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Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.037
 wR factor = 0.110
Data-to-parameter ratio = 14.9

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

1-(3-Chlorobenzyl)pyrimidine-2,4(1*H*,3*H*)-dione

The title compound, $\text{C}_{11}\text{H}_9\text{ClN}_2\text{O}_2$, crystallizes with weak intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bond interactions. The dihedral angle between the pyrimidine and benzene rings is $86.82(7)^\circ$.

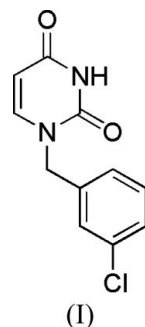
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Comment

Nucleoside analogues have been the cornerstones of chemotherapy against cancer and viral diseases (De Clercq, 1983). However, clinical toxicities arise for various reasons such as limited uptake, high susceptibility to catabolism and rapid emergence of resistance towards viral or cancer cells. This has led to diversification to non-nucleoside inhibitors (NNIs) (Ragno *et al.*, 2004). In order to discover more biologically active pyrimidine compounds, the title compound, (I), was synthesized and its crystal structure determined (Fig. 1).



In the crystal structure of the title molecule, (I), the heterocycle contains the structural unit CONHCO rather than $\text{HOC}=\text{N}-\text{CO}$ and makes a dihedral angle of $86.82(7)^\circ$ with the benzene ring. In the crystal structure, there are weak intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bond interactions (Fig. 2), which generate a chain running parallel to the b axis.

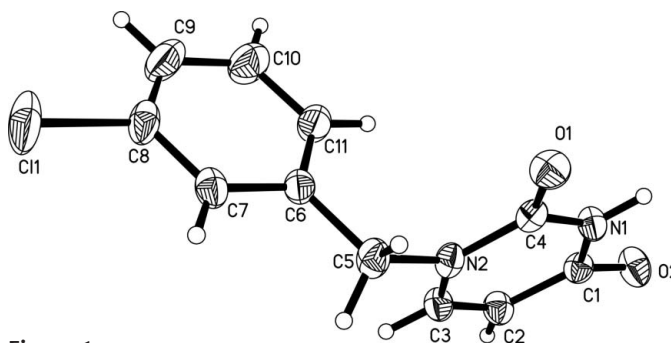


Figure 1
View of the title compound (I), with displacement ellipsoids drawn at the 30% probability level.

Experimental

Uracil (5 mmol) and anhydrous potassium carbonate (6 mmol) were mixed in *N,N*-dimethylformamide (20 ml). A solution of 3-chlorobenzyl chloride (0.81 g, 5 mmol) in acetone (10 ml) was then added dropwise, with stirring, at room temperature, and the mixture was stirred for another 10 h and then refluxed for 4 h. The solvent was evaporated *in vacuo* and the residue was washed with water. The resulting white precipitate was filtered off and purified by column chromatography on silica gel (petroleum ether:ethyl acetate = 2:1). The title compound was recrystallized from ethyl acetate and single crystals of (I) were obtained (m.p. 437–438 K).

Crystal data

$C_{11}H_9ClN_2O_2$	$D_x = 1.445 \text{ Mg m}^{-3}$
$M_r = 236.65$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1736 reflections
$a = 10.0756 (19) \text{ \AA}$	$\theta = 2.7\text{--}26.0^\circ$
$b = 7.0683 (13) \text{ \AA}$	$\mu = 0.34 \text{ mm}^{-1}$
$c = 15.620 (3) \text{ \AA}$	$T = 294 (2) \text{ K}$
$\beta = 102.030 (3)^\circ$	Prism, colourless
$V = 1088.0 (4) \text{ \AA}^3$	$0.28 \times 0.20 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2221 independent reflections
φ and ω scans	1394 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	$R_{\text{int}} = 0.029$
$T_{\text{min}} = 0.907$, $T_{\text{max}} = 0.969$	$\theta_{\text{max}} = 26.4^\circ$
5918 measured reflections	$h = -7 \rightarrow 12$
	$k = -8 \rightarrow 8$
	$l = -19 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.1563P]$
$R[F^2 > 2\sigma(F^2)] = 0.037$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.110$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
2221 reflections	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$
149 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

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

O2—C1	1.234 (2)	N1—C4	1.377 (2)
N1—C1	1.376 (2)	N1—H1	0.87 (2)
C3—N2—C5—C6	−81.3 (2)	N2—C5—C6—C11	−11.3 (3)
C4—N2—C5—C6	99.57 (19)	N2—C5—C6—C7	169.68 (16)

Table 2

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

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N1—H1 \cdots O2 ⁱ	0.87 (2)	1.94 (2)	2.805 (2)	177 (2)

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

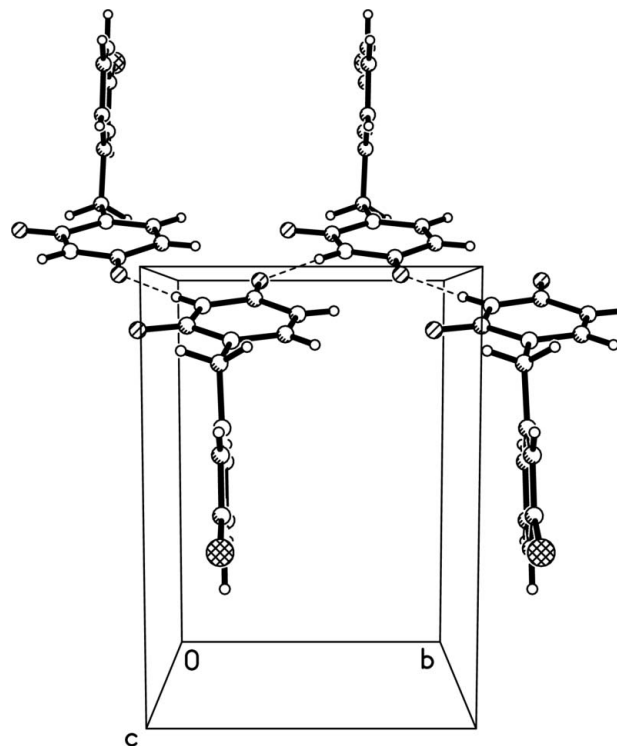


Figure 2

A portion of the N—H \cdots O hydrogen-bonded chain. Hydrogen bonds are shown as dashed lines.

H1, attached to N1, was refined freely. Other H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 \AA , and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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