \rightarrow 9$
 $l = -21 \rightarrow 17$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0668P)^2]$
 $R[F^2 > 2\sigma(F^2)] = 0.043$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.117$ $(\Delta/\sigma)_{max} < 0.001$
 $S = 0.98$ $\Delta\rho_{max} = 0.16 \text{ e \AA}^{-3}$
 3995 reflections $\Delta\rho_{min} = -0.22 \text{ e \AA}^{-3}$
 246 parameters Extinction correction: *SHELXL97*
 H -atom parameters constrained (Sheldrick, 1997)
 Extinction coefficient: $0.023 (2)$

Table 1

Selected geometric parameters (\AA , $^\circ$).

C1–N1	1.4735 (18)	C14–N2	1.4243 (16)
C5–N1	1.4715 (16)	C15–O2	1.3791 (15)
C6–N3	1.3015 (15)	C16–N3	1.3694 (16)
C6–N1	1.3904 (16)	C18–O2	1.3852 (15)
C6–N2	1.3961 (15)	C7–N2	1.4501 (15)
C14–O1	1.2174 (15)		
N1–C1–C2	110.02 (12)	N15–C16–N3	123.87 (11)
N1–C5–C4	110.02 (11)	N3–C16–C17	129.97 (12)
N3–C6–N1	120.37 (10)	C19–C18–O2	125.01 (13)
N3–C6–N2	123.83 (11)	O2–C18–C17	111.55 (11)
N1–C6–N2	115.76 (10)	C8–C7–N2	120.29 (11)
O1–C14–C15	128.27 (12)	C6–N3–C16	115.31 (10)
O1–C14–N2	121.65 (11)	C6–N2–C14	123.33 (10)
C15–C14–N2	110.04 (10)	C6–N1–C5	115.10 (10)
C16–C15–O2	112.87 (11)	C15–O2–C18	104.38 (10)
N2–C14–C15–C16	−3.14 (18)	N1–C6–N3–C16	−179.70 (11)
N2–C14–C15–O2	177.82 (11)	N3–C6–N2–C14	2.12 (19)
O2–C15–C16–N3	−177.53 (11)	N1–C6–N2–C7	6.34 (17)
C20–C19–C18–O2	179.72 (12)	N3–C6–N1–C5	18.96 (17)
C22–C17–C18–O2	−179.73 (11)	C14–C15–O2–C18	179.87 (12)
C16–C17–C18–O2	0.96 (14)	C17–C18–O2–C15	−1.05 (13)
N2–C7–C8–C9	−178.73 (12)		

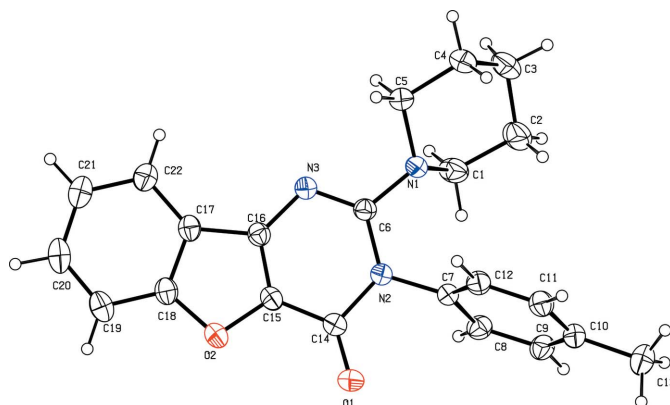


Figure 1 View of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

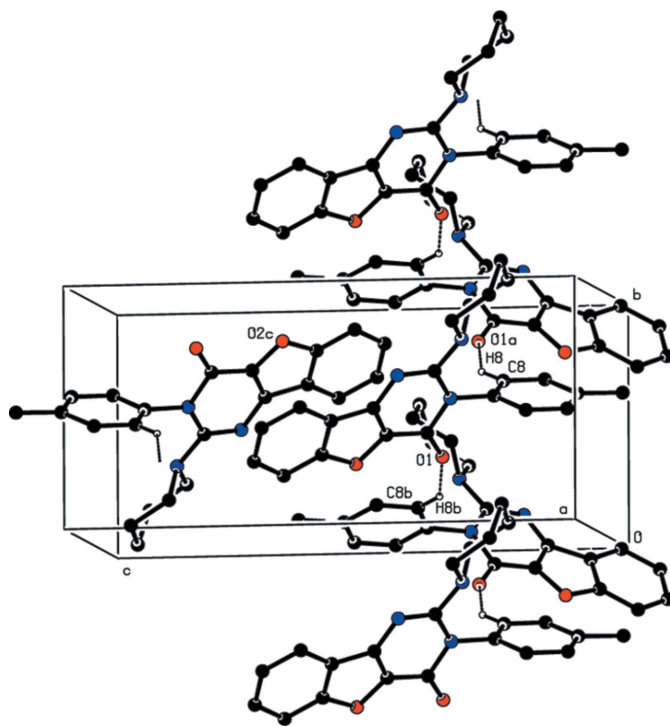


Figure 2 Packing view of (I), showing the C–H...O hydrogen bonds (dashed lines) and the π – π stacking interactions. H atoms attached to C atoms have been omitted for clarity. The suffixes a, b and c correspond to symmetry codes $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$, $(\frac{1}{2} - x, \frac{1}{2} - y, \frac{1}{2} - z)$ and $(2 - x, 1 - y, 1 - z)$, respectively.

Table 2 Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8–H8...O1 ⁱ	0.93	2.51	3.2369 (16)	135

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

The H atoms were positioned geometrically [$C-H = 0.93$ (CH), 0.97 (CH₂) and 0.96 \AA (CH₃)] 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: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; 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*.

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