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

## Wen Ma[a]* and Hongyan Li[b]

[a]College of Chemistry and Chemical Engineering, Xinyang Normal Universty, Xinyang, Henan 464000, People's Republic of China, and [b]College of Life Science, Xinyang Normal Universty, Xinyang, Henan 464000, People's Republic of China
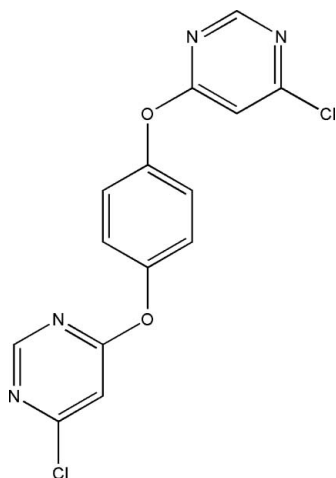Correspondence e-mail: maw0809@yahoo.com.cn

In the title compound, $C_{14}H_8Cl_2N_4O_2$, 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 C—H···O and C—H···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

| | |
|---|---|
| $C_{14}H_8Cl_2N_4O_2$ | $V = 1432.02$ (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 | $R_{int} = 0.050$ |

### Refinement

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

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C10—H10···O2$^i$ | 0.93 | 2.57 | 3.463 (3) | 161 |
| C3—H3···N2$^{ii}$ | 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: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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).

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

## References

Bruker (2007). *SAINT-Plus* and *SMART*. Bruker AXS, Inc., Madison, Wisconsin, USA.

Friend, R. H., Gymer, R. W., Holmes, A. B., Burrroughes, J. H., Marks, R. N., Taliani, C., Bradley, D. C. C., Dos Santos, D. A., Brédas, J. L., Lögdlund, M. & Salaneck, W. R. (1999). *Nature (London)*, **397**, 121–C128.

Halim, M., Pillow, J. N. G., Samuel, I. D. W. & Burn, P. L. (1999). *Adv. Mater.* **11**, 371–C374.

Maes, W., Amabilino, D. B. & Dehaen, W. (2003). *Tetrahedron*, **59**, 3937–C3943.

Meng, H. & Huang, W. J. (2000). *Org. Chem.* **65**, 3894–C3901.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

**supplementary materials**

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

**W. Ma** and **H. Li**

### 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 $CH_3CN$, the solution was stirred at room temperature for 0.5 h with an excess of anhydrous $K_2CO_3$(2.5 equiv.). After another 0.5 h of reflux, 4,6-dichloropyrimidine2.59 g (10 mmol) in 20 ml $CH_3CN$ was added and the mixture was refluxed for 4 h. After evaporation of the solvent, water was added and the mixture was extracted with $CH_2Cl_2$ and dried over $MgSO_4$. The products were purified by column chromatography (hexanes/ethyl acetate, 5:1) and obtained as white solids. Colourless block-shapped crystals were obtained by evaporation of $CH_2Cl_2$.

### 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 = 0.93Å and $U_{iso}(H)= 1.2U_{eq}$.
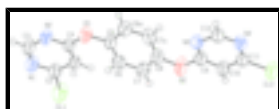
### Figures


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*].
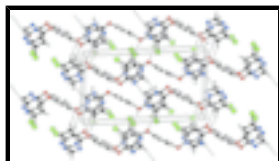

Fig. 2. Part of the crystal structure with hydrogen bonds shown by dashed lines.

# supplementary materials

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

*Crystal data*

| | |
|---|---|
| $C_{14}H_8Cl_2N_4O_2$ | $F_{000} = 680$ |
| $M_r = 335.14$ | $D_x = 1.555$ Mg m$^{-3}$ |
| Monoclinic, $C2/m$ | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| Hall symbol: -C 2y | Cell parameters from 3581 reflections |
| $a = 19.0760$ (5) Å | $\theta = 2.8–27.8º$ |
| $b = 6.9693$ (2) Å | $\mu = 0.47$ mm$^{-1}$ |
| $c = 10.7893$ (3) Å | $T = 298$ (2) K |
| $\beta = 93.301$ (3)º | Block, colorless |
| $V = 1432.02$ (7) Å$^3$ | $0.20 \times 0.10 \times 0.10$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD<br>diffractometer | 1156 reflections with $I > 2\sigma(I)$' |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.050$ |
| Monochromator: graphite | $\theta_{max} = 25.0º$ |
| $T = 298(2)$ K | $\theta_{min} = 1.9º$ |
| $\varphi$ and $\omega$ scans | $h = -22 \rightarrow 22$ |
| Absorption correction: none | $k = -8 \rightarrow 7$ |
| 6918 measured reflections | $l = -12 \rightarrow 12$ |
| 1372 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.037$ | $w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.2906P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.105$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.07$ | $\Delta\rho_{max} = 0.22$ e Å$^{-3}$ |
| 1372 reflections | $\Delta\rho_{min} = -0.25$ e Å$^{-3}$ |
| 128 parameters | Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.0062 (13) |
| Secondary atom site location: difference Fourier map | |

*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 ($Å^2$)*

|      | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|------|------|------|------|------|
| 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 ($Å^2$)*

|      | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|------|------|------|------|------|------|------|
| 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 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 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<sup>i</sup>—C5—C6 | 122.8 (2) | N4—C12—N3 | 127.8 (2) |
| C6<sup>i</sup>—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—Cl1 | 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<sup>i</sup>—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<sup>i</sup> | 2.4 (3) | Cl2—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<sup>i</sup>—C5—O1—C4 | 90.53 (16) |
| C9—C10—C11—N4 | 0.0 | C6—C5—O1—C4 | −90.53 (16) |
| C9—C10—C11—Cl2 | 180.0 | N3—C9—O2—C8 | 0.0 |
| C1—C2—N1—C3 | 0.0 | C10—C9—O2—C8 | 180.0 |
| Cl1—C2—N1—C3 | 180.0 | C7<sup>i</sup>—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⋯*A* | *D*—H | H⋯*A* | *D*⋯*A* | *D*—H⋯*A* |
|---|---|---|---|---|
| C10—H10⋯O2<sup>ii</sup> | 0.93 | 2.57 | 3.463 (3) | 161 |
| C3—H3⋯N2<sup>iii</sup> | 0.93 | 2.48 | 3.355 (3) | 157 |

Symmetry codes: (ii) −*x*, *y*, −*z*; (iii) −*x*+1, *y*, −*z*+1.

**Fig. 1**

**Fig. 2**