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Ethyl 1-[(4-acetyl-2-methoxyphenoxy)-methyl]cyclopropane-1-carboxylate

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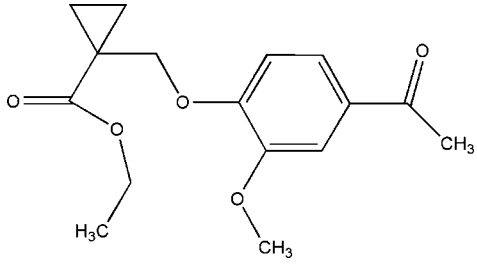
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.062; wR factor = 0.168; data-to-parameter ratio = 14.2.

In the title compound, $C_{16}H_{20}O_5$, the dihedral angle between the planar rings, *viz*. benzene and cyclopropane, is 52.1 (2)°. Molecules are connected in the crystal *via* weak intermolecular C—H···O hydrogen bonds, forming chains in the [001] direction.

Related literature

For details of the synthesis, see: Chen (2008).



Experimental

Crystal data

$C_{16}H_{20}O_5$ M_r = 292.32
Monoclinic, $P2_1/c$
a = 12.663 (3) Å
b = 8.5020 (17) Å
c = 14.676 (3) Å
β = 107.25 (3)°
V = 1509.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.981, T_{max} = 0.991
2874 measured reflections
2732 independent reflections
1473 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.168
S = 0.93
2732 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···O1^{i}	0.93	2.56	3.329 (4)	140

Symmetry code: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2213).

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supporting information

Acta Cryst. (2009). E**65**, o305 [doi:10.1107/S1600536809000956]

Ethyl 1-[(4-acetyl-2-methoxyphenoxy)methyl]cyclopropane-1-carboxylate

Ting Tang, Lei Gao, Hong-Sheng Jia, Ya-Ming Wu and Hong-Fei Ma

S1. Comment

The title compound, (I), is one of the most important intermediates in the synthesis of 7-[(1-aminocyclopropyl)methoxy]-*N*-(1*H*-indol-5-yl)-6-methoxyquinolin-4-amine, which has advantageous pharmacological properties and inhibits the activity of protein tyrosine kinases (Chen, 2008). We report here the crystal structure of (I).

All bond lengths and angles are within expected ranges. Both benzene and cyclopropane rings are planar, and make a dihedral angle of 52.1 (2)° (rings C3···C8 and C11···C13). Molecules are linked together *via* intermolecular C—H···O hydrogen bonds, which may be effective to the stabilization of the crystal structure.

S2. Experimental

The title compound was synthesized using a method similar to that reported recently (Chen, 2008). The crystals were obtained by evaporating the acetone slowly at room temperature for about 14 d.

S3. Refinement

H atoms were positioned geometrically, with C—H = 0.93 (aromatic), 0.96 (methyl) or 0.97 Å (methylene), and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(carrier C), where x = 1.5 for methyl groups and x = 1.2 otherwise. Methyl groups were allowed to rotate about their C—C bonds.

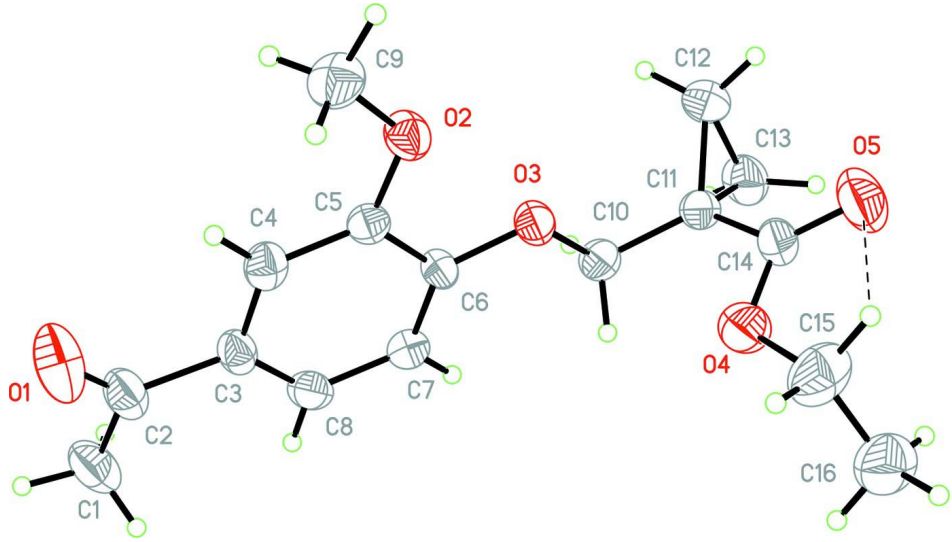

Figure 1

A drawing of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. Intramolecular C—H···O hydrogen bond is shown as a dashed line.

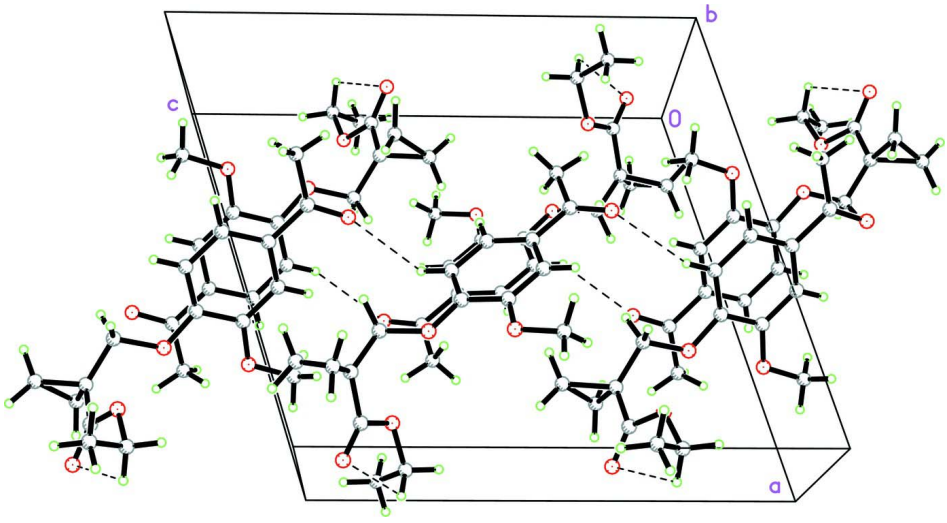

Figure 2

A packing diagram for (I). C—H···O hydrogen bonds are shown as dashed lines.

Ethyl 1-[(4-acetyl-2-methoxyphenoxy)methyl]cyclopropane-1-carboxylate

Crystal data

$C_{16}H_{20}O_5$
$M_r = 292.32$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 12.663$ (3) Å
$b = 8.5020$ (17) Å
$c = 14.676$ (3) Å
$\beta = 107.25$ (3)°
$V = 1509.0$ (5) Å^3
$Z = 4$
$F(000) = 624$
$D_x = 1.287$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.10$ mm^{-1}
$T = 298$ K
Plate, colorless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.981$, $T_{max} = 0.991$
2874 measured reflections
2732 independent reflections
1473 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 25.3°$, $\theta_{min} = 1.7°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 10$
$l = -17 \rightarrow 17$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.168$
$S = 0.93$
2732 reflections
193 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.15P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5800 (2)	0.2003 (4)	0.7367 (2)	0.0809 (10)
C1	0.7006 (3)	0.3398 (5)	0.6698 (3)	0.0673 (13)
H1A	0.7445	0.3495	0.7353	0.101*
H1B	0.6823	0.4426	0.6428	0.101*
H1C	0.7418	0.2838	0.6348	0.101*
O2	0.26250 (19)	−0.0087 (3)	0.46428 (15)	0.0472 (6)
C2	0.5977 (3)	0.2525 (5)	0.6646 (3)	0.0522 (10)
O3	0.27923 (17)	0.1243 (3)	0.31287 (15)	0.0431 (6)
C3	0.5143 (3)	0.2248 (4)	0.5700 (2)	0.0403 (8)
O4	0.1020 (2)	0.3513 (3)	0.20147 (19)	0.0602 (8)
C4	0.4262 (3)	0.1219 (4)	0.5639 (2)	0.0417 (9)
H4A	0.4197	0.0740	0.6189	0.050*
C5	0.3492 (3)	0.0907 (4)	0.4776 (2)	0.0360 (8)
O5	−0.0032 (2)	0.2019 (4)	0.0853 (2)	0.0883 (11)
C6	0.3593 (3)	0.1627 (4)	0.3945 (2)	0.0375 (8)
C7	0.4462 (3)	0.2622 (4)	0.3995 (2)	0.0415 (9)
H7A	0.4534	0.3086	0.3442	0.050*
C8	0.5237 (3)	0.2938 (4)	0.4871 (3)	0.0464 (9)
H8A	0.5823	0.3616	0.4901	0.056*
C9	0.2450 (3)	−0.0810 (5)	0.5458 (3)	0.0565 (11)
H9A	0.1861	−0.1562	0.5259	0.085*
H9B	0.2256	−0.0022	0.5849	0.085*
H9C	0.3115	−0.1334	0.5817	0.085*
C10	0.2902 (3)	0.1857 (4)	0.2248 (2)	0.0413 (9)
H10A	0.3545	0.1404	0.2118	0.050*
H10B	0.2994	0.2990	0.2292	0.050*
C11	0.1888 (3)	0.1450 (4)	0.1471 (2)	0.0414 (8)
C12	0.1746 (3)	−0.0231 (5)	0.1132 (3)	0.0567 (11)
H12A	0.2312	−0.0979	0.1453	0.068*
H12B	0.1001	−0.0653	0.0930	0.068*
C13	0.2038 (3)	0.0959 (5)	0.0513 (2)	0.0590 (11)
H13A	0.1471	0.1268	−0.0064	0.071*
H13B	0.2784	0.0942	0.0461	0.071*
C14	0.0850 (3)	0.2319 (5)	0.1393 (3)	0.0497 (10)
C15	0.0056 (4)	0.4466 (6)	0.2022 (3)	0.0750 (14)
H15A	−0.0610	0.3843	0.1777	0.090*
H15B	0.0113	0.4756	0.2675	0.090*
C16	−0.0033 (4)	0.5888 (6)	0.1448 (4)	0.0806 (14)
H16A	−0.0661	0.6490	0.1485	0.121*
H16B	−0.0123	0.5605	0.0797	0.121*
H16C	0.0626	0.6506	0.1687	0.121*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0617 (18)	0.131 (3)	0.0465 (16)	0.0004 (19)	0.0099 (14)	−0.0115 (18)
C1	0.052 (2)	0.071 (3)	0.068 (3)	−0.009 (2)	0.001 (2)	−0.023 (2)
O2	0.0434 (13)	0.0557 (16)	0.0413 (13)	−0.0115 (13)	0.0106 (10)	0.0021 (12)
C2	0.048 (2)	0.057 (3)	0.045 (2)	0.007 (2)	0.0029 (18)	−0.015 (2)
O3	0.0417 (13)	0.0481 (15)	0.0364 (12)	−0.0089 (12)	0.0065 (10)	0.0034 (12)
C3	0.0379 (19)	0.043 (2)	0.0363 (18)	0.0070 (17)	0.0048 (15)	−0.0084 (17)
O4	0.0503 (15)	0.0526 (17)	0.0738 (18)	0.0038 (14)	0.0125 (13)	−0.0084 (15)
C4	0.0370 (18)	0.046 (2)	0.042 (2)	0.0080 (17)	0.0112 (15)	−0.0006 (17)
C5	0.0375 (18)	0.0311 (18)	0.0394 (18)	0.0050 (16)	0.0113 (15)	−0.0037 (15)
O5	0.0523 (18)	0.089 (2)	0.101 (2)	0.0043 (17)	−0.0115 (17)	−0.026 (2)
C6	0.0354 (18)	0.036 (2)	0.0351 (18)	−0.0015 (16)	0.0019 (14)	−0.0046 (15)
C7	0.045 (2)	0.035 (2)	0.044 (2)	0.0015 (17)	0.0112 (16)	0.0007 (17)
C8	0.041 (2)	0.035 (2)	0.058 (2)	−0.0016 (17)	0.0063 (17)	−0.0061 (18)
C9	0.058 (2)	0.061 (3)	0.057 (2)	−0.006 (2)	0.0264 (19)	0.010 (2)
C10	0.045 (2)	0.041 (2)	0.0385 (19)	−0.0018 (17)	0.0129 (15)	0.0033 (16)
C11	0.050 (2)	0.037 (2)	0.0351 (18)	−0.0027 (17)	0.0087 (15)	0.0003 (16)
C12	0.066 (3)	0.043 (2)	0.056 (2)	−0.002 (2)	0.0093 (19)	−0.0094 (19)
C13	0.071 (3)	0.066 (3)	0.037 (2)	−0.005 (2)	0.0111 (18)	−0.005 (2)
C14	0.042 (2)	0.051 (2)	0.047 (2)	−0.0062 (19)	0.0001 (18)	0.005 (2)
C15	0.061 (3)	0.069 (3)	0.102 (4)	0.020 (2)	0.034 (3)	0.010 (3)
C16	0.060 (3)	0.071 (3)	0.112 (4)	0.007 (3)	0.026 (3)	0.002 (3)

Geometric parameters (Å, º)

O1—C2	1.227 (4)	C8—H8A	0.9300
C1—C2	1.482 (5)	C9—H9A	0.9600
C1—H1A	0.9600	C9—H9B	0.9600
C1—H1B	0.9600	C9—H9C	0.9600
C1—H1C	0.9600	C10—C11	1.483 (4)
O2—C5	1.352 (4)	C10—H10A	0.9700
O2—C9	1.419 (4)	C10—H10B	0.9700
C2—C3	1.493 (5)	C11—C14	1.482 (5)
O3—C6	1.360 (3)	C11—C12	1.506 (5)
O3—C10	1.438 (4)	C11—C13	1.532 (5)
C3—C8	1.387 (5)	C12—C13	1.478 (5)
C3—C4	1.399 (5)	C12—H12A	0.9700
O4—C14	1.339 (4)	C12—H12B	0.9700
O4—C15	1.468 (4)	C13—H13A	0.9700
C4—C5	1.376 (4)	C13—H13B	0.9700
C4—H4A	0.9300	C15—C16	1.459 (6)
C5—C6	1.405 (5)	C15—H15A	0.9700
O5—C14	1.189 (4)	C15—H15B	0.9700
C6—C7	1.373 (4)	C16—H16A	0.9600
C7—C8	1.393 (5)	C16—H16B	0.9600
C7—H7A	0.9300	C16—H16C	0.9600

C2—C1—H1A	109.5	C11—C10—H10A	110.0
C2—C1—H1B	109.5	O3—C10—H10B	110.0
H1A—C1—H1B	109.5	C11—C10—H10B	110.0
C2—C1—H1C	109.5	H10A—C10—H10B	108.4
H1A—C1—H1C	109.5	C14—C11—C10	119.3 (3)
H1B—C1—H1C	109.5	C14—C11—C12	115.5 (3)
C5—O2—C9	118.1 (3)	C10—C11—C12	117.9 (3)
O1—C2—C1	121.3 (3)	C14—C11—C13	114.4 (3)
O1—C2—C3	119.0 (4)	C10—C11—C13	117.0 (3)
C1—C2—C3	119.7 (4)	C12—C11—C13	58.2 (2)
C6—O3—C10	117.3 (2)	C13—C12—C11	61.7 (2)
C8—C3—C4	118.9 (3)	C13—C12—H12A	117.6
C8—C3—C2	121.8 (3)	C11—C12—H12A	117.6
C4—C3—C2	119.3 (3)	C13—C12—H12B	117.6
C14—O4—C15	117.2 (3)	C11—C12—H12B	117.6
C5—C4—C3	120.9 (3)	H12A—C12—H12B	114.7
C5—C4—H4A	119.5	C12—C13—C11	60.0 (2)
C3—C4—H4A	119.5	C12—C13—H13A	117.8
O2—C5—C4	125.3 (3)	C11—C13—H13A	117.8
O2—C5—C6	115.2 (3)	C12—C13—H13B	117.8
C4—C5—C6	119.5 (3)	C11—C13—H13B	117.8
O3—C6—C7	124.9 (3)	H13A—C13—H13B	114.9
O3—C6—C5	115.1 (3)	O5—C14—O4	123.1 (4)
C7—C6—C5	120.0 (3)	O5—C14—C11	125.5 (4)
C6—C7—C8	120.1 (3)	O4—C14—C11	111.4 (3)
C6—C7—H7A	119.9	C16—C15—O4	112.0 (4)
C8—C7—H7A	119.9	C16—C15—H15A	109.2
C3—C8—C7	120.5 (3)	O4—C15—H15A	109.2
C3—C8—H8A	119.7	C16—C15—H15B	109.2
C7—C8—H8A	119.7	O4—C15—H15B	109.2
O2—C9—H9A	109.5	H15A—C15—H15B	107.9
O2—C9—H9B	109.5	C15—C16—H16A	109.5
H9A—C9—H9B	109.5	C15—C16—H16B	109.5
O2—C9—H9C	109.5	H16A—C16—H16B	109.5
H9A—C9—H9C	109.5	C15—C16—H16C	109.5
H9B—C9—H9C	109.5	H16A—C16—H16C	109.5
O3—C10—C11	108.3 (3)	H16B—C16—H16C	109.5
O3—C10—H10A	110.0		

O1—C2—C3—C8	173.4 (4)	C2—C3—C8—C7	178.2 (3)
C1—C2—C3—C8	−7.6 (5)	C6—C7—C8—C3	0.3 (5)
O1—C2—C3—C4	−9.0 (5)	C6—O3—C10—C11	−173.7 (3)
C1—C2—C3—C4	170.0 (3)	O3—C10—C11—C14	74.4 (4)
C8—C3—C4—C5	−0.8 (5)	O3—C10—C11—C12	−74.2 (4)
C2—C3—C4—C5	−178.5 (3)	O3—C10—C11—C13	−140.7 (3)
C9—O2—C5—C4	3.6 (5)	C14—C11—C12—C13	104.1 (4)
C9—O2—C5—C6	−177.7 (3)	C10—C11—C12—C13	−106.1 (4)

C3—C4—C5—O2	178.9 (3)	C14—C11—C13—C12	−106.0 (4)
C3—C4—C5—C6	0.2 (5)	C10—C11—C13—C12	107.5 (4)
C10—O3—C6—C7	3.6 (5)	C15—O4—C14—O5	0.7 (6)
C10—O3—C6—C5	−175.5 (3)	C15—O4—C14—C11	−179.3 (3)
O2—C5—C6—O3	1.2 (4)	C10—C11—C14—O5	−173.6 (4)
C4—C5—C6—O3	179.9 (3)	C12—C11—C14—O5	−24.2 (6)
O2—C5—C6—C7	−178.1 (3)	C13—C11—C14—O5	40.7 (5)
C4—C5—C6—C7	0.7 (5)	C10—C11—C14—O4	6.4 (5)
O3—C6—C7—C8	179.9 (3)	C12—C11—C14—O4	155.8 (3)
C5—C6—C7—C8	−1.0 (5)	C13—C11—C14—O4	−139.3 (3)
C4—C3—C8—C7	0.6 (5)	C14—O4—C15—C16	−96.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···O1^{i}	0.93	2.56	3.329 (4)	140
C15—H15*A*···O5	0.97	2.32	2.678 (6)	101

Symmetry code: (i) *x*, −*y*+1/2, *z*−1/2.