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3-Benzoyl-7-methoxy-2*H*-chromen-2-one

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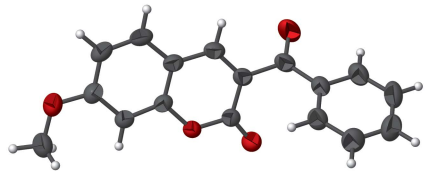
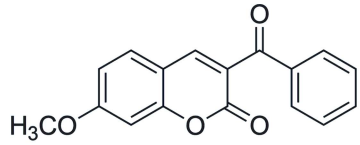
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{12}O_4$, the dihedral angle between the coumarin ring system (r.m.s. deviation = 0.018 Å) and the phenyl ring is 55.96 (8)°. In the crystal, weak C—H···O interactions connect the molecules into a three-dimensional network and aromatic π–π stacking interactions are also observed [shortest centroid–centroid separation = 3.6692 (9) Å].

3D view



Chemical scheme



Structure description

3-Carbonylcoumarin represents an important structural element in anticoagulant agents (Sandhu *et al.*, 2014), and acts as a monoamine oxidase (MAO)-B inhibitor (Mertens *et al.*, 2014). As part of our work on the synthesis of 3-aroylcoumarins, we report the crystal structure of the title compound (Fig. 1). This study provides an opportunity to investigate the geometry of 3-aroylcoumarin derivatives with no strong intermolecular interactions.

The C7—O1 and C8—O2 bond lengths are 1.218 (2) and 1.205 (2) Å, respectively. They are shorter than the standard C=O bond length (1.231 Å; Gao *et al.*, 2014) due to conjugation with the aromatic ring. The coumarin ring is almost planar (r.m.s. deviation = 0.018 Å) and subtends a dihedral angle of 55.96 (8)° with the phenyl ring. The main twist occurs about the C7—C9 bond [C6—C7—C9—C8 = −51.8 (2)°]. The methyl group (atom C17) is approximately coplanar with its attached ring [deviation = 0.111 (2) Å].

In the crystal, weak C—H···O interactions (Table 1) connect the molecules into a three-dimensional network and aromatic π–π stacking interactions are also observed [shortest centroid–centroid separation = 3.6692 (9) Å].

Synthesis and crystallization

The synthesis of 3-benzoyl-7-methoxy-2*H*-chromen-2-one is based on our reported literature procedure (Yuan *et al.*, 2015). In a 25 ml Schlenk tube, 7-methoxyl coumarin (0.25 mmol, 44 mg), benzaldehyde (1.0 mmol, 106 mg), TBHP (1.0 mmol) and $FeCl_2$

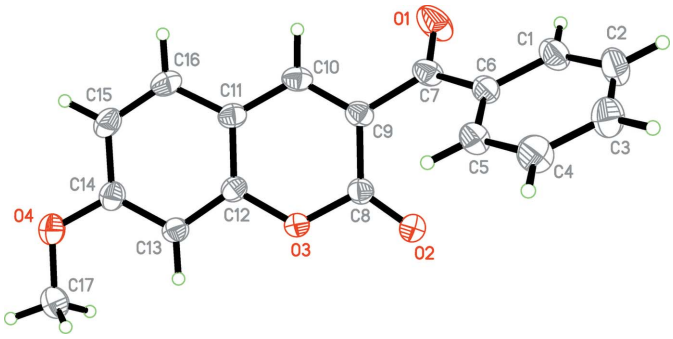


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

(0.025 mmol, 31.5 mg) were added and charged with nitrogen (3 cycles). Chlorobenzene (2 ml) was then added, and the reaction mixture was heated on an oil bath at 120°C for 12 h (monitored by TLC). After the reaction mixture had cooled to room temperature and the solvent had been removed with the aid of a rotary evaporator, 2 ml ethylacetate was added to the residue. The solution was filtrated, and the filtrate was distilled under vacuum. The crude product was purified by silica gel column chromatography using ethyl acetate/petroleum ether (1:5) as eluant to obtain the desired product as colourless prismatic crystals, m.p. 134–135°C.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.06 (*s*, 1H), 7.87 (*d*, J_{H-H} = 7.2 Hz, 2H), 7.65 (*td*, J_{H-H} = 8.7 Hz, J_{H-H} = 1.5 Hz, 1H), 7.60 (*dd*, J_{H-H} = 7.9 Hz, J_{H-H} = 1.5 Hz, 1H), 7.48 (*d*, J_{H-H} = 7.9 Hz, 2H), 7.40 (*t*, J_{H-H} = 8.3 Hz, 1H), 7.35 (*td*, J_{H-H} = 7.8 Hz, J_{H-H} = 0.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 191.7 (C═O), 158.4 (C═O), 154.7, 145.4 (CH), 136.2, 133.8 (CH), 133.7 (CH), 129.6 (CH), 129.2 (CH), 128.6 (CH), 125.0 (CH), 118.2, 116.9 (CH). IR (KBr) ν (cm^{-1}): 1714, 1654 (C═O), 1608, 1565, 1448 (Ar–). MS (ESI) *m*/*z*: 251.3 $[M + H]^+$ (calculated for $C_{16}H_{11}O_3^+$ 251.1).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O4^{i}	0.93	2.58	3.503 (2)	171
C5—H5···O2^{ii}	0.93	2.49	3.330 (2)	151
C13—H13···O1^{iii}	0.93	2.37	3.281 (2)	165

Symmetry codes: (i) $x-1, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x+1, y, z$; (iii) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{12}O_4$
M_r	280.27
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	291
a, *b*, *c* (Å)	4.07206 (11), 11.9589 (3), 27.5626 (7)
β (°)	90.306 (2)
V (Å^3)	1342.21 (6)
Z	4
Radiation type	Cu *K*α
μ (mm^{-1})	0.82
Crystal size (mm)	0.3 × 0.2 × 0.2
Data collection	
Diffractometer	Agilent Xcalibur Eos Gemini
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min}, T_{max}	0.946, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4863, 2384, 1994
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.597
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.044, 0.122, 1.07
No. of reflections	2384
No. of parameters	191
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.14, −0.22

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2017). **2**, x170373 [https://doi.org/10.1107/S241431461700373X]

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Crystal data

$C_{17}H_{12}O_4$
M_r = 280.27
Monoclinic, $P2_1/c$
a = 4.07206 (11) Å
b = 11.9589 (3) Å
c = 27.5626 (7) Å
β = 90.306 (2)°
V = 1342.21 (6) Å^3
Z = 4
$F(000)$ = 584
D_x = 1.387 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2065 reflections
θ = 3.7–71.9°
μ = 0.82 mm^{-1}
T = 291 K
Prism, colourless
0.3 × 0.2 × 0.2 mm

Data collection

Agilent Xcalibur Eos Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.2312 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2013)
T_{min} = 0.946, T_{max} = 1.000
4863 measured reflections
2384 independent reflections
1994 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 67.1°, θ_{min} = 3.2°
h = −3→4
k = −11→14
l = −32→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.122
S = 1.07
2384 reflections
191 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.1354P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0239 (5)	0.58205 (12)	0.35344 (5)	0.0833 (5)
O2	−0.1454 (3)	0.88869 (10)	0.30432 (4)	0.0551 (3)
O3	0.1358 (3)	0.86182 (9)	0.23776 (4)	0.0443 (3)
O4	0.6558 (4)	0.80906 (12)	0.08545 (4)	0.0626 (4)
C1	0.0047 (5)	0.71111 (17)	0.43741 (7)	0.0596 (5)
H1	−0.1134	0.6447	0.4389	0.071*
C2	0.0569 (6)	0.7723 (2)	0.47901 (7)	0.0698 (6)
H2	−0.0263	0.7473	0.5084	0.084*
C3	0.2323 (6)	0.8706 (2)	0.47706 (7)	0.0723 (6)
H3	0.2659	0.9123	0.5051	0.087*
C4	0.3582 (6)	0.90725 (18)	0.43354 (7)	0.0664 (5)
H4	0.4782	0.9734	0.4324	0.080*
C5	0.3070 (4)	0.84630 (15)	0.39172 (6)	0.0516 (4)
H5	0.3932	0.8713	0.3625	0.062*
C6	0.1279 (4)	0.74808 (14)	0.39307 (6)	0.0454 (4)
C7	0.0744 (5)	0.67775 (14)	0.34951 (6)	0.0506 (4)
C8	0.0393 (4)	0.82732 (13)	0.28306 (5)	0.0422 (3)
C9	0.1607 (4)	0.71949 (12)	0.29987 (5)	0.0434 (4)
C10	0.3356 (4)	0.65378 (13)	0.26960 (6)	0.0465 (4)
H10	0.3984	0.5828	0.2799	0.056*
C11	0.4259 (4)	0.69057 (12)	0.22246 (6)	0.0432 (4)
C12	0.3228 (4)	0.79611 (12)	0.20769 (5)	0.0401 (3)
C13	0.3930 (4)	0.84099 (13)	0.16282 (5)	0.0437 (4)
H13	0.3213	0.9122	0.1542	0.052*
C14	0.5743 (4)	0.77607 (14)	0.13099 (6)	0.0478 (4)
C15	0.6868 (5)	0.66990 (15)	0.14458 (7)	0.0543 (4)
H15	0.8112	0.6278	0.1231	0.065*
C16	0.6141 (4)	0.62805 (13)	0.18936 (6)	0.0516 (4)
H16	0.6898	0.5575	0.1981	0.062*
C17	0.5324 (6)	0.91387 (18)	0.06893 (7)	0.0675 (5)
H17A	0.5942	0.9250	0.0357	0.101*
H17B	0.6225	0.9728	0.0885	0.101*
H17C	0.2974	0.9144	0.0714	0.101*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1370 (15)	0.0514 (8)	0.0617 (8)	−0.0372 (9)	0.0034 (9)	0.0076 (6)
O2	0.0616 (7)	0.0509 (7)	0.0531 (6)	0.0095 (6)	0.0150 (5)	0.0038 (5)
O3	0.0553 (7)	0.0364 (5)	0.0413 (5)	0.0069 (5)	0.0065 (4)	0.0026 (4)
O4	0.0776 (9)	0.0641 (8)	0.0462 (6)	0.0104 (7)	0.0148 (6)	−0.0017 (6)
C1	0.0651 (11)	0.0604 (10)	0.0534 (10)	−0.0051 (9)	0.0078 (8)	0.0128 (8)
C2	0.0811 (14)	0.0863 (15)	0.0422 (9)	0.0055 (11)	0.0101 (8)	0.0071 (9)
C3	0.0889 (15)	0.0808 (14)	0.0472 (10)	0.0024 (12)	−0.0054 (9)	−0.0113 (10)
C4	0.0786 (13)	0.0625 (11)	0.0581 (11)	−0.0120 (10)	−0.0084 (9)	−0.0057 (9)

C5	0.0548 (10)	0.0535 (9)	0.0466 (8)	−0.0071 (8)	0.0004 (7)	0.0034 (7)
C6	0.0480 (8)	0.0460 (8)	0.0421 (8)	−0.0007 (7)	0.0018 (6)	0.0060 (6)
C7	0.0598 (10)	0.0399 (8)	0.0521 (9)	−0.0081 (7)	−0.0010 (7)	0.0071 (7)
C8	0.0461 (8)	0.0377 (7)	0.0428 (8)	−0.0039 (6)	0.0022 (6)	−0.0005 (6)
C9	0.0512 (8)	0.0352 (7)	0.0438 (8)	−0.0069 (6)	−0.0033 (6)	0.0008 (6)
C10	0.0552 (9)	0.0334 (7)	0.0508 (8)	−0.0013 (6)	−0.0080 (7)	0.0010 (6)
C11	0.0477 (8)	0.0346 (7)	0.0473 (8)	−0.0004 (6)	−0.0040 (6)	−0.0035 (6)
C12	0.0417 (8)	0.0365 (7)	0.0419 (7)	0.0000 (6)	−0.0011 (6)	−0.0055 (6)
C13	0.0491 (9)	0.0380 (7)	0.0440 (8)	0.0033 (6)	0.0002 (6)	−0.0011 (6)
C14	0.0498 (9)	0.0493 (9)	0.0443 (8)	−0.0002 (7)	0.0023 (6)	−0.0073 (7)
C15	0.0603 (10)	0.0475 (9)	0.0551 (9)	0.0064 (8)	0.0062 (7)	−0.0143 (7)
C16	0.0587 (10)	0.0360 (8)	0.0603 (10)	0.0077 (7)	−0.0024 (7)	−0.0067 (7)
C17	0.0833 (14)	0.0706 (13)	0.0488 (9)	0.0062 (11)	0.0108 (9)	0.0070 (9)

Geometric parameters (Å, º)

O1—C7	1.218 (2)	C7—C9	1.500 (2)
O2—C8	1.205 (2)	C8—C9	1.456 (2)
O3—C8	1.3744 (18)	C9—C10	1.352 (2)
O3—C12	1.3751 (18)	C10—H10	0.9300
O4—C14	1.359 (2)	C10—C11	1.422 (2)
O4—C17	1.424 (2)	C11—C12	1.390 (2)
C1—H1	0.9300	C11—C16	1.410 (2)
C1—C2	1.376 (3)	C12—C13	1.380 (2)
C1—C6	1.395 (2)	C13—H13	0.9300
C2—H2	0.9300	C13—C14	1.387 (2)
C2—C3	1.376 (3)	C14—C15	1.400 (2)
C3—H3	0.9300	C15—H15	0.9300
C3—C4	1.378 (3)	C15—C16	1.366 (3)
C4—H4	0.9300	C16—H16	0.9300
C4—C5	1.379 (3)	C17—H17A	0.9600
C5—H5	0.9300	C17—H17B	0.9600
C5—C6	1.383 (2)	C17—H17C	0.9600
C6—C7	1.481 (2)		
C8—O3—C12	122.55 (12)	C10—C9—C8	119.85 (14)
C14—O4—C17	117.61 (14)	C9—C10—H10	119.2
C2—C1—H1	119.8	C9—C10—C11	121.57 (14)
C2—C1—C6	120.45 (19)	C11—C10—H10	119.2
C6—C1—H1	119.8	C12—C11—C10	118.00 (14)
C1—C2—H2	120.0	C12—C11—C16	117.15 (14)
C3—C2—C1	119.96 (18)	C16—C11—C10	124.85 (14)
C3—C2—H2	120.0	O3—C12—C11	120.63 (14)
C2—C3—H3	120.0	O3—C12—C13	115.85 (13)
C2—C3—C4	120.08 (19)	C13—C12—C11	123.51 (14)
C4—C3—H3	120.0	C12—C13—H13	121.2
C3—C4—H4	119.9	C12—C13—C14	117.56 (14)
C3—C4—C5	120.3 (2)	C14—C13—H13	121.2

C5—C4—H4	119.9	O4—C14—C13	123.72 (16)
C4—C5—H5	119.9	O4—C14—C15	115.43 (15)
C4—C5—C6	120.25 (17)	C13—C14—C15	120.85 (15)
C6—C5—H5	119.9	C14—C15—H15	119.9
C1—C6—C7	118.57 (16)	C16—C15—C14	120.14 (15)
C5—C6—C1	118.99 (16)	C16—C15—H15	119.9
C5—C6—C7	122.38 (15)	C11—C16—H16	119.6
O1—C7—C6	120.57 (16)	C15—C16—C11	120.78 (15)
O1—C7—C9	118.22 (16)	C15—C16—H16	119.6
C6—C7—C9	121.09 (14)	O4—C17—H17A	109.5
O2—C8—O3	116.18 (14)	O4—C17—H17B	109.5
O2—C8—C9	126.62 (14)	O4—C17—H17C	109.5
O3—C8—C9	117.17 (13)	H17A—C17—H17B	109.5
C8—C9—C7	120.28 (14)	H17A—C17—H17C	109.5
C10—C9—C7	119.75 (14)	H17B—C17—H17C	109.5

O1—C7—C9—C8	132.2 (2)	C7—C9—C10—C11	−179.27 (15)
O1—C7—C9—C10	−43.9 (3)	C8—O3—C12—C11	−0.6 (2)
O2—C8—C9—C7	−4.0 (3)	C8—O3—C12—C13	178.34 (14)
O2—C8—C9—C10	172.08 (17)	C8—C9—C10—C11	4.6 (2)
O3—C8—C9—C7	178.13 (13)	C9—C10—C11—C12	−1.3 (2)
O3—C8—C9—C10	−5.8 (2)	C9—C10—C11—C16	178.42 (16)
O3—C12—C13—C14	−178.47 (13)	C10—C11—C12—O3	−0.8 (2)
O4—C14—C15—C16	−179.02 (16)	C10—C11—C12—C13	−179.67 (14)
C1—C2—C3—C4	−0.6 (4)	C10—C11—C16—C15	179.45 (16)
C1—C6—C7—O1	−12.3 (3)	C11—C12—C13—C14	0.5 (2)
C1—C6—C7—C9	171.84 (16)	C12—O3—C8—O2	−174.28 (14)
C2—C1—C6—C5	0.9 (3)	C12—O3—C8—C9	3.8 (2)
C2—C1—C6—C7	178.16 (19)	C12—C11—C16—C15	−0.8 (2)
C2—C3—C4—C5	0.5 (4)	C12—C13—C14—O4	178.81 (15)
C3—C4—C5—C6	0.2 (3)	C12—C13—C14—C15	−1.3 (2)
C4—C5—C6—C1	−0.9 (3)	C13—C14—C15—C16	1.1 (3)
C4—C5—C6—C7	−178.07 (18)	C14—C15—C16—C11	0.0 (3)
C5—C6—C7—O1	164.90 (19)	C16—C11—C12—O3	179.44 (14)
C5—C6—C7—C9	−11.0 (3)	C16—C11—C12—C13	0.6 (2)
C6—C1—C2—C3	−0.2 (3)	C17—O4—C14—C13	−3.4 (3)
C6—C7—C9—C8	−51.8 (2)	C17—O4—C14—C15	176.72 (16)
C6—C7—C9—C10	132.12 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O4^{i}	0.93	2.58	3.503 (2)	171
C5—H5···O2^{ii}	0.93	2.49	3.330 (2)	151
C13—H13···O1^{iii}	0.93	2.37	3.281 (2)	165

Symmetry codes: (i) $x-1, -y+3/2, z+1/2$; (ii) $x+1, y, z$; (iii) $-x, y+1/2, -z+1/2$.