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2-Chloro-4-(4-methoxybenzyloxy)pyrimidine

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.009 Å
R factor = 0.141
wR factor = 0.417
Data-to-parameter ratio = 14.3

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

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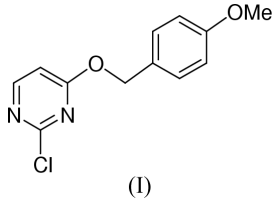
The structure of the title compound, $C_{12}H_{11}ClN_2O_2$, prepared in a synthetic study on spicamycin derivatives, has been determined. In the crystal structure, the molecules lie on mirror planes and form a herring-bone structure.

Comment

Spicamycin is an antitumor antibiotic consisting of a hepto-pyranose and an adenine moiety. The title compound, (I), was prepared in a synthetic study on spicamycin derivatives possessing various heterocyclic bases (Suzuki, Suzuki *et al.*, 2002). Coupling of (I) with the heptose moiety of spicamycin, followed by deprotection, gave the spicamycin derivative (5-hydroxypyrimidinyl)aminospicamycin (Suzuki, Matuura & Chida, 2002). Since the geometry of the compound could not be fully determined based on the NMR spectra, the X-ray analysis of (I) has been carried out.



(I)

The molecule of (I) is perfectly planar, since all the non-H atoms lie on a mirror plane (Fig. 1). The molecules form a herring-bone structure on the mirror planes at $y = \frac{1}{4}$ and $\frac{3}{4}$ in the crystal (Fig. 2). The displacement parameters of atom Cl1 show large anisotropy (Fig. 3), suggesting positional disorder or a stacking fault along the b axis.

Experimental

Basic hydrolysis of 2,4-dichloropyrimidine (Kazimierczuk *et al.*, 1972), followed by alkylation with 4-methoxybenzyl chloride in aqueous NaOH–CH_2Cl_2, in the presence of tetrabutylammonium iodide, afforded the title compound, (I). The crystal of (I) obtained from a toluene/methanol solution by slow evaporation did not have any planar faces. A transparent part was cut from the solid for the X-ray measurements. The crystal specimen used became opaque white in air after two weeks.

Crystal data

$C_{12}H_{11}ClN_2O_2$
M_r = 250.68
Orthorhombic, *Pnma*
a = 12.311 (2) Å
b = 6.788 (2) Å
c = 14.222 (2) Å
V = 1188.5 (4) Å^3
Z = 4
D_x = 1.401 Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
θ = 12.0–12.5°
μ = 0.31 mm^{-1}
T = 299 K
Prism, colorless
0.50 × 0.50 × 0.50 mm

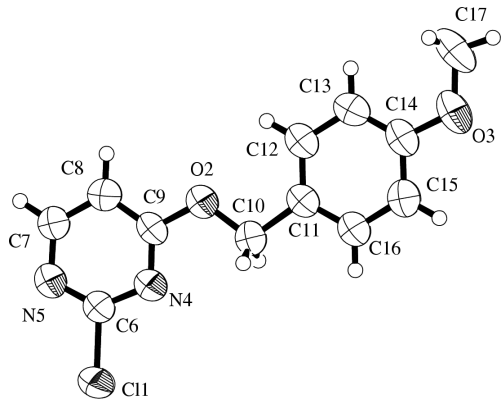


Figure 1
The molecular structure of (I), with displacement ellipsoids plotted at the 50% probability level.

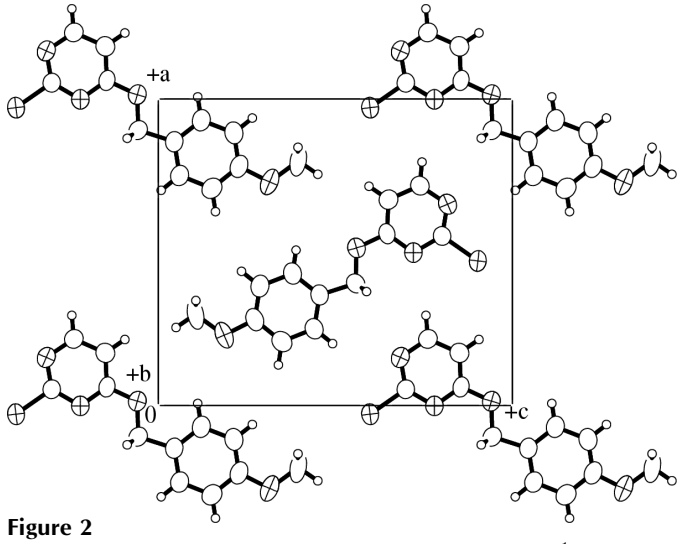


Figure 2
The molecular arrangement of (I) on a mirror plane at $y = \frac{1}{4}$.

Data collection

Rigaku AFC-7*R* diffractometer	$R_{int} = 0.024$
ω–2θ scans	$\theta_{max} = 27.5^\circ$
Absorption correction: by integration (Coppens *et al.*, 1965)	$h = -6 \rightarrow 15$
	$k = -3 \rightarrow 8$
$T_{min} = 0.861$, $T_{max} = 0.875$	$l = 0 \rightarrow 18$
1660 measured reflections	3 standard reflections
1477 independent reflections	every 150 reflections
1017 reflections with $I > 2\sigma(I)$	intensity decay: 3.9%

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.141$	$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.417$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.79$	$\Delta\rho_{max} = 0.43$ e Å^{-3}
1477 reflections	$\Delta\rho_{min} = -1.06$ e Å^{-3}
103 parameters	

Table 1
Selected geometric parameters (Å).

Cl1—C6	1.751 (6)	N5—C7	1.336 (8)
N4—C6	1.330 (7)	C7—C8	1.381 (8)
N4—C9	1.310 (6)	C8—C9	1.370 (8)
N5—C6	1.296 (8)		

X-ray intensity data were measured for $+h,+k,+l$ ($\theta < 27.5^\circ$) and for $-h,\pm k,+l$ ($\theta < 11^\circ$). During the data collection (over 20 h), the standard reflections showed a decay of 3.9%, for which a correction was applied. The possible space groups suggested by the systematic absences were *Pnma* and $Pn2_1a$ (a non-standard setting of $Pna2_1$). Some direct-methods calculations assuming space group $Pn2_1$a indicated a flat molecular structure; a similar result was obtained for *Pnma*. The C and N atoms of the pyrimidine ring were assigned based on their atomic displacement parameters, and the H atoms bonded to C atoms were confirmed from difference syntheses. All H atoms were positioned geometrically and fixed with $U_{iso}(H) = 1.2U_{eq}$(parent atom). The $R(F)$ and $wR(F^2)$ values are relatively high, which may be due to the positional disorder of the Cl atom. In fact, the displacement parameters of the Cl atom are strongly anisotropic, and U_{22}/U_{11} and U_{22}/U_{33} are 3.5 and 5.3, respectively. However, tentative refinement with the Cl atom shifted from the mirror plane did not improve the R values.

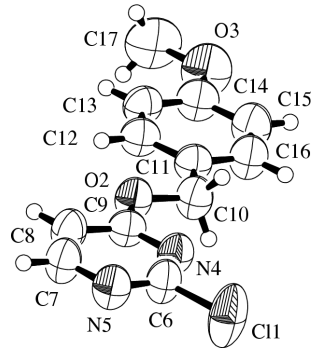


Figure 3
Another view of the molecular structure of (I), showing the large anisotropy in the displacement parameters of the Cl atom.

Data collection: *WinAFC Diffractometer Control Software* (Rigaku, 1999); cell refinement: *WinAFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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