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Key indicators

Single-crystal X-ray study
T = 296 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
R factor = 0.043
wR factor = 0.120
Data-to-parameter ratio = 16.5

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

7-(4-Bromophenyl)-5-cyanomethyl-1,3-dimethylpyrido[2,3-d]pyrimidine-2,4-dione

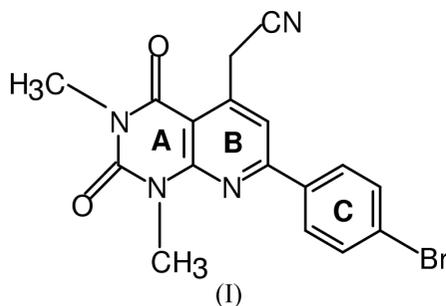
The positions of the cyanomethyl and 4-bromophenyl substituents in the title compound, $\text{C}_{17}\text{H}_{13}\text{BrN}_4\text{O}_2$, have been confirmed by single-crystal X-ray structure determination. The crystal packing shows weak hydrogen-bonding and aromatic π - π interactions.

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Comment

The therapeutic importance (Furuya & Ohataki, 1994*a,b*; Heber *et al.*, 1993; Piper *et al.*, 1989) of suitably functionalized pyrido[2,3-*d*]pyrimidine ring systems encouraged us to develop an innovative synthesis in which different substituents could be arranged in a pharmacophoric pattern to display diverse pharmacological activities of higher order. The title compound, (I), was prepared (Srivastava *et al.*, 2000) by ring-transformation reactions of 6-aryl-3-cyano-4-methylthio-2*H*-pyran-2-one and 6-amino-1,3-dimethyluracil. The present X-ray crystallography study has been undertaken to confirm the structure of (I).



The conformation of (I) along with the atom-numbering scheme is shown in Fig. 1. The molecule contains one fused-ring system (*A/B*) to which a bromophenyl ring (*C*) is attached at the 7 position. All the rings are planar [deviations of the atoms from their least-squares planes are within the range -0.015 (3)– 0.011 (3) \AA]. The bromophenyl ring is coplanar with the *A/B* ring system [torsion angle $\text{N}8-\text{C}7-\text{C}71-\text{C}72 = 179.0$ (4) $^\circ$]. The cyano group is twisted out of the *A/B* plane. The crystal structure analysis reveals the presence of weak hydrogen-bonding interactions, such as $\text{C}-\text{H}\cdots\text{N}$, $\text{C}-\text{H}\cdots\text{Br}$ and $\text{C}-\text{H}\cdots\text{O}$ (Fig. 2 and Table 1). Moreover, the crystal packing (Fig. 3) also shows intermolecular stacking as a result of π - π interactions between rings *B* and *C* (Hunter & Sanders, 1990). The overlapping rings (*B* and *C*) are in a 'parallel-displaced orientation'; the average distance of separation between their mean planes is 3.480 (6) \AA , the angle between them being 1.0 (3) $^\circ$. The different intermolecular interactions seen in the crystal structure of (I) might well be a reflection of the possible interactions in the molecular recognition process

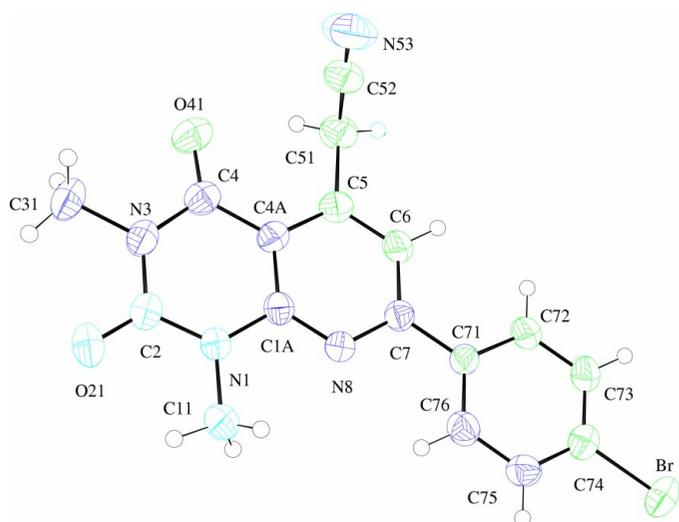


Figure 1
ORTEP (Johnson, 1965) diagram showing the molecular structure of (I) with labelling for non-H atoms and displacement ellipsoids at the 50% probability level.

in view of the diverse pharmacological properties associated with the suitably functionalized pyrido[2,3-*d*]pyrimidines.

Experimental

The synthesis of (I) was carried out by reaction of 6-amino-1,3-dimethyluracil with 6-(4-bromophenyl)-3-cyano-4-methylthio-2H-pyran-2-one (Srivastava *et al.*, 2000). Diffraction-quality crystals were grown by slow evaporation from a solution in a dichloromethane–ethyl acetate mixture at room temperature.

Crystal data

$C_{17}H_{13}BrN_4O_2$
 $M_r = 385.22$
 Triclinic, $P\bar{1}$
 $a = 8.728(3) \text{ \AA}$
 $b = 12.087(4) \text{ \AA}$
 $c = 8.672(3) \text{ \AA}$
 $\alpha = 108.22(3)^\circ$
 $\beta = 113.43(2)^\circ$
 $\gamma = 91.24(3)^\circ$
 $V = 786.1(5) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.628 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 24 reflections
 $\theta = 14.7\text{--}15.0^\circ$
 $\mu = 2.63 \text{ mm}^{-1}$
 $T = 296(2) \text{ K}$
 Block, colourless
 $0.4 \times 0.3 \times 0.1 \text{ mm}$

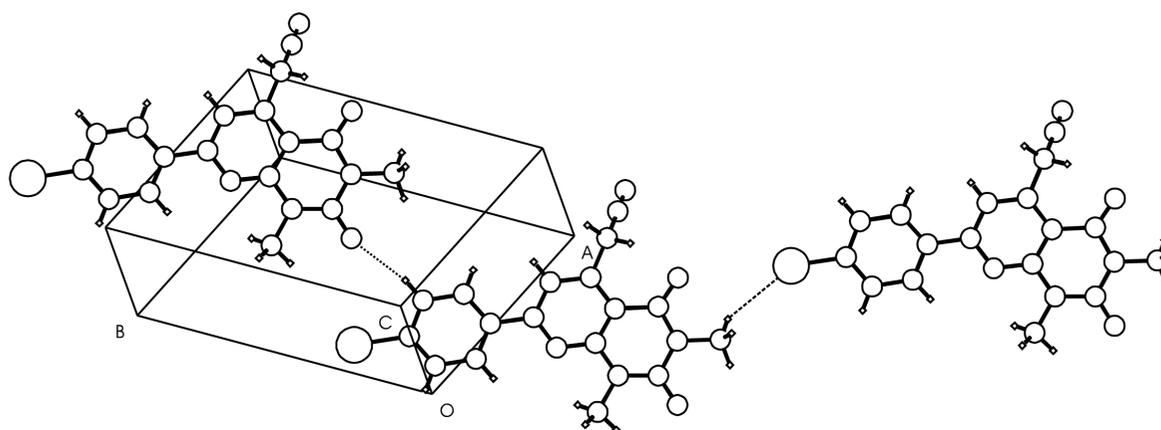


Figure 2
PLUTO (Motherwell & Clegg, 1978) crystal-packing diagram showing the C–H...O hydrogen bonds as dotted lines and the C–H...Br hydrogen bonds as dashed lines.

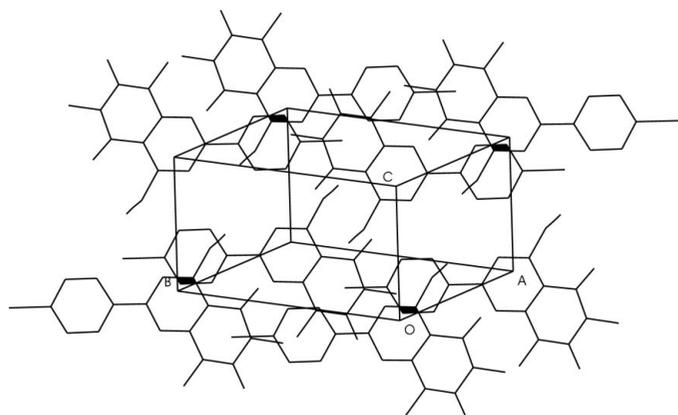


Figure 3
The crystal packing (perpendicular view to the plane through atoms C6, C7 and N8 of ring B) showing the partial overlapping (as indicated by black shading) of rings B and C owing to intermolecular stacking.

Data collection

Rigaku AFC-5R diffractometer
 ω - 2θ scans
 Absorption correction: empirical (TEXSAN; Molecular Structure Corporation/Rigaku Corporation, 1998)
 $T_{\min} = 0.509$, $T_{\max} = 0.769$
 3619 measured reflections
 3619 independent reflections

2254 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 27.5^\circ$
 $h = -11 \rightarrow 11$
 $k = -15 \rightarrow 0$
 $l = -10 \rightarrow 11$
 3 standard reflections every 150 reflections
 intensity decay: <0.2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.120$
 $S = 1.08$
 3616 reflections
 219 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.8316P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.48 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.43 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C31-H31B \cdots Br^i$	0.96	3.07	3.701 (5)	125
$C31-H31B \cdots N8^{ii}$	0.96	2.81	3.639 (6)	145
$C31-H31C \cdots O41^{iii}$	0.96	2.66	3.600 (6)	166
$C6-H6 \cdots N53^{iv}$	0.93	2.76	3.565 (7)	146
$C72-H72 \cdots N53^{iv}$	0.93	2.65	3.476 (7)	148
$C73-H73 \cdots O21^v$	0.93	2.32	3.223 (6)	165

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

All the H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms.

Data collection and cell refinement: *Rigaku/AFC Diffractometer Control Software* (Rigaku, 1995); data reduction: *TEXSAN* (Molecular Structure Corporation/Rigaku Corporation, 1998); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *NRCVAX* (Gabe *et al.*, 1989); software used to prepare material for publication: *SHELXL93*.

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