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1,4-Bis(6-chloropyrimidin-4-yloxy)-benzene

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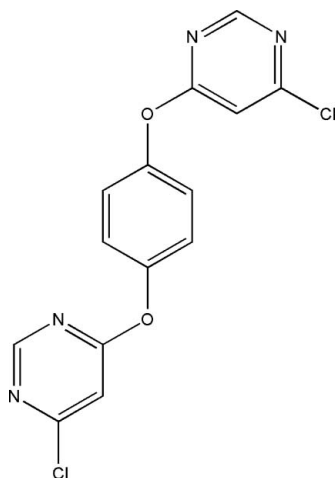
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.037; wR factor = 0.105; data-to-parameter ratio = 10.7.

In the title compound, $\text{C}_{14}\text{H}_8\text{Cl}_2\text{N}_4\text{O}_2$, all atoms of the 6-chloropyrimidin-4-yloxy group and the C atoms at the *para* positions of the central benzene ring lie on a crystallographic mirror plane. The complete benzene ring is generated by the mirror plane and hence the dihedral angles between the pyrimidine rings and the benzene ring are exactly 90° . The crystal structure is stabilized by weak $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds.

Related literature

 For background information, see: Halim *et al.* (1999); Meng & Huang (2000); Maes *et al.* (2003); Friend *et al.* (1999).


Experimental

Crystal data

$\text{C}_{14}\text{H}_8\text{Cl}_2\text{N}_4\text{O}_2$	$V = 1432.02$ (7) Å ³
$M_r = 335.14$	$Z = 4$
Monoclinic, $C2/m$	Mo $K\alpha$ radiation
$a = 19.0760$ (5) Å	$\mu = 0.47$ mm ⁻¹
$b = 6.9693$ (2) Å	$T = 298$ (2) K
$c = 10.7893$ (3) Å	$0.20 \times 0.10 \times 0.10$ mm
$\beta = 93.301$ (3)°	

Data collection

Bruker SMART CCD diffractometer	1372 independent reflections
Absorption correction: none	1156 reflections with $I > 2\sigma(I)$
6918 measured reflections	$R_{\text{int}} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	128 parameters
$wR(F^2) = 0.105$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.22$ e Å ⁻³
1372 reflections	$\Delta\rho_{\text{min}} = -0.25$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}10-\text{H}10\cdots\text{O}2^i$	0.93	2.57	3.463 (3)	161
$\text{C}3-\text{H}3\cdots\text{N}2^{ii}$	0.93	2.48	3.355 (3)	157

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2746).

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supplementary materials

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1,4-Bis(6-chloropyrimidin-4-yloxy)benzene

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Comment

In recent publications it has been shown that polymers consisting of heterocyclic building blocks can be used in organic light-emitting diodes (LEDs) because of their electroluminescent properties. (Halim *et al.*, 1999; Friend *et al.*, 1999; Meng & Huang, 2000; Maes *et al.*, 2003). We report here the synthesis and crystal structure the title compound (I) (Fig. 1). All atoms of the 6-chloropyrimidin-4-yloxy group and the C atoms at the para positions of the central benzene ring lie on a crystallographic mirror plane. The symmetry complete benzene ring is generated by the mirror plane and hence the dihedral angles between the pyrimidine rings and the benzene ring are exactly 90°. The crystal packing is stabilized by weak intermolecular hydrogen bonds interactions. Each molecule acts as a donor and acceptor to form C–H···O and C–H···N hydrogen bonds with two other symmetry related molecules, forming a chain run parallel to [101] (Fig. 2; Table 2).

Experimental

Hydroquinone 0.55 g (5 mmol) was dissolved in 50 ml CH₃CN, the solution was stirred at room temperature for 0.5 h with an excess of anhydrous K₂CO₃ (2.5 equiv.). After another 0.5 h of reflux, 4,6-dichloropyrimidine 2.59 g (10 mmol) in 20 ml CH₃CN was added and the mixture was refluxed for 4 h. After evaporation of the solvent, water was added and the mixture was extracted with CH₂Cl₂ and dried over MgSO₄. The products were purified by column chromatography (hexanes/ethyl acetate, 5:1) and obtained as white solids. Colourless block-shaped crystals were obtained by evaporation of CH₂Cl₂.

Refinement

After being located in the difference map, all H-atoms were fixed geometrically at ideal positions and allowed to ride on the parent C atoms with C–H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$.

Figures

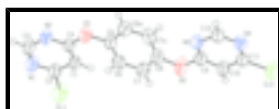


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level [Symmetry codes: (a) $x, -y, z$].

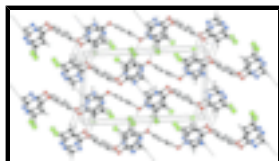


Fig. 2. Part of the crystal structure with hydrogen bonds shown by dashed lines.

1,4-Bis(6-chloropyrimidin-4-yloxy)benzene

Crystal data

$C_{14}H_8Cl_2N_4O_2$

$M_r = 335.14$

Monoclinic, $C2/m$

Hall symbol: $-C 2y$

$a = 19.0760$ (5) Å

$b = 6.9693$ (2) Å

$c = 10.7893$ (3) Å

$\beta = 93.301$ (3)°

$V = 1432.02$ (7) Å³

$Z = 4$

$F_{000} = 680$

$D_x = 1.555$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3581 reflections

$\theta = 2.8$ – 27.8 °

$\mu = 0.47$ mm⁻¹

$T = 298$ (2) K

Block, colorless

$0.20 \times 0.10 \times 0.10$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

φ and ω scans

Absorption correction: none

6918 measured reflections

1372 independent reflections

1156 reflections with $I > 2\sigma(I)$

$R_{int} = 0.050$

$\theta_{max} = 25.0$ °

$\theta_{min} = 1.9$ °

$h = -22$ → 22

$k = -8$ → 7

$l = -12$ → 12

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.105$

$S = 1.07$

1372 reflections

128 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.2906P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.22$ e Å⁻³

$\Delta\rho_{min} = -0.25$ e Å⁻³

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.0062 (13)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.34316 (10)	0.0000	0.22381 (18)	0.0495 (5)
H1	0.3002	0.0000	0.1786	0.059*
C2	0.40565 (11)	0.0000	0.16857 (19)	0.0543 (6)
C3	0.46576 (12)	0.0000	0.3504 (2)	0.0666 (7)
H3	0.5089	0.0000	0.3950	0.080*
C4	0.34857 (11)	0.0000	0.35261 (19)	0.0467 (5)
C5	0.22554 (11)	0.0000	0.36269 (19)	0.0512 (6)
C6	0.19419 (8)	0.1719 (3)	0.33512 (14)	0.0589 (4)
H6	0.2162	0.2867	0.3578	0.071*
C7	0.12865 (9)	0.1712 (3)	0.27244 (15)	0.0636 (5)
H7	0.1058	0.2859	0.2523	0.076*
C8	0.09825 (11)	0.0000	0.24084 (19)	0.0581 (7)
C9	-0.02669 (11)	0.0000	0.21792 (19)	0.0529 (6)
C10	-0.08588 (12)	0.0000	0.1385 (2)	0.0570 (6)
H10	-0.0839	0.0000	0.0526	0.068*
C11	-0.14794 (11)	0.0000	0.1964 (2)	0.0525 (5)
C12	-0.09188 (11)	0.0000	0.3830 (2)	0.0553 (6)
H12	-0.0939	0.0000	0.4689	0.066*
Cl1	0.40498 (4)	0.0000	0.00858 (5)	0.0916 (3)
Cl2	-0.22616 (3)	0.0000	0.10747 (6)	0.0829 (3)
N1	0.46804 (10)	0.0000	0.22748 (18)	0.0676 (6)
N2	0.40951 (9)	0.0000	0.41739 (17)	0.0572 (5)
N3	-0.02774 (9)	0.0000	0.33990 (16)	0.0533 (5)
N4	-0.15287 (9)	0.0000	0.31826 (17)	0.0566 (5)
O1	0.29285 (8)	0.0000	0.42430 (14)	0.0619 (5)
O2	0.03558 (8)	0.0000	0.16482 (14)	0.0793 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0393 (11)	0.0687 (14)	0.0399 (11)	0.000	-0.0027 (9)	0.000
C2	0.0453 (12)	0.0777 (15)	0.0400 (11)	0.000	0.0022 (9)	0.000

supplementary materials

C3	0.0407 (12)	0.109 (2)	0.0495 (13)	0.000	-0.0062 (10)	0.000
C4	0.0421 (11)	0.0573 (12)	0.0403 (10)	0.000	0.0002 (8)	0.000
C5	0.0397 (11)	0.0797 (16)	0.0345 (10)	0.000	0.0054 (8)	0.000
C6	0.0543 (9)	0.0708 (11)	0.0520 (9)	-0.0059 (8)	0.0062 (7)	0.0019 (8)
C7	0.0528 (9)	0.0824 (12)	0.0561 (9)	0.0116 (9)	0.0073 (7)	0.0109 (9)
C8	0.0390 (11)	0.102 (2)	0.0340 (10)	0.000	0.0072 (8)	0.000
C9	0.0434 (11)	0.0779 (15)	0.0378 (11)	0.000	0.0067 (9)	0.000
C10	0.0484 (12)	0.0836 (16)	0.0389 (11)	0.000	0.0011 (9)	0.000
C11	0.0440 (11)	0.0612 (14)	0.0519 (12)	0.000	-0.0009 (9)	0.000
C12	0.0515 (13)	0.0762 (16)	0.0389 (11)	0.000	0.0078 (9)	0.000
Cl1	0.0605 (4)	0.1762 (9)	0.0387 (4)	0.000	0.0072 (3)	0.000
Cl2	0.0467 (4)	0.1308 (7)	0.0695 (5)	0.000	-0.0106 (3)	0.000
N1	0.0415 (10)	0.1109 (17)	0.0503 (11)	0.000	0.0013 (9)	0.000
N2	0.0441 (10)	0.0836 (14)	0.0429 (9)	0.000	-0.0057 (8)	0.000
N3	0.0452 (10)	0.0774 (13)	0.0375 (9)	0.000	0.0050 (8)	0.000
N4	0.0447 (10)	0.0750 (13)	0.0509 (11)	0.000	0.0095 (8)	0.000
O1	0.0430 (8)	0.1051 (13)	0.0376 (7)	0.000	0.0019 (6)	0.000
O2	0.0403 (9)	0.1598 (19)	0.0379 (8)	0.000	0.0040 (7)	0.000

Geometric parameters (Å, °)

C1—C2	1.363 (3)	C7—C8	1.361 (2)
C1—C4	1.388 (3)	C7—H7	0.9300
C1—H1	0.9300	C8—C7 ⁱ	1.361 (2)
C2—N1	1.317 (3)	C8—O2	1.410 (3)
C2—Cl1	1.725 (2)	C9—N3	1.317 (3)
C3—N2	1.328 (3)	C9—O2	1.348 (3)
C3—N1	1.329 (3)	C9—C10	1.377 (3)
C3—H3	0.9300	C10—C11	1.370 (3)
C4—N2	1.322 (3)	C10—H10	0.9300
C4—O1	1.350 (2)	C11—N4	1.323 (3)
C5—C6 ⁱ	1.364 (2)	C11—Cl2	1.727 (2)
C5—C6	1.364 (2)	C12—N4	1.322 (3)
C5—O1	1.411 (3)	C12—N3	1.334 (3)
C6—C7	1.387 (2)	C12—H12	0.9300
C6—H6	0.9300		
C2—C1—C4	114.92 (19)	C7 ⁱ —C8—C7	122.4 (2)
C2—C1—H1	122.5	C7 ⁱ —C8—O2	118.70 (11)
C4—C1—H1	122.5	C7—C8—O2	118.70 (11)
N1—C2—C1	125.3 (2)	N3—C9—O2	119.27 (19)
N1—C2—Cl1	115.94 (16)	N3—C9—C10	124.21 (19)
C1—C2—Cl1	118.76 (17)	O2—C9—C10	116.52 (19)
N2—C3—N1	128.1 (2)	C11—C10—C9	114.6 (2)
N2—C3—H3	115.9	C11—C10—H10	122.7
N1—C3—H3	115.9	C9—C10—H10	122.7
N2—C4—O1	113.24 (18)	N4—C11—C10	124.4 (2)
N2—C4—C1	122.83 (19)	N4—C11—Cl2	116.32 (17)
O1—C4—C1	123.93 (19)	C10—C11—Cl2	119.24 (18)

C6 ⁱ —C5—C6	122.8 (2)	N4—C12—N3	127.8 (2)
C6 ⁱ —C5—O1	118.57 (10)	N4—C12—H12	116.1
C6—C5—O1	118.57 (10)	N3—C12—H12	116.1
C5—C6—C7	118.36 (17)	C2—N1—C3	113.6 (2)
C5—C6—H6	120.8	C4—N2—C3	115.20 (19)
C7—C6—H6	120.8	C9—N3—C12	114.53 (19)
C8—C7—C6	118.97 (17)	C12—N4—C11	114.45 (18)
C8—C7—H7	120.5	C4—O1—C5	117.07 (16)
C6—C7—H7	120.5	C9—O2—C8	119.40 (16)
C4—C1—C2—N1	0.0	C1—C4—N2—C3	0.0
C4—C1—C2—C11	180.0	N1—C3—N2—C4	0.0
C2—C1—C4—N2	0.0	O2—C9—N3—C12	180.0
C2—C1—C4—O1	180.0	C10—C9—N3—C12	0.0
C6 ⁱ —C5—C6—C7	-2.3 (3)	N4—C12—N3—C9	0.0
O1—C5—C6—C7	178.76 (14)	N3—C12—N4—C11	0.0
C5—C6—C7—C8	-0.1 (3)	C10—C11—N4—C12	0.0
C6—C7—C8—C7 ⁱ	2.4 (3)	C12—C11—N4—C12	180.0
C6—C7—C8—O2	-172.75 (14)	N2—C4—O1—C5	180.0
N3—C9—C10—C11	0.0	C1—C4—O1—C5	0.0
O2—C9—C10—C11	180.0	C6 ⁱ —C5—O1—C4	90.53 (16)
C9—C10—C11—N4	0.0	C6—C5—O1—C4	-90.53 (16)
C9—C10—C11—C12	180.0	N3—C9—O2—C8	0.0
C1—C2—N1—C3	0.0	C10—C9—O2—C8	180.0
C11—C2—N1—C3	180.0	C7 ⁱ —C8—O2—C9	92.31 (16)
N2—C3—N1—C2	0.0	C7—C8—O2—C9	-92.31 (16)
O1—C4—N2—C3	180.0		

Symmetry codes: (i) $x, -y, z$.

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10—H10 ⁱⁱ —O2 ⁱⁱ	0.93	2.57	3.463 (3)	161
C3—H3 ⁱⁱⁱ —N2 ⁱⁱⁱ	0.93	2.48	3.355 (3)	157

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

Fig. 1

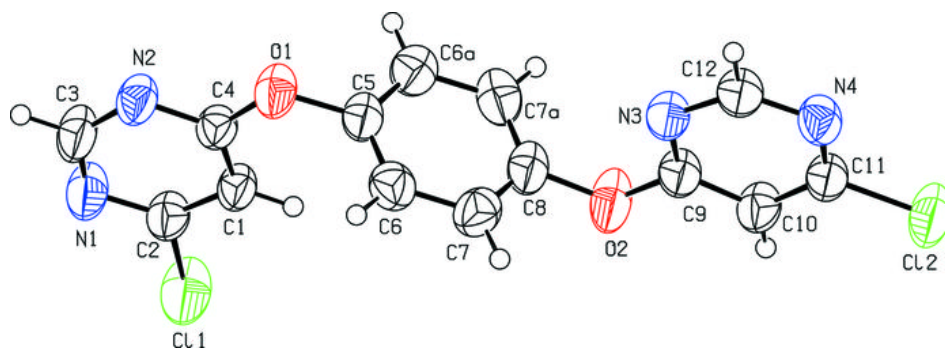


Fig. 2

