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## 2-[4-(Chloromethyl)phenoxy]-4,6-dimethoxypyrimidine

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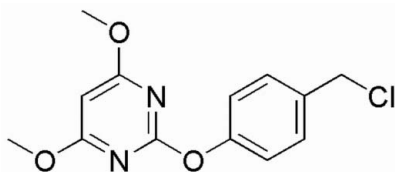
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 Key indicators: single-crystal X-ray study;  $T = 113$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.054;  $wR$  factor = 0.134; data-to-parameter ratio = 13.6.

The title compound,  $\text{C}_{13}\text{H}_{13}\text{ClN}_2\text{O}_3$ , was synthesized in the course of the search for novel bioactive pyrimidine derivatives. The C—O—C angle at the phenoxy O atom is widened to  $119.87$  ( $18$ )°. The dihedral angle between the pyrimidine and benzene rings is  $64.2$  ( $3$ )°.

## Related literature

For the biological activity of pyrimidine derivatives, see: Amin *et al.* (2011); Chen *et al.* (2009); Popova *et al.* (1999); Sagi *et al.* (2011); Stec *et al.* (2008). For related structures of 2-phenoxy-pyrimidines, see: Shah Bakhtiar *et al.* (2009a,b). For standard bond lengths, see: Allen *et al.* (1987).



## Experimental

## Crystal data

 $\text{C}_{13}\text{H}_{13}\text{ClN}_2\text{O}_3$ 
 $M_r = 280.70$ 

 Monoclinic,  $P2_1/c$   
 $a = 8.3998$  (17) Å  
 $b = 23.145$  (5) Å  
 $c = 7.7967$  (16) Å  
 $\beta = 117.28$  (3)°  
 $V = 1347.2$  (6) Å<sup>3</sup>
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.29$  mm<sup>-1</sup>  
 $T = 113$  K  
 $0.30 \times 0.25 \times 0.20$  mm

## Data collection

 Rigaku SCXmini diffractometer  
 Absorption correction: multi-scan  
 (*CrystalClear*; Rigaku, 2008)  
 $T_{\min} = 0.912$ ,  $T_{\max} = 0.942$ 

 11307 measured reflections  
 2358 independent reflections  
 1899 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.039$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.134$   
 $S = 1.09$   
 2358 reflections

 174 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.62$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.52$  e Å<sup>-3</sup>

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YK2059).

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## supplementary materials

*Acta Cryst.* (2012). E68, o2109 [doi:10.1107/S1600536812026220]

**2-[4-(Chloromethyl)phenoxy]-4,6-dimethoxypyrimidine**

Shi-Hong Shen, Kui Hu, Li Sun, Xiao-Long Fan and Hong Dai

**Comment**

In the past few years, many pyrimidine derivatives have drawn much attention in agrochemical and medicinal research because of their diverse bioactivities such as fungicidal, herbicidal and pharmacological activities (Popova *et al.*, 1999; Stec *et al.*, 2008; Chen *et al.*, 2009; Amin *et al.*, 2011; Sagi *et al.*, 2011). In the search of novel biologically active molecules, we have synthesized new pyrimidines. We report here the crystal structure of the target compound. It contains two planar groups, the benzene ring (C7/C8/C9/C10/C11/C12), and the substituted pyrimidine ring (N1/C1/C2/C3/N2/C6) (Fig.1). All bond lengths and angles in the title compound lie within normal ranges (Allen *et al.*, 1987) and are similar to those observed in the related 2-phenoxy pyrimidines (Shah Bakhtiar *et al.*, 2009*a,b*). The plane of pyrimidine ring makes a dihedral angle of 64.2 (3)° with the plane of benzene ring.

**Experimental**

[4-(4,6-dimethoxypyrimidin-2-yloxy)phenyl]methanol (5 mmol) was dissolved in 50 ml of CH<sub>2</sub>Cl<sub>2</sub>. Thionyl chloride (5.5 mmol) was then added dropwise, while cooling on an ice bath. The resulting solution was heated to 298 K for 3 h, and the mixture was poured into 50 ml of ice water. The organic layer was washed with saturated brine (3 x 30 ml) and dried over anhydrous sodium sulfate. The solvent was evaporated under reduced pressure, and the residue was recrystallized from a mixture of petroleum ether/ethyl acetate to obtain colourless crystals. Mp: 354–356 K.

**Refinement**

All H atoms were placed in calculated positions, with C—H = 0.93, 0.96 and 0.97 Å for CH, CH<sub>3</sub> and CH<sub>2</sub> groups, respectively, and included in the final cycles of refinement using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

**Computing details**

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear* (Rigaku, 2008); data reduction: *CrystalClear* (Rigaku, 2008); 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: *SHELXTL* (Sheldrick, 2008).

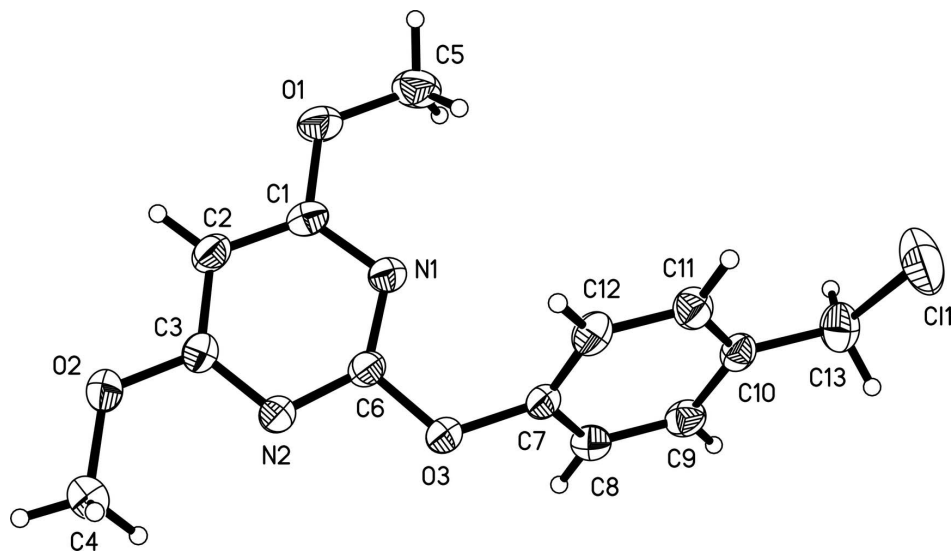


Figure 1

View of the title compound showing atom labelling scheme. Thermal ellipsoids drawn at the 30% probability level.

## 2-[4-(Chloromethyl)phenoxy]-4,6-dimethoxypyrimidine

### Crystal data

$C_{13}H_{13}ClN_2O_3$

$M_r = 280.70$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 8.3998\ (17)\ \text{\AA}$

$b = 23.145\ (5)\ \text{\AA}$

$c = 7.7967\ (16)\ \text{\AA}$

$\beta = 117.28\ (3)^\circ$

$V = 1347.2\ (6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 584$

$D_x = 1.384\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 11126 reflections

$\theta = 3.1\text{--}27.6^\circ$

$\mu = 0.29\ \text{mm}^{-1}$

$T = 113\ \text{K}$

Prism, colourless

$0.30 \times 0.25 \times 0.20\ \text{mm}$

### Data collection

Rigaku SCXmini

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution:  $14.22\ \text{pixels mm}^{-1}$

$\omega$  and  $\varphi$  scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2008)

$T_{\min} = 0.912$ ,  $T_{\max} = 0.942$

11307 measured reflections

2358 independent reflections

1899 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 3.1^\circ$

$h = -9 \rightarrow 9$

$k = -27 \rightarrow 27$

$l = -9 \rightarrow 9$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.134$

$S = 1.09$

2358 reflections

174 parameters

0 restraints

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

$$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.8134P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.62 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.52 \text{ e } \text{\AA}^{-3}$$

*Special details*

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	1.0641 (3)	0.96956 (10)	0.2361 (4)	0.0471 (6)
C2	1.2474 (3)	0.97248 (11)	0.3080 (4)	0.0517 (6)
H2	1.3138	1.0040	0.3785	0.062*
C3	1.3259 (3)	0.92547 (10)	0.2683 (3)	0.0446 (6)
C4	1.5903 (4)	0.87913 (13)	0.2908 (5)	0.0638 (8)
H4A	1.5472	0.8434	0.3166	0.096*
H4B	1.7177	0.8815	0.3689	0.096*
H4C	1.5624	0.8807	0.1569	0.096*
C5	0.7922 (4)	1.01267 (13)	0.2028 (5)	0.0692 (8)
H5A	0.7339	0.9971	0.0744	0.104*
H5B	0.7470	1.0507	0.2032	0.104*
H5C	0.7689	0.9882	0.2882	0.104*
C6	1.0600 (3)	0.88216 (10)	0.1124 (3)	0.0437 (6)
C7	0.7881 (3)	0.82754 (10)	-0.0485 (4)	0.0454 (6)
C8	0.7325 (3)	0.78267 (11)	0.0275 (4)	0.0524 (6)
H8	0.8158	0.7593	0.1241	0.063*
C9	0.5507 (4)	0.77293 (11)	-0.0426 (4)	0.0509 (6)
H9	0.5123	0.7425	0.0072	0.061*
C10	0.4246 (3)	0.80752 (11)	-0.1854 (3)	0.0448 (6)
C11	0.4845 (3)	0.85228 (11)	-0.2603 (4)	0.0507 (6)
H11	0.4017	0.8758	-0.3565	0.061*
C12	0.6662 (4)	0.86220 (11)	-0.1929 (4)	0.0520 (6)
H12	0.7054	0.8919	-0.2446	0.062*
C13	0.2290 (4)	0.79504 (14)	-0.2462 (4)	0.0653 (8)
H13A	0.2013	0.8076	-0.1442	0.078*
H13B	0.2104	0.7536	-0.2602	0.078*
Cl1	0.07844 (11)	0.82848 (5)	-0.46320 (16)	0.1037 (4)
N1	0.9659 (3)	0.92397 (8)	0.1384 (3)	0.0452 (5)
N2	1.2352 (2)	0.87930 (9)	0.1678 (3)	0.0450 (5)
O1	0.9816 (3)	1.01557 (8)	0.2666 (3)	0.0647 (6)
O2	1.5055 (2)	0.92689 (8)	0.3357 (3)	0.0565 (5)
O3	0.9740 (2)	0.83341 (7)	0.0165 (3)	0.0576 (5)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0597 (15)	0.0372 (13)	0.0493 (14)	0.0004 (11)	0.0293 (13)	-0.0034 (11)
C2	0.0567 (15)	0.0407 (14)	0.0558 (15)	-0.0089 (11)	0.0242 (13)	-0.0105 (12)
C3	0.0450 (13)	0.0441 (14)	0.0448 (14)	-0.0059 (11)	0.0206 (11)	-0.0004 (11)
C4	0.0492 (15)	0.0636 (18)	0.079 (2)	0.0021 (13)	0.0293 (15)	-0.0057 (15)
C5	0.073 (2)	0.0577 (18)	0.091 (2)	0.0119 (15)	0.0504 (18)	-0.0029 (16)
C6	0.0476 (13)	0.0373 (13)	0.0476 (14)	-0.0038 (10)	0.0232 (11)	-0.0047 (10)
C7	0.0420 (12)	0.0390 (13)	0.0553 (15)	-0.0055 (10)	0.0223 (11)	-0.0138 (11)
C8	0.0535 (15)	0.0410 (14)	0.0590 (16)	0.0046 (11)	0.0227 (13)	0.0017 (12)
C9	0.0594 (15)	0.0390 (14)	0.0620 (16)	-0.0044 (11)	0.0345 (14)	0.0017 (12)
C10	0.0468 (13)	0.0457 (14)	0.0438 (13)	-0.0065 (11)	0.0224 (11)	-0.0096 (11)
C11	0.0515 (14)	0.0496 (15)	0.0443 (14)	-0.0016 (11)	0.0163 (12)	0.0027 (11)
C12	0.0577 (15)	0.0454 (15)	0.0546 (15)	-0.0115 (12)	0.0271 (13)	-0.0012 (12)
C13	0.0529 (16)	0.077 (2)	0.0670 (18)	-0.0118 (14)	0.0280 (14)	-0.0035 (16)
C11	0.0508 (5)	0.1230 (9)	0.1064 (8)	-0.0071 (5)	0.0095 (5)	0.0305 (6)
N1	0.0478 (11)	0.0396 (11)	0.0497 (12)	0.0002 (9)	0.0236 (10)	-0.0043 (9)
N2	0.0444 (11)	0.0408 (11)	0.0518 (12)	-0.0035 (9)	0.0238 (10)	-0.0049 (9)
O1	0.0698 (12)	0.0451 (11)	0.0882 (15)	0.0026 (9)	0.0442 (11)	-0.0150 (10)
O2	0.0443 (9)	0.0555 (11)	0.0666 (12)	-0.0082 (8)	0.0227 (9)	-0.0125 (9)
O3	0.0451 (10)	0.0445 (10)	0.0826 (13)	-0.0061 (8)	0.0288 (9)	-0.0208 (9)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C1—N1	1.341 (3)	C6—O3	1.363 (3)
C1—O1	1.350 (3)	C7—C12	1.379 (4)
C1—C2	1.378 (4)	C7—C8	1.378 (4)
C2—C3	1.379 (3)	C7—O3	1.410 (3)
C2—H2	0.9300	C8—C9	1.385 (4)
C3—N2	1.337 (3)	C8—H8	0.9300
C3—O2	1.351 (3)	C9—C10	1.386 (4)
C4—O2	1.442 (3)	C9—H9	0.9300
C4—H4A	0.9600	C10—C11	1.391 (3)
C4—H4B	0.9600	C10—C13	1.514 (3)
C4—H4C	0.9600	C11—C12	1.387 (4)
C5—O1	1.435 (3)	C11—H11	0.9300
C5—H5A	0.9600	C12—H12	0.9300
C5—H5B	0.9600	C13—C11	1.760 (3)
C5—H5C	0.9600	C13—H13A	0.9700
C6—N1	1.322 (3)	C13—H13B	0.9700
C6—N2	1.332 (3)		
N1—C1—O1	119.3 (2)	C7—C8—C9	118.8 (2)
N1—C1—C2	123.4 (2)	C7—C8—H8	120.6
O1—C1—C2	117.2 (2)	C9—C8—H8	120.6
C1—C2—C3	115.5 (2)	C8—C9—C10	121.4 (2)
C1—C2—H2	122.3	C8—C9—H9	119.3
C3—C2—H2	122.3	C10—C9—H9	119.3
N2—C3—O2	118.8 (2)	C9—C10—C11	118.5 (2)

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N2—C3—C2	124.0 (2)	C9—C10—C13	117.5 (2)
O2—C3—C2	117.2 (2)	C11—C10—C13	124.0 (2)
O2—C4—H4A	109.5	C12—C11—C10	120.7 (2)
O2—C4—H4B	109.5	C12—C11—H11	119.6
H4A—C4—H4B	109.5	C10—C11—H11	119.6
O2—C4—H4C	109.5	C7—C12—C11	119.3 (2)
H4A—C4—H4C	109.5	C7—C12—H12	120.4
H4B—C4—H4C	109.5	C11—C12—H12	120.4
O1—C5—H5A	109.5	C10—C13—C11	114.6 (2)
O1—C5—H5B	109.5	C10—C13—H13A	108.6
H5A—C5—H5B	109.5	C11—C13—H13A	108.6
O1—C5—H5C	109.5	C10—C13—H13B	108.6
H5A—C5—H5C	109.5	C11—C13—H13B	108.6
H5B—C5—H5C	109.5	H13A—C13—H13B	107.6
N1—C6—N2	129.5 (2)	C6—N1—C1	114.1 (2)
N1—C6—O3	119.1 (2)	C6—N2—C3	113.5 (2)
N2—C6—O3	111.5 (2)	C1—O1—C5	118.6 (2)
C12—C7—C8	121.2 (2)	C3—O2—C4	118.3 (2)
C12—C7—O3	121.5 (2)	C6—O3—C7	119.87 (18)
C8—C7—O3	117.1 (2)		

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