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Methyl 2-[2-(6-chloropyrimidin-4-yl-oxy)phenyl]-3,3-dimethoxypropanoate

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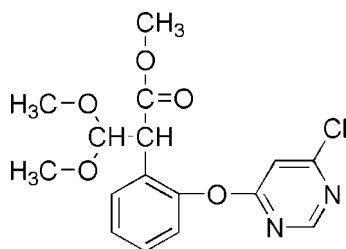
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 Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.078; wR factor = 0.173; data-to-parameter ratio = 14.9.

In the title compound, $\text{C}_{16}\text{H}_{17}\text{ClN}_2\text{O}_5$, the dihedral angle between the aromatic rings is $77.36(4)^\circ$. An intramolecular $\text{C}-\text{H}\cdots\text{O}$ interaction results in the formation of a planar [r.m.s. deviation = $0.103(2)$ Å] five-membered ring, which is oriented at a dihedral angle of $4.84(4)^\circ$ with respect to the adjacent benzene ring. In the crystal structure, weak intermolecular $\text{C}-\text{H}\cdots\pi$ interactions are found.

Related literature

 For a related structure, see: Bowden & Brown (1996). For bond-length data, see: Allen *et al.* (1987).


Experimental

Crystal data

 $\text{C}_{16}\text{H}_{17}\text{ClN}_2\text{O}_5$
 $M_r = 352.77$
 Triclinic, $P\bar{1}$
 $a = 9.5030(19)$ Å

 $b = 10.051(2)$ Å
 $c = 11.162(2)$ Å
 $\alpha = 101.24(3)^\circ$
 $\beta = 108.47(3)^\circ$
 $\gamma = 113.42(3)^\circ$
 $V = 862.6(5)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation

 $\mu = 0.25$ mm⁻¹
 $T = 294$ K
 $0.20 \times 0.20 \times 0.05$ mm

Data collection

 Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.952$, $T_{\max} = 0.988$
 3346 measured reflections

 3140 independent reflections
 1427 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 3 standard reflections
 frequency: 120 min
 intensity decay: 1%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.078$
 $wR(F^2) = 0.173$
 $S = 1.07$
 3140 reflections

 211 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.43$ e Å⁻³
 $\Delta\rho_{\min} = -0.45$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6A}\cdots\text{O5}$	0.98	2.25	2.777 (6)	113
$\text{C1}-\text{H1B}\cdots\text{Cg2}^i$	0.96	2.97	3.696 (5)	134
$\text{C16}-\text{H16A}\cdots\text{Cg1}^i$	0.93	2.85	3.661 (4)	146

 Symmetry code: (i) $-x + 1, -y + 1, -z$. Cg1 and Cg2 are centroids of the $\text{C7}-\text{C12}$ and $\text{N1}/\text{N2}/\text{C13}-\text{C16}$ rings, respectively.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2749).

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supplementary materials

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Methyl 2-[2-(6-chloropyrimidin-4-yloxy)phenyl]-3,3-dimethoxypropanoate

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Comment

The title compound can be used as an intermediate in the preparation of azoxystrobin, which is an important fungicide (Bowden & Brown, 1996). We report herein the crystal structure of the title compound, which is of interest to us in the field.

In the molecule of the title compound (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C7-C12) and B (N1/N2/C13-C16) are, of course, planar and the dihedral angle between them is $A/B = 77.36(4)^\circ$. Intramolecular C-H \cdots O interaction (Table 1) results in the formation of a planar five-membered ring C (O5/C6-C8/H6A), which is oriented with respect to the adjacent ring A at a dihedral angle of $A/C = 4.84(4)^\circ$.

In the crystal structure, weak C—H \cdots π interactions (Table 1) are found.

Experimental

The title compound was prepared according to a literature method (Bowden & Brown, 1996). Crystals suitable for X-ray analysis were obtained by dissolving the title compound in methanol and evaporating the solvent slowly at room temperature for 8 d.

Refinement

H atoms were positioned geometrically with C-H = 0.93, 0.98 and 0.96 Å for aromatic, methine and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H atoms.

Figures

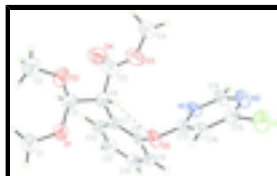


Fig. 1. The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bond is shown as dashed line.

Methyl 2-[2-(6-chloropyrimidin-4-yloxy)phenyl]-3,3-dimethoxypropanoate

Crystal data

$\text{C}_{16}\text{H}_{17}\text{ClN}_2\text{O}_5$

$M_r = 352.77$

Triclinic, $P\bar{1}$

$Z = 2$

$F_{000} = 368$

$D_x = 1.358 \text{ Mg m}^{-3}$

supplementary materials

Hall symbol: -P 1
 $a = 9.5030 (19) \text{ \AA}$
 $b = 10.051 (2) \text{ \AA}$
 $c = 11.162 (2) \text{ \AA}$
 $\alpha = 101.24 (3)^\circ$
 $\beta = 108.47 (3)^\circ$
 $\gamma = 113.42 (3)^\circ$
 $V = 862.6 (5) \text{ \AA}^3$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Cell parameters from 25 reflections
 $\theta = 9\text{--}12^\circ$
 $\mu = 0.25 \text{ mm}^{-1}$
 $T = 294 \text{ K}$
Needle, colorless
 $0.20 \times 0.20 \times 0.05 \text{ mm}$

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294 \text{ K}$

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.952$, $T_{\max} = 0.988$

3346 measured reflections

3140 independent reflections

1427 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 25.3^\circ$

$\theta_{\min} = 2.1^\circ$

$h = 0 \rightarrow 11$

$k = -12 \rightarrow 11$

$l = -13 \rightarrow 12$

3 standard reflections

every 120 min

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.078$

$wR(F^2) = 0.173$

$S = 1.07$

3140 reflections

211 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

$$w = 1/[\sigma^2(F_o^2) + (0.060P)^2]$$

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

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

$\Delta\rho_{\max} = 0.43 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.45 \text{ e \AA}^{-3}$

Extinction correction: none

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.16863 (16)	0.54848 (17)	0.23573 (15)	0.0918 (5)
O1	-0.7131 (4)	0.4178 (4)	-0.3468 (3)	0.0806 (10)
O2	-0.6406 (5)	0.2829 (5)	-0.4869 (4)	0.1119 (14)
O3	-0.5192 (5)	0.0798 (5)	-0.3027 (4)	0.1150 (14)
O4	-0.7857 (7)	-0.0410 (6)	-0.4408 (5)	0.1371 (18)
O5	-0.4458 (3)	0.3794 (3)	-0.0203 (3)	0.0658 (9)
N1	-0.3799 (5)	0.1982 (4)	0.0434 (4)	0.0631 (11)
N2	-0.0880 (5)	0.2702 (5)	0.1614 (4)	0.0727 (11)
C1	-0.8435 (9)	0.4438 (9)	-0.4217 (6)	0.131 (3)
H1B	-0.8231	0.5446	-0.3730	0.196*
H1C	-0.9517	0.3648	-0.4345	0.196*
H1D	-0.8443	0.4398	-0.5086	0.196*
C2	-0.6907 (10)	0.2967 (8)	-0.6060 (6)	0.140 (3)
H2B	-0.6124	0.2972	-0.6432	0.210*
H2C	-0.6938	0.3925	-0.5950	0.210*
H2D	-0.8026	0.2105	-0.6664	0.210*
C3	-0.7194 (7)	0.2788 (5)	-0.4025 (5)	0.0710 (14)
H3A	-0.8400	0.2004	-0.4578	0.085*
C4	-0.5126 (8)	-0.0639 (7)	-0.3384 (6)	0.1142 (16)
H4A	-0.3983	-0.0432	-0.2896	0.171*
H4B	-0.5469	-0.1044	-0.4343	0.171*
H4C	-0.5883	-0.1389	-0.3148	0.171*
C5	-0.6598 (11)	0.0736 (9)	-0.3573 (8)	0.1142 (16)
C6	-0.6513 (6)	0.2253 (6)	-0.2981 (5)	0.0688 (13)
H6A	-0.5298	0.3025	-0.2460	0.083*
C7	-0.7250 (6)	0.2132 (5)	-0.1948 (5)	0.0517 (11)
C8	-0.6183 (5)	0.2823 (4)	-0.0593 (5)	0.0464 (10)
C9	-0.6753 (6)	0.2753 (5)	0.0390 (5)	0.0609 (13)
H9A	-0.5986	0.3259	0.1305	0.073*
C10	-0.8481 (7)	0.1921 (6)	0.0000 (6)	0.0699 (14)
H10A	-0.8884	0.1850	0.0656	0.084*
C11	-0.9602 (6)	0.1202 (6)	-0.1340 (7)	0.0793 (16)
H11A	-1.0770	0.0644	-0.1607	0.095*
C12	-0.8968 (6)	0.1316 (5)	-0.2302 (5)	0.0691 (14)
H12A	-0.9732	0.0823	-0.3218	0.083*
C13	-0.3283 (6)	0.3428 (5)	0.0417 (4)	0.0536 (11)
C14	-0.2533 (6)	0.1731 (5)	0.1042 (5)	0.0720 (14)
H14A	-0.2858	0.0737	0.1068	0.086*
C15	-0.0453 (6)	0.4133 (5)	0.1578 (4)	0.0586 (12)
C16	-0.1620 (5)	0.4550 (5)	0.0963 (4)	0.0575 (12)
H16A	-0.1295	0.5538	0.0923	0.069*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0588 (8)	0.0939 (11)	0.1023 (11)	0.0264 (7)	0.0267 (8)	0.0357 (8)
O1	0.104 (3)	0.067 (2)	0.066 (2)	0.047 (2)	0.023 (2)	0.0268 (18)
O2	0.161 (4)	0.177 (4)	0.075 (3)	0.115 (3)	0.076 (3)	0.085 (3)
O3	0.118 (3)	0.119 (3)	0.127 (3)	0.084 (3)	0.050 (3)	0.033 (3)
O4	0.137 (4)	0.122 (3)	0.136 (4)	0.083 (3)	0.037 (3)	0.013 (3)
O5	0.0501 (19)	0.0528 (18)	0.097 (2)	0.0245 (16)	0.0248 (17)	0.0461 (18)
N1	0.065 (2)	0.042 (2)	0.092 (3)	0.0293 (19)	0.037 (2)	0.035 (2)
N2	0.064 (3)	0.073 (3)	0.099 (3)	0.039 (2)	0.039 (2)	0.050 (3)
C1	0.169 (7)	0.195 (7)	0.096 (5)	0.143 (6)	0.064 (5)	0.062 (5)
C2	0.239 (9)	0.150 (6)	0.093 (5)	0.133 (6)	0.082 (6)	0.067 (5)
C3	0.100 (4)	0.053 (3)	0.064 (3)	0.036 (3)	0.039 (3)	0.028 (3)
C4	0.127 (4)	0.126 (4)	0.125 (4)	0.092 (3)	0.056 (3)	0.047 (3)
C5	0.127 (4)	0.126 (4)	0.125 (4)	0.092 (3)	0.056 (3)	0.047 (3)
C6	0.084 (3)	0.078 (3)	0.058 (3)	0.046 (3)	0.034 (3)	0.032 (2)
C7	0.059 (3)	0.044 (2)	0.060 (3)	0.029 (2)	0.027 (3)	0.027 (2)
C8	0.051 (3)	0.038 (2)	0.057 (3)	0.027 (2)	0.020 (3)	0.025 (2)
C9	0.090 (4)	0.045 (3)	0.053 (3)	0.037 (3)	0.030 (3)	0.024 (2)
C10	0.085 (4)	0.061 (3)	0.104 (5)	0.044 (3)	0.063 (4)	0.053 (3)
C11	0.048 (3)	0.063 (3)	0.120 (5)	0.021 (3)	0.032 (4)	0.044 (4)
C12	0.068 (4)	0.054 (3)	0.065 (3)	0.023 (3)	0.019 (3)	0.014 (3)
C13	0.062 (3)	0.048 (3)	0.062 (3)	0.032 (2)	0.031 (2)	0.025 (2)
C14	0.068 (3)	0.053 (3)	0.113 (4)	0.039 (3)	0.041 (3)	0.044 (3)
C15	0.062 (3)	0.059 (3)	0.060 (3)	0.025 (3)	0.036 (3)	0.027 (2)
C16	0.053 (3)	0.048 (3)	0.068 (3)	0.021 (2)	0.027 (3)	0.026 (2)

Geometric parameters (\AA , $^\circ$)

C1—C15	1.720 (5)	C3—H3A	0.9800
O1—C1	1.410 (6)	C4—H4A	0.9600
O1—C3	1.383 (5)	C4—H4B	0.9600
O2—C2	1.318 (6)	C4—H4C	0.9600
O2—C3	1.373 (5)	C5—C6	1.498 (8)
O3—C4	1.453 (6)	C6—C7	1.528 (6)
O3—C5	1.250 (7)	C6—H6A	0.9800
O4—C5	1.186 (8)	C7—C8	1.363 (5)
O5—C8	1.393 (4)	C7—C12	1.376 (6)
O5—C13	1.341 (5)	C8—C9	1.370 (6)
N1—C13	1.343 (5)	C9—C10	1.378 (6)
N1—C14	1.327 (5)	C9—H9A	0.9300
N2—C14	1.316 (5)	C10—C11	1.361 (7)
N2—C15	1.343 (5)	C10—H10A	0.9300
C1—H1B	0.9600	C11—C12	1.390 (7)
C1—H1C	0.9600	C11—H11A	0.9300
C1—H1D	0.9600	C12—H12A	0.9300
C2—H2B	0.9600	C13—C16	1.359 (5)

C2—H2C	0.9600	C14—H14A	0.9300
C2—H2D	0.9600	C15—C16	1.370 (5)
C3—C6	1.452 (6)	C16—H16A	0.9300
C3—O1—C1	117.8 (4)	C5—C6—C7	108.8 (4)
C2—O2—C3	126.5 (5)	C3—C6—H6A	106.1
C5—O3—C4	117.8 (5)	C5—C6—H6A	106.1
C13—O5—C8	121.1 (3)	C7—C6—H6A	106.1
C14—N1—C13	114.1 (4)	C8—C7—C12	116.5 (4)
C14—N2—C15	114.3 (4)	C8—C7—C6	119.9 (4)
O1—C1—H1B	109.5	C12—C7—C6	123.6 (4)
O1—C1—H1C	109.5	C7—C8—C9	123.0 (4)
H1B—C1—H1C	109.5	C7—C8—O5	117.6 (4)
O1—C1—H1D	109.5	C9—C8—O5	118.9 (4)
H1B—C1—H1D	109.5	C8—C9—C10	118.9 (4)
H1C—C1—H1D	109.5	C8—C9—H9A	120.5
O2—C2—H2B	109.5	C10—C9—H9A	120.5
O2—C2—H2C	109.5	C11—C10—C9	120.4 (5)
H2B—C2—H2C	109.5	C11—C10—H10A	119.8
O2—C2—H2D	109.5	C9—C10—H10A	119.8
H2B—C2—H2D	109.5	C10—C11—C12	118.7 (5)
H2C—C2—H2D	109.5	C10—C11—H11A	120.6
O2—C3—O1	114.2 (4)	C12—C11—H11A	120.6
O2—C3—C6	109.9 (4)	C7—C12—C11	122.4 (5)
O1—C3—C6	111.5 (4)	C7—C12—H12A	118.8
O2—C3—H3A	107.0	C11—C12—H12A	118.8
O1—C3—H3A	107.0	O5—C13—N1	119.0 (4)
C6—C3—H3A	107.0	O5—C13—C16	117.2 (4)
O3—C4—H4A	109.5	N1—C13—C16	123.9 (4)
O3—C4—H4B	109.5	N2—C14—N1	128.5 (4)
H4A—C4—H4B	109.5	N2—C14—H14A	115.7
O3—C4—H4C	109.5	N1—C14—H14A	115.7
H4A—C4—H4C	109.5	N2—C15—C16	123.5 (4)
H4B—C4—H4C	109.5	N2—C15—C1	116.9 (4)
O4—C5—O3	123.8 (7)	C16—C15—C1	119.6 (4)
O4—C5—C6	124.5 (7)	C13—C16—C15	115.6 (4)
O3—C5—C6	111.6 (7)	C13—C16—H16A	122.2
C3—C6—C5	112.0 (5)	C15—C16—H16A	122.2
C3—C6—C7	117.1 (4)		
C2—O2—C3—O1	-67.6 (7)	C13—O5—C8—C7	-114.9 (4)
C2—O2—C3—C6	166.3 (6)	C13—O5—C8—C9	72.8 (5)
C1—O1—C3—O2	87.6 (6)	C7—C8—C9—C10	1.4 (6)
C1—O1—C3—C6	-147.2 (5)	O5—C8—C9—C10	173.3 (3)
C4—O3—C5—O4	-3.6 (11)	C8—C9—C10—C11	-1.0 (6)
C4—O3—C5—C6	173.0 (5)	C9—C10—C11—C12	0.4 (7)
O2—C3—C6—C5	-53.2 (6)	C8—C7—C12—C11	0.5 (6)
O1—C3—C6—C5	179.1 (5)	C6—C7—C12—C11	178.8 (4)
O2—C3—C6—C7	-179.9 (4)	C10—C11—C12—C7	-0.1 (7)
O1—C3—C6—C7	52.5 (6)	C8—O5—C13—N1	12.4 (6)

supplementary materials

O4—C5—C6—C3	-54.2 (10)	C8—O5—C13—C16	-169.3 (4)
O3—C5—C6—C3	129.3 (6)	C14—N1—C13—O5	178.4 (4)
O4—C5—C6—C7	76.8 (8)	C14—N1—C13—C16	0.2 (6)
O3—C5—C6—C7	-99.7 (6)	C15—N2—C14—N1	0.9 (8)
C3—C6—C7—C8	-128.5 (4)	C13—N1—C14—N2	0.0 (8)
C5—C6—C7—C8	103.3 (5)	C14—N2—C15—C16	-2.0 (7)
C3—C6—C7—C12	53.2 (6)	C14—N2—C15—Cl	177.9 (4)
C5—C6—C7—C12	-75.0 (6)	O5—C13—C16—C15	-179.4 (4)
C12—C7—C8—C9	-1.1 (6)	N1—C13—C16—C15	-1.2 (7)
C6—C7—C8—C9	-179.5 (4)	N2—C15—C16—C13	2.2 (7)
C12—C7—C8—O5	-173.1 (3)	Cl—C15—C16—C13	-177.7 (3)
C6—C7—C8—O5	8.4 (5)		

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C6—H6A \cdots O5	0.98	2.25	2.777 (6)	113
C1—H1B \cdots Cg2 ⁱ	0.96	2.97	3.696 (5)	134
C16—H16A \cdots Cg1 ⁱ	0.93	2.85	3.661 (4)	146

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

Fig. 1

