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7-Methoxy-3-(4-methoxyphenyl)-4-oxo-4*H*-chromen-5-yl acetate

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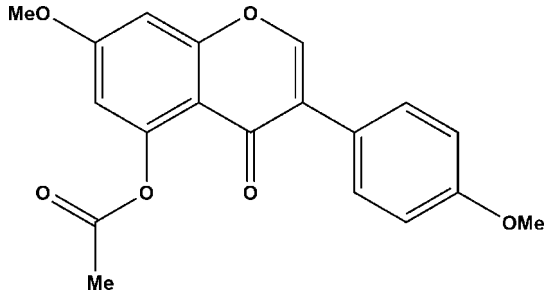
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.179; data-to-parameter ratio = 16.2.

In the genistein-related title compound, $C_{19}H_{16}O_6$, the dihedral angle between the two benzene-ring planes is 52.81 (9)°.

Related literature

For background literature, see: Kim *et al.* (2004); Li *et al.* (2006). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{19}H_{16}O_6$
M_r = 340.32
Monoclinic, $P2_1/c$
a = 14.2159 (16) Å
b = 12.7207 (15) Å
c = 9.2200 (11) Å
β = 101.668 (2)°
V = 1632.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.970, T_{max} = 0.986
11970 measured reflections
3720 independent reflections
2142 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.180
S = 1.03
3720 reflections
229 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2519).

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supplementary materials

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7-Methoxy-3-(4-methoxyphenyl)-4-oxo-4*H*-chromen-5-yl acetate

H.-Q. Li, L. Shi, Z.-P. Xiao and H.-L. Zhu

Comment

Genistein derivatives play an important role in organic chemistry (Kim *et al.*, 2004; Li *et al.*, 2006). In the genistein derived title compound, (I) (Fig. 1), the bond lengths and angles are within their normal ranges (Allen *et al.*, 1987). The dihedral angle between the least-squares planes of the two benzene rings (C1—C6 and C9—C14) is 52.81 (9) °. The crystal packing for (I) is stabilized by van der Waals forces.

Experimental

Genistein (0.27 g, 1 mmol), iodomethane (0.26 ml, 2 mmol) and potassium carbonate (0.14 g, 1 mmol) in 50 ml of dry acetone were sonicated. After the completion of reaction, the mixture was cooled to room temperature followed by filtration. The filtrate was distilled to give a yellow solid and the solid was dissolved in 15 ml dry pyridine, then acetic anhydride (0.22 ml, 11 mmol) was added, and the mixed solution was stirred at room temperature for 24 h. After that the solution was poured into a 10% solution of aqueous hydrochloric acid (50 ml). The white deposits that precipitated were separated from the solvents by filtration. They were washed with aqueous saturated sodium bicarbonate twice. The solid was dissolved in acetone (15 ml) and stirred for about 10 min to give a clear solution. After keeping the solution in air for 10 d, colorless blocks of (I) were formed at the bottom of the vesssl on slow evaporation of the solvent. They were collected, washed three times with acetone and dried in a vacuum desiccator using $CaCl_2$. The compound was isolated in 90% yield.

Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

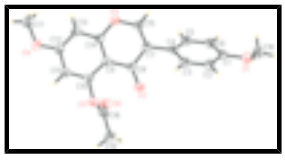


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids for the non-hydrogen atoms.

7-Methoxy-3-(4-methoxyphenyl)-4-oxo-4*H*-chromen-5-yl acetate

Crystal data

$C_{19}H_{16}O_6$	F_{000} = 712
M_r = 340.32	D_x = 1.384 Mg m^{-3}

Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1520 reflections
$a = 14.2159$ (16) Å	$\theta = 2.3$–$26.6°$
$b = 12.7207$ (15) Å	$\mu = 0.10$ mm^{-1}
$c = 9.2200$ (11) Å	$T = 293$ (2) K
$\beta = 101.668$ (2)°	Block, colourless
$V = 1632.9$ (3) Å^3	0.30 × 0.20 × 0.14 mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	3720 independent reflections
Radiation source: fine-focus sealed tube	2142 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.075$
$T = 293$(2) K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 2.2°$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -15 \rightarrow 18$
$T_{min} = 0.970$, $T_{max} = 0.986$	$k = -16 \rightarrow 16$
11970 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.064$	H-atom parameters constrained
$wR(F^2) = 0.180$	$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
3720 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
229 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2$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}
O1	0.46623 (14)	0.37820 (16)	0.9119 (2)	0.0650 (6)
O2	0.30929 (12)	0.56268 (14)	0.2772 (2)	0.0565 (5)
O3	0.10811 (12)	0.33410 (13)	0.15629 (19)	0.0507 (5)
O4	0.00398 (13)	0.47929 (15)	−0.32341 (19)	0.0602 (5)
O5	0.25889 (13)	0.64147 (14)	−0.00261 (19)	0.0552 (5)
O6	0.17240 (16)	0.73789 (15)	0.1245 (2)	0.0708 (6)
C1	0.40595 (19)	0.38621 (19)	0.7774 (3)	0.0489 (7)
C2	0.44923 (19)	0.3960 (2)	0.6551 (3)	0.0535 (7)
H2	0.5159	0.3969	0.6689	0.064*
C3	0.39525 (17)	0.4043 (2)	0.5159 (3)	0.0498 (7)
H3	0.4257	0.4115	0.4362	0.060*
C4	0.29485 (17)	0.40231 (18)	0.4904 (3)	0.0431 (6)
C5	0.25296 (19)	0.3930 (2)	0.6119 (3)	0.0500 (7)
H5	0.1863	0.3914	0.5976	0.060*
C6	0.30669 (19)	0.3859 (2)	0.7548 (3)	0.0529 (7)
H6	0.2763	0.3810	0.8349	0.063*
C7	0.23594 (17)	0.40769 (19)	0.3372 (3)	0.0432 (6)
C8	0.25006 (16)	0.49320 (18)	0.2385 (3)	0.0417 (6)
C9	0.18639 (16)	0.49020 (18)	0.0908 (3)	0.0403 (6)
C10	0.18742 (17)	0.56427 (18)	−0.0231 (3)	0.0441 (6)
C11	0.12689 (18)	0.5588 (2)	−0.1573 (3)	0.0483 (6)
H11	0.1296	0.6096	−0.2289	0.058*
C12	0.06065 (17)	0.4767 (2)	−0.1873 (3)	0.0462 (6)
C13	0.05577 (17)	0.40139 (19)	−0.0815 (3)	0.0448 (6)
H13	0.0121	0.3462	−0.1008	0.054*
C14	0.11834 (17)	0.41057 (18)	0.0554 (3)	0.0412 (6)
C15	0.16718 (18)	0.3364 (2)	0.2905 (3)	0.0496 (7)
H15	0.1595	0.2835	0.3568	0.060*
C16	0.4267 (2)	0.3860 (3)	1.0399 (3)	0.0706 (9)
H16A	0.3935	0.4518	1.0391	0.106*
H16B	0.4772	0.3823	1.1263	0.106*
H16C	0.3824	0.3292	1.0416	0.106*
C17	−0.0563 (2)	0.3907 (2)	−0.3728 (3)	0.0623 (8)
H17A	−0.0173	0.3291	−0.3727	0.094*
H17B	−0.0918	0.4036	−0.4714	0.094*
H17C	−0.1002	0.3802	−0.3075	0.094*
C18	0.2461 (2)	0.7245 (2)	0.0823 (3)	0.0543 (7)
C19	0.3320 (2)	0.7942 (2)	0.1069 (4)	0.0750 (10)
H19A	0.3287	0.8400	0.0233	0.113*
H19B	0.3891	0.7520	0.1186	0.113*
H19C	0.3337	0.8354	0.1946	0.113*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0571 (12)	0.0754 (14)	0.0582 (12)	0.0086 (10)	0.0014 (10)	0.0018 (10)
O2	0.0502 (11)	0.0498 (11)	0.0642 (12)	−0.0153 (9)	−0.0011 (9)	0.0073 (9)
O3	0.0487 (11)	0.0431 (10)	0.0573 (11)	−0.0097 (8)	0.0033 (9)	0.0003 (8)
O4	0.0613 (12)	0.0660 (12)	0.0482 (10)	−0.0073 (10)	−0.0010 (9)	−0.0054 (9)
O5	0.0584 (12)	0.0506 (11)	0.0569 (11)	−0.0114 (9)	0.0121 (9)	0.0011 (9)
O6	0.0667 (14)	0.0524 (12)	0.0938 (16)	0.0037 (10)	0.0178 (12)	−0.0104 (10)
C1	0.0451 (15)	0.0412 (14)	0.0562 (16)	0.0037 (12)	0.0007 (13)	0.0012 (12)
C2	0.0360 (14)	0.0598 (17)	0.0639 (18)	0.0006 (12)	0.0082 (13)	0.0043 (14)
C3	0.0389 (15)	0.0543 (16)	0.0579 (16)	0.0031 (12)	0.0137 (13)	0.0056 (13)
C4	0.0407 (14)	0.0363 (13)	0.0526 (15)	0.0006 (10)	0.0099 (12)	0.0036 (11)
C5	0.0354 (14)	0.0516 (16)	0.0620 (17)	0.0008 (12)	0.0073 (12)	0.0033 (13)
C6	0.0469 (16)	0.0551 (16)	0.0584 (17)	−0.0008 (13)	0.0148 (13)	0.0046 (13)
C7	0.0359 (13)	0.0419 (14)	0.0522 (15)	0.0026 (11)	0.0097 (11)	0.0023 (11)
C8	0.0343 (13)	0.0368 (13)	0.0548 (15)	0.0012 (11)	0.0110 (11)	−0.0019 (11)
C9	0.0340 (13)	0.0369 (13)	0.0502 (14)	0.0026 (10)	0.0089 (11)	−0.0042 (11)
C10	0.0422 (14)	0.0389 (13)	0.0530 (15)	−0.0046 (11)	0.0139 (12)	−0.0026 (11)
C11	0.0502 (15)	0.0496 (15)	0.0453 (14)	−0.0018 (12)	0.0097 (12)	0.0015 (12)
C12	0.0443 (14)	0.0480 (15)	0.0459 (15)	0.0064 (12)	0.0084 (12)	−0.0095 (12)
C13	0.0385 (14)	0.0396 (13)	0.0557 (15)	0.0005 (11)	0.0084 (12)	−0.0107 (12)
C14	0.0404 (14)	0.0334 (12)	0.0510 (14)	0.0029 (10)	0.0121 (11)	0.0004 (11)
C15	0.0462 (16)	0.0431 (14)	0.0573 (16)	−0.0011 (12)	0.0054 (13)	0.0084 (12)
C16	0.080 (2)	0.077 (2)	0.0516 (17)	0.0027 (17)	0.0051 (16)	0.0032 (15)
C17	0.0552 (18)	0.0654 (19)	0.0611 (17)	−0.0015 (14)	−0.0009 (14)	−0.0127 (14)
C18	0.0589 (19)	0.0414 (15)	0.0598 (17)	0.0042 (14)	0.0053 (15)	0.0091 (13)
C19	0.071 (2)	0.0523 (17)	0.093 (2)	−0.0193 (16)	−0.0043 (18)	0.0041 (16)

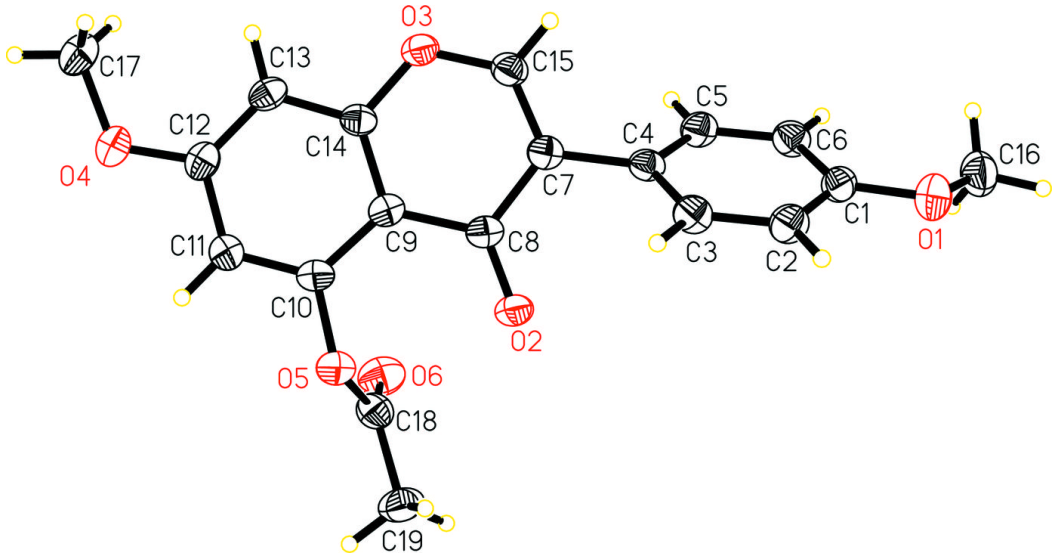
Geometric parameters (Å, °)

O1—C1	1.361 (3)	C7—C8	1.458 (3)
O1—C16	1.410 (3)	C8—C9	1.475 (3)
O2—C8	1.223 (3)	C9—C14	1.393 (3)
O3—C15	1.348 (3)	C9—C10	1.413 (3)
O3—C14	1.374 (3)	C10—C11	1.359 (3)
O4—C12	1.348 (3)	C11—C12	1.396 (3)
O4—C17	1.433 (3)	C11—H11	0.9300
O5—C18	1.349 (3)	C12—C13	1.380 (4)
O5—C10	1.398 (3)	C13—C14	1.394 (3)
O6—C18	1.201 (3)	C13—H13	0.9300
C1—C6	1.384 (4)	C15—H15	0.9300
C1—C2	1.395 (4)	C16—H16A	0.9600
C2—C3	1.359 (3)	C16—H16B	0.9600
C2—H2	0.9300	C16—H16C	0.9600
C3—C4	1.399 (3)	C17—H17A	0.9600
C3—H3	0.9300	C17—H17B	0.9600
C4—C5	1.376 (4)	C17—H17C	0.9600

C4—C7	1.491 (3)	C18—C19	1.489 (4)
C5—C6	1.386 (3)	C19—H19A	0.9600
C5—H5	0.9300	C19—H19B	0.9600
C6—H6	0.9300	C19—H19C	0.9600
C7—C15	1.339 (3)		
C1—O1—C16	118.3 (2)	C10—C11—H11	120.1
C15—O3—C14	118.33 (19)	C12—C11—H11	120.1
C12—O4—C17	118.9 (2)	O4—C12—C13	124.7 (2)
C18—O5—C10	116.7 (2)	O4—C12—C11	114.8 (2)
O1—C1—C6	124.9 (3)	C13—C12—C11	120.5 (2)
O1—C1—C2	116.4 (2)	C12—C13—C14	117.9 (2)
C6—C1—C2	118.8 (2)	C12—C13—H13	121.0
C3—C2—C1	120.9 (2)	C14—C13—H13	121.0
C3—C2—H2	119.6	O3—C14—C9	121.3 (2)
C1—C2—H2	119.6	O3—C14—C13	114.7 (2)
C2—C3—C4	121.3 (3)	C9—C14—C13	124.0 (2)
C2—C3—H3	119.4	C7—C15—O3	126.1 (2)
C4—C3—H3	119.4	C7—C15—H15	117.0
C5—C4—C3	117.4 (2)	O3—C15—H15	117.0
C5—C4—C7	121.5 (2)	O1—C16—H16A	109.5
C3—C4—C7	121.1 (2)	O1—C16—H16B	109.5
C4—C5—C6	122.2 (2)	H16A—C16—H16B	109.5
C4—C5—H5	118.9	O1—C16—H16C	109.5
C6—C5—H5	118.9	H16A—C16—H16C	109.5
C1—C6—C5	119.5 (3)	H16B—C16—H16C	109.5
C1—C6—H6	120.3	O4—C17—H17A	109.5
C5—C6—H6	120.3	O4—C17—H17B	109.5
C15—C7—C8	119.2 (2)	H17A—C17—H17B	109.5
C15—C7—C4	120.5 (2)	O4—C17—H17C	109.5
C8—C7—C4	120.3 (2)	H17A—C17—H17C	109.5
O2—C8—C7	122.0 (2)	H17B—C17—H17C	109.5
O2—C8—C9	123.1 (2)	O6—C18—O5	121.8 (3)
C7—C8—C9	114.9 (2)	O6—C18—C19	127.5 (3)
C14—C9—C10	115.0 (2)	O5—C18—C19	110.6 (3)
C14—C9—C8	120.2 (2)	C18—C19—H19A	109.5
C10—C9—C8	124.9 (2)	C18—C19—H19B	109.5
C11—C10—O5	117.9 (2)	H19A—C19—H19B	109.5
C11—C10—C9	122.9 (2)	C18—C19—H19C	109.5
O5—C10—C9	119.0 (2)	H19A—C19—H19C	109.5
C10—C11—C12	119.8 (2)	H19B—C19—H19C	109.5
C16—O1—C1—C6	−10.1 (4)	C14—C9—C10—C11	0.0 (3)
C16—O1—C1—C2	169.5 (2)	C8—C9—C10—C11	−179.5 (2)
O1—C1—C2—C3	179.9 (2)	C14—C9—C10—O5	−174.3 (2)
C6—C1—C2—C3	−0.5 (4)	C8—C9—C10—O5	6.2 (3)
C1—C2—C3—C4	−0.6 (4)	O5—C10—C11—C12	173.8 (2)
C2—C3—C4—C5	0.8 (4)	C9—C10—C11—C12	−0.6 (4)
C2—C3—C4—C7	−177.8 (2)	C17—O4—C12—C13	−9.8 (3)
C3—C4—C5—C6	0.1 (4)	C17—O4—C12—C11	170.9 (2)

C7—C4—C5—C6	178.8 (2)	C10—C11—C12—O4	179.7 (2)
O1—C1—C6—C5	−179.0 (2)	C10—C11—C12—C13	0.4 (4)
C2—C1—C6—C5	1.4 (4)	O4—C12—C13—C14	−178.9 (2)
C4—C5—C6—C1	−1.2 (4)	C11—C12—C13—C14	0.4 (3)
C5—C4—C7—C15	−50.3 (3)	C15—O3—C14—C9	−1.4 (3)
C3—C4—C7—C15	128.4 (3)	C15—O3—C14—C13	178.8 (2)
C5—C4—C7—C8	127.3 (3)	C10—C9—C14—O3	−178.9 (2)
C3—C4—C7—C8	−54.1 (3)	C8—C9—C14—O3	0.6 (3)
C15—C7—C8—O2	177.2 (2)	C10—C9—C14—C13	0.9 (3)
C4—C7—C8—O2	−0.4 (4)	C8—C9—C14—C13	−179.6 (2)
C15—C7—C8—C9	−1.6 (3)	C12—C13—C14—O3	178.7 (2)
C4—C7—C8—C9	−179.2 (2)	C12—C13—C14—C9	−1.1 (3)
O2—C8—C9—C14	−177.9 (2)	C8—C7—C15—O3	1.0 (4)
C7—C8—C9—C14	0.9 (3)	C4—C7—C15—O3	178.5 (2)
O2—C8—C9—C10	1.5 (4)	C14—O3—C15—C7	0.6 (4)
C7—C8—C9—C10	−179.7 (2)	C10—O5—C18—O6	−8.3 (4)
C18—O5—C10—C11	106.5 (3)	C10—O5—C18—C19	174.2 (2)
C18—O5—C10—C9	−79.0 (3)		

Fig. 1


O3
C17
C13
C15
C5
C12
C14
C4
C6
C16
O4
C7
C1
O1
C11
C9
C8
C3
C2
C10
O2
O5
O6
C18
C19