Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(4-Methylphenyl)-2-morpholinobenzo[4,5]-furo[3,2-*d*]pyrimidin-4(3*H*)-one

Yong Sun[a]* and Yang-Gen Hu[b]

[a]Yunyang Teachers College, Danjiangkou 442700, People's Republic of China, and [b]Department of Medicinal Chemistry, Yunyang Medical College, Shiyan 442000, People's Republic of China

Correspondence e-mail: suny6135@126.com

Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.132
Data-to-parameter ratio = 16.0

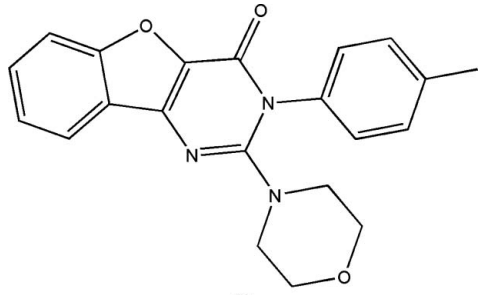
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 November 2005
Accepted 17 November 2005
Online 23 November 2005

In the title compound, $C_{21}H_{19}N_3O_3$, 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 governed by π–π and intermolecular C—H···O hydrogen-bonding interactions. The C—H···O bonds link the molecules into dimers.

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 the derivatives of heterocycles *via* the aza-Wittig reaction. The title compound, (I), may be used as a new precursor for obtaining bioactive molecules.



(I)

Fig. 1 shows the molecular structure of (I) with the atomic numbering scheme. The three fused rings of the benzofuro[3,2-*d*]pyrimidine system are almost coplanar. The morpholine ring has a total puckering amplitude of 1.022 (3) Å (Cremer & Pople, 1975) and a distorted chair form

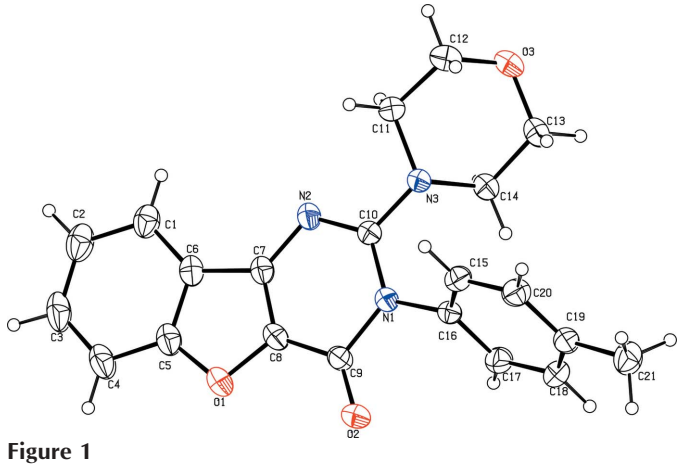


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

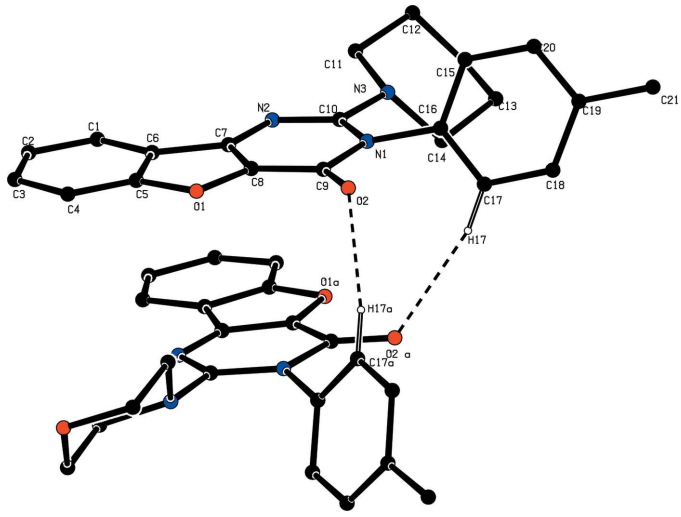


Figure 2
Part of the crystal structure of (I), showing hydrogen-bonding associations (dashed lines) and π–π stacking interactions.

[$\varphi = -31.0$ (4) and $\theta = 57.8$ (5)°]. The bond lengths and angles (Table 1) are in agreement with reported literature values (Allen *et al.*, 1987).

Intermolecular C—H···O hydrogen bonds (Table 2, and Figs. 2 and 3) seem to be effective in stabilizing the crystal structure. There are also intermolecular π–π interactions (Fig. 2). In the benzofuro[3,2-*d*]pyrimidine system, the centroid-to-centroid distance is 3.616 (13) Å. The dihedral angle between the rings O1/C5–C8 and C1–C6 is 1.19 (1)°. The angles between the ring-centroid vectors and the ring normals [17.67 (2) and 19.94 (2)°] support the existence of π–π interactions (Janiak, 2000).

In the crystal structure, C—H···O intermolecular hydrogen bonds link the molecules into dimers. As can be seen from the packing diagram (Fig. 3), the dimers are stacked along the *b* axis.

Experimental

Phenyl isocyanate (3 mmol) was added to a solution of iminophosphorane (1.40 g, 3 mmol) in dry dichloromethane (15 ml) under nitrogen at room temperature. When the reaction mixture had stood 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, (II), which was used directly without further purification. Morpholine (3 mmol) was added to a solution of (II) in dichloromethane (15 ml). 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. The solution was concentrated under reduced pressure and the residue was recrystallized from ethanol to give the title compound (I) (yield 0.89 g, 82%, m.p. 466 K). Suitable crystals were obtained by vapor diffusion of ethanol into a dichloromethane solution at room temperature. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.43 (*s*, 3H, CH_3), 3.18 (*t*, 4H, CH_2, J = 4.8 Hz), 3.46 (*t*, 4H, CH_2, J = 4.4 Hz), 7.26–8.03 (*m*, 8H, Ar—H). MS (EI 70 eV) *m*/*z* (%): 361 (M^+, 73), 316 (88), 275 (81), 130 (100), 91 (95). Elemental analysis calculated for $C_{21}H_{19}N_3O_3$: C 69.41, H 5.82, N 11.56%; found: C 69.32, H 5.75, N 11.69%.

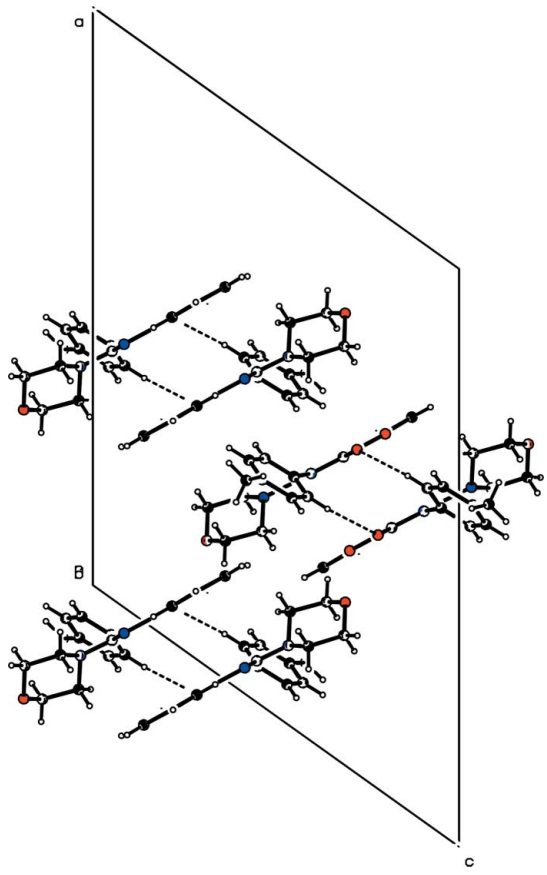


Figure 3
Packing diagram of (I). Hydrogen bonds are shown as dashed lines.

Crystal data

$C_{21}H_{19}N_3O_3$	D_x = 1.335 Mg m^{-3}
M_r = 361.39	Mo $K\alpha$ radiation
Monoclinic, *C*2/*c*	Cell parameters from 3724 reflections
a = 22.8042 (16) Å	θ = 2.2–24.5°
b = 10.9268 (8) Å	μ = 0.09 mm^{-1}
c = 17.7465 (12) Å	*T* = 292 (2) K
β = 125.562 (1)°	Block, colorless
V = 3597.3 (4) Å^3	0.30 × 0.20 × 0.20 mm
Z = 8	

Data collection

Bruker SMART CCD area-detector diffractometer	2780 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.034
Absorption correction: none	θ_{max} = 27.0°
14758 measured reflections	*h* = −29 → 29
3923 independent reflections	*k* = −13 → 13
	l = −22 → 22

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.7865P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.132	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
3923 reflections	
245 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C5—O1	1.381 (2)	C10—N2	1.3042 (19)
C5—C6	1.388 (3)	C10—N3	1.388 (2)
C6—C7	1.448 (2)	C11—N3	1.464 (2)
C7—C8	1.350 (2)	C11—C12	1.500 (3)
C7—N2	1.365 (2)	C12—O3	1.414 (3)
C8—O1	1.3833 (19)	C13—O3	1.417 (2)
C9—O2	1.2165 (19)	C13—C14	1.505 (2)
C9—N1	1.434 (2)	C14—N3	1.470 (2)
N3—C11—C12	109.42 (15)	N3—C14—C13	109.69 (15)
O3—C12—C11	111.69 (17)	C11—N3—C14	110.35 (14)
O3—C13—C14	111.36 (15)	C12—O3—C13	109.42 (15)
N3—C11—C12—O3	58.5 (2)	C13—C14—N3—C11	55.07 (19)
O3—C13—C14—N3	−57.6 (2)	C11—C12—O3—C13	−60.5 (2)
C12—C11—N3—C14	−55.3 (2)	C14—C13—O3—C12	59.9 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17···O2^{i}	0.93	2.47	3.372 (2)	164

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

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) values of 1.2 (1.5 for methyl) times 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. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
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.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* 97, pp. 1354–1358.
Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3885–3896.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.