Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

5-(4-Chlorophenyl)-7-phenylpyrido[2,3-*d*]-pyrimidine-1,8-dione dimethylformamide solvate

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Received 22 December 2005
Accepted 3 January 2006
Online 7 January 2006

Key indicators

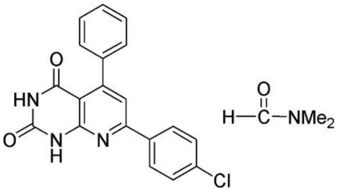
Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.094
Data-to-parameter ratio = 12.8

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

The title compound, $C_{19}H_{12}N_3O_2\cdot C_3H_7NO$, was synthesized by the reaction of 1-(4-chlorophenyl)-3-phenylprop-2-en-1-one and 6-aminouracil in the presence of KF–alumina in ethyl alcohol; its crystal structure was determined at low temperature [193 (2) K]. The crystal structure is stabilized by N—H···O, C—H···O and C—H···Cl hydrogen bonds.

Comment

The synthesis of pyridopyrimidines is of importance in organic chemistry, since it has been reported that they possess biological and pharmacological activities, such as antifolate (Rosowsky *et al.*, 1995), antibacterial (Nargund *et al.*, 1991), tyrosine kinase (Thompson *et al.*, 1995), antimicrobial (Donkor *et al.*, 1995), and calcium channel antagonist activity (Pastor *et al.*, 1994), This prompted us to investigate these compounds. The use of fluoride salts in a variety of synthetic reactions has been recognized in recent years (Clark, 1980). In particular, potassium fluoride coated with alumina (KF–alumina) has been a versatile solid-supported reagent developed for alkylation (Ando & Yamawaki, 1979), and over the years the reagent has found application in a large number of organic reactions (Wang *et al.*, 2003, Wang *et al.*, 2004). We report here the crystal structure of the title compound, (I).



(I)

The bond lengths and angles are unexceptional. The pyridine ring is essentially coplanar with the C14–C19 benzene ring and the pyrimidine ring, forming dihedral angles of 3.1 (1) and 2.7 (1)°, respectively. The dihedral angle between the pyridine ring and the C8–C13 phenyl ring is 50.1 (1)°; the dihedral angle between the two benzene rings is 51.0 (1)°.

The crystal structure is stabilized by N—H···O, C—H···O and C—H···Cl hydrogen bonds (Table 1, Fig. 2).

Experimental

The title compound, (I), was prepared by the reaction of 1-(4-chlorophenyl)-3-phenyl-2-propen-1-one (2 mmol, 0.48 g) and 6-aminouracil (2 mmol, 0.26 g) in the presence of KF–alumina (0.25 g) in ethyl alcohol at 363 K for 6 h (yield 87%, m.p. 561–562 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a dimethylformamide solution.

Elemental analysis calculated: C 65.24, H 3.46, N 12.01%; found: C 65.09, H 3.52, N 12.28%. ^{1}H NMR (DMSO-d_6): 2.74 (s, 3H, CH$_3$), 2.90 (s, 3H, CH$_3$), 7.43 (s, 5H, ArH), 7.57 (s, 1H, ArH), 7.61 (d, J = 8.4 Hz, 2H, ArH), 7.96 (s, 1H, CHO), 8.25 (d, J = 8.4 Hz, 2H, ArH), 11.21 (s, 1H, NH), 11.83 (s, 1H, NH); IR (cm^{-1}): 3174 (NH), 3061 (ArH), 1712 (C=O), 1690 (C=O), 1587, 1553, 1490 (benzene ring).

Crystal data

$C_{19}H_{12}ClN_3O_2 \cdot C_3H_7NO$
$M_r = 422.86$
Triclinic, $P\overline{1}$
$a = 7.6985$ (7) Å
$b = 11.7486$ (10) Å
$c = 12.0533$ (8) Å
$\alpha = 72.813$ (7)°
$\beta = 72.868$ (7)°
$\gamma = 80.952$ (8)°
$V = 992.34$ (14) Å^3
$Z = 2$
$D_x = 1.415$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 3710 reflections
$\theta = 3.0$–25.3°
$\mu = 0.23$ mm^{-1}
$T = 193$ (2) K
Block, yellow
$0.52 \times 0.31 \times 0.17$ mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{min} = 0.892$, $T_{max} = 0.963$
9845 measured reflections
3611 independent reflections
3249 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 25.4°$
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 14$
$l = -12 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.094$
$S = 1.08$
3611 reflections
282 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.3685P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3^{i}	0.90 (2)	1.90 (2)	2.7918 (18)	168.2 (18)
N3—H3···O1^{ii}	0.89 (2)	1.95 (2)	2.8381 (17)	169.8 (17)
C10—H10···O2^{iii}	0.95	2.40	3.328 (2)	164
C13—H13···O3^{iv}	0.95	2.57	3.514 (2)	172
C20—H20···O1^{v}	0.95	2.57	3.167 (2)	121
C21—H21B···O2^{vi}	0.98	2.55	3.399 (2)	145
C16—H16···Cl1^{vii}	0.95	2.86	3.7763 (17)	162

Symmetry codes: (i) $x, y, z-1$; (ii) $-x, -y, -z$; (iii) $-x, -y, -z+1$; (iv) $-x, -y+1, -z+1$; (v) $x, y, z+1$; (vi) $x+1, y, z$; (vii) $-x+1, -y+2, -z$.

The carbon-bound H atoms were positioned geometrically and refined as riding, with C—H = 0.95–0.98 Å and U_{iso}(H) = 1.2U_{eq}(carrier atom). H2 and H3 were refined freely; N2—H2 = 0.90 (2) and N3—H3 = 0.89 (2) Å.

Data collection: *CRYSTALCLEAR* (Rigaku, 1999); cell refinement: *CRYSTALCLEAR*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

We thank the Foundation of the Natural Science Foundation (04 KJB150139) of the Education Committee of Jiangsu Province for financial support.

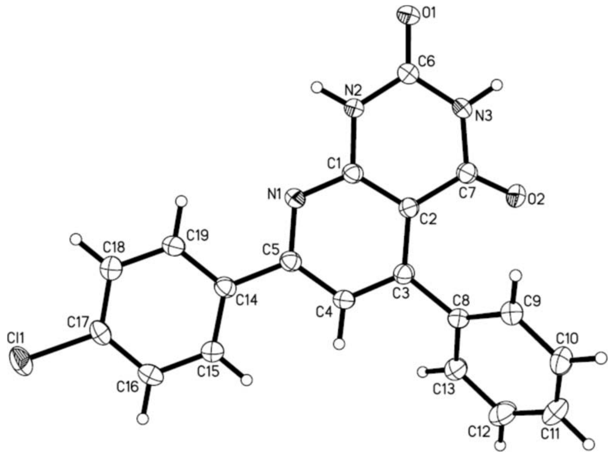


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level and the atom-numbering scheme. The DMF molecule of crystallization has been omitted for clarity.

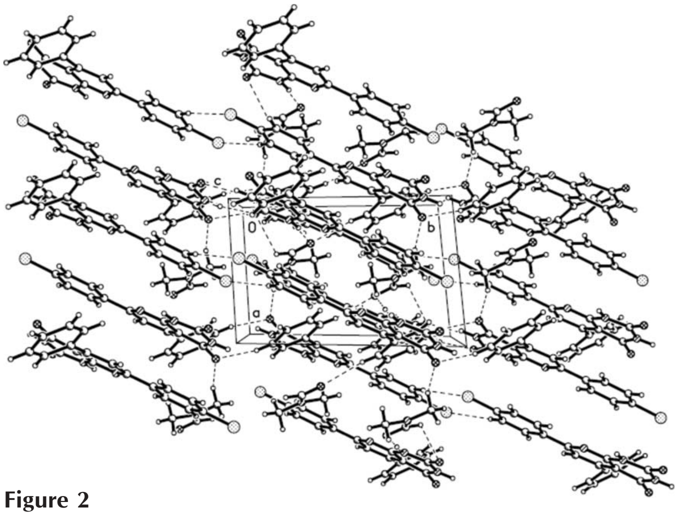


Figure 2
A packing diagram of (I). Dashed lines indicate hydrogen bonds.

References

Ando, T. & Yamawaki, J. (1979). *Chem. Lett.* pp. 755–758.
Clark, J. H. (1980). *Chem. Rev.* **80**, 429–452.
Donkor, I. O., Klein, C. L., Liang, L., Zhu, N., Bradley, E. & Clark, A. M. (1995). *J. Pharm. Sci.* **84**, 661–664.
Jacobson, R. (1998). Private communication to the Rigaku Corporation, Tokyo, Japan.
Nargund, L. V. G., Reddy, Y. S. R. & Jose, R. (1991). *Indian Drugs*, **29**, 45–46.
Pastor, A., Alajarin, R., Vaquero, J. J., Alvarez-Builla, J., Fau de Casa-Juana, M., Sunkel, C., Priego, J. G., Fonseca, I. & Sanz-Aparicio, J. (1994). *Tetrahedron*, **50**, 8085–8098.
Rigaku (1999). *CRYSTALCLEAR*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2003). *CrystalStructure*. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Rosowsky, A., Mota, C. E. & Queener, S. F. (1995). *J. Heterocycl. Chem.* **32**, 335–340.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany;.
Thompson, A. M., Bridges, A. J., Fry, D. W., Kraker, A. J. & Denny, W. A. (1995). *J. Med. Chem.* **38**, 3780–3788.
Wang, X. S., Shi, D. Q. & Tu, S. J. (2003). *Synth. Commun.* **33**, 119–126.
Wang, X. S., Shi, D. Q., Yu, H. Z., Wang, G. F. & Tu, S. J. (2004). *Synth. Commun.* **34**, 509–514.

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