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2-Oxo-2*H*-chromen-3-yl benzoate

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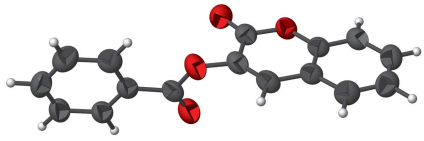
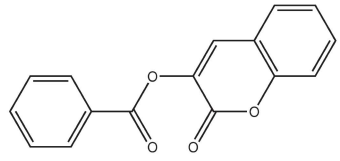
In the title compound, $C_{16}H_{10}O_4$, the dihedral angle between the coumarin ring system (r.m.s. deviation = 0.015 Å) and the benzoate group is 83.58 (9)°, which compares to a value of 81.8° obtained from a DFT calculation at the B3LYP/6–311 G(d,p) level. In the crystal, C—O···π and C—H···π interactions and aromatic π–π [Cg···Cg = 3.7214 (14) and 3.7059 (14) Å] stacking generate a three-dimensional network.

3D view



Chemical scheme



Structure description

Coumarin-based ion receptors, fluorescent probes, and biological stains have extensive applications in monitoring enzyme activity as well as accurate pharmacological and pharmacokinetic properties in living cells (Chen *et al.*, 2013; Guha *et al.*, 2012). As part of our ongoing studies in this area, we now present herein the synthesis and structure of the title compound (Fig. 1).

As expected, the coumarin ring system is almost planar, the maximum deviation from the plane of 0.022 (2) Å is for atom C9. The torsion angles C10—C8—O4—C7 [107.8 (2)°], C8—O4—C7—C6 [−170.95 (15)°] and O4—C7—C6—C1 [176.48 (15)°] are typical of the torsional freedom permitted by the rotation of the benzoate group at position 3. The greatest conformational freedom of the molecule resides, therefore, in the benzoate bridge of compound, composed by C8—O4—C7—C6.

In the crystal, there are C—H···π and C—O···π contacts present (Table 1 and Fig. 2) and also π-π- stacking interactions. The H···π and O···π separations are comparable with those cited by Imai *et al.* (2008) from a database analysis, which concluded that such interactions were attractive, with interaction energies of *ca* 2 kcal mol^{-1}, comparable to those typical of weak hydrogen bonds. These interactions result in the formation of zigzag

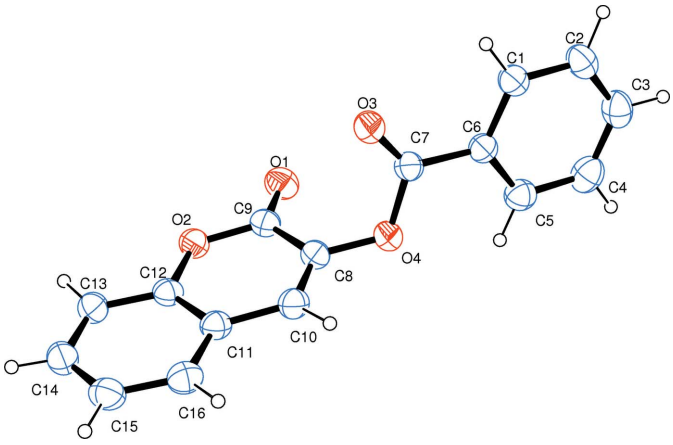


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

chains propagating along the *c*-axis direction. The supramolecular aggregation in the crystal is completed by the presence of slipped parallel π–π interactions, forming columns along the *c*-axis direction. The most significant interactions are $Cg2\cdots Cg2^{i}$ = 3.7216 (14) Å [inter-planar distance = 3.4024 (9) Å, slippage = 1.508 Å, where *Cg*2 is the centroid of the C1/C2/C4–C6 ring; symmetry code: (i) $-x, 1-y, 1-z$] and $Cg2\cdots Cg2^{ii}$ = 3.7058 (14) Å [inter-planar distance = 3.4309 (9) Å, slippage = 1.401 Å, symmetry code: (ii) $1-x, 1-y, 1-z$].

Synthesis and crystallization

In a 100 ml flask with a water condenser were introduced successively 25 ml of dried diethyl ether, 6.17 mmol of benzoyl chloride and 3.2 ml of dried triethylamine. While stirring strongly, 6.17 mmol of chroman-2,3-dione were added in small portions. The reaction mixture was left under agitation for 2 h at room temperature and then refluxed for 2 h. The mixture was poured in a separating funnel containing 40 ml of chloroform and washed with diluted hydrochloric acid solution until the pH was 2-3. The organic layer was extracted, washed with water to neutrality, dried over $MgSO_4$ and the solvent removed. The resulting precipitate (crude product) was filtered off with suction, washed with petroleum ether and recrystallized from a solvent mixture of chloroform–hexane (1:3, *v*/*v*). Yellow crystals of the title compound were obtained in a yield of 84%; m.p. 423–426 K.

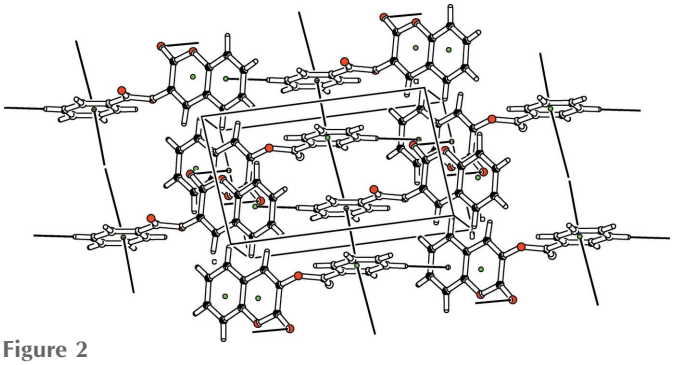


Figure 2
Packing diagram of the title compound, viewed along the *b* axis.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···*Cg*3^{i}	1.00 (2)	2.94 (2)	3.841 (2)	150.5 (2)
C9—O1···*Cg*1^{ii}	1.20 (1)	3.15 (1)	3.464 (2)	95 (1)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{10}O_4$
M_r	266.24
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	298
a, *b*, *c* (Å)	6.9243 (6), 7.7262 (8), 11.8168 (6)
α, β, γ (°)	84.550 (6), 81.852 (6), 83.023 (8)
V (Å^3)	619.22 (9)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.10
Crystal size (mm)	0.34 × 0.12 × 0.06
Data collection	
Diffractometer	Agilent Supernova Dual diffractometer with an Atlas detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min}, T_{max}	0.499, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7628, 2283, 1724
R_{int}	0.039
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.608
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.051, 0.159, 1.04
No. of reflections	2283
No. of parameters	221
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.28

Computer programs: *CrysAlis PRO* (Agilent, 2011), *SHELXS2013* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Two reflections were omitted owing to bad agreement.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{16}H_{10}O_4$
$M_r = 266.24$
Triclinic, $P\overline{1}$
$a = 6.9243$ (6) Å
$b = 7.7262$ (8) Å
$c = 11.8168$ (6) Å
$\alpha = 84.550$ (6)°
$\beta = 81.852$ (6)°
$\gamma = 83.023$ (8)°
$V = 619.22$ (9) Å^3
$Z = 2$
$F(000) = 276$
$D_x = 1.428$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2675 reflections
$\theta = 5.8$–$69.2°$
$\mu = 0.10$ mm^{-1}
$T = 298$ K
Prism, yellow
0.34 × 0.12 × 0.06 mm

Data collection

Agilent Supernova Dual diffractometer with an Atlas detector
Radiation source: sealed X-ray tube
Detector resolution: 5.3048 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (Crysalis PRO; Agilent, 2011)
$T_{min} = 0.499$, $T_{max} = 1.000$
7628 measured reflections
2283 independent reflections
1724 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 25.6°$, $\theta_{min} = 1.8°$
$h = -8\rightarrow8$
$k = -9\rightarrow9$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.159$
$S = 1.04$
2283 reflections
221 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0998P)^2 + 0.0263P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ 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.

Refinement. All H atoms were refined using a riding model, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic, and C—H = 0.99 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for the methylene H atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
H10	0.994 (4)	0.658 (3)	0.393 (2)	0.068 (6)*
H4	0.825 (4)	0.121 (4)	0.016 (3)	0.093 (8)*
H3	0.789 (3)	0.243 (3)	−0.168 (2)	0.076 (7)*
H1	0.711 (3)	0.728 (3)	−0.0468 (19)	0.063 (6)*
H5	0.790 (3)	0.285 (3)	0.171 (2)	0.068 (6)*
H2	0.731 (3)	0.548 (3)	−0.202 (2)	0.071 (7)*
H13	0.401 (4)	0.916 (3)	0.688 (2)	0.073 (6)*
H16	1.079 (4)	0.800 (3)	0.565 (2)	0.073 (7)*
H15	0.977 (4)	0.946 (3)	0.732 (2)	0.083 (7)*
H14	0.634 (3)	1.000 (3)	0.791 (2)	0.070 (6)*
O2	0.46650 (18)	0.75322 (17)	0.51161 (11)	0.0538 (4)
O4	0.7627 (2)	0.54314 (18)	0.27349 (11)	0.0618 (4)
O1	0.3793 (2)	0.6280 (2)	0.36899 (13)	0.0666 (4)
C8	0.7208 (3)	0.6366 (2)	0.37014 (15)	0.0543 (5)
C11	0.8071 (3)	0.7664 (2)	0.52978 (16)	0.0518 (5)
O3	0.6978 (2)	0.79125 (18)	0.16416 (12)	0.0661 (4)
C6	0.7503 (2)	0.5198 (2)	0.07680 (15)	0.0496 (4)
C9	0.5118 (3)	0.6699 (2)	0.41218 (16)	0.0532 (5)
C12	0.6084 (3)	0.8027 (2)	0.56933 (15)	0.0495 (4)
C13	0.5431 (3)	0.8890 (2)	0.66733 (16)	0.0570 (5)
C10	0.8604 (3)	0.6801 (3)	0.42525 (17)	0.0561 (5)
C7	0.7320 (3)	0.6361 (2)	0.17134 (15)	0.0522 (4)
C1	0.7338 (3)	0.5970 (3)	−0.03279 (17)	0.0553 (5)
C5	0.7800 (3)	0.3397 (3)	0.0954 (2)	0.0618 (5)
C3	0.7787 (3)	0.3156 (3)	−0.1056 (2)	0.0686 (6)
C16	0.9429 (3)	0.8200 (3)	0.5925 (2)	0.0639 (5)
C2	0.7489 (3)	0.4928 (3)	−0.12395 (18)	0.0636 (5)
C15	0.8781 (4)	0.9067 (3)	0.6904 (2)	0.0676 (6)
C4	0.7933 (3)	0.2371 (3)	0.0027 (2)	0.0718 (6)
C14	0.6802 (4)	0.9404 (3)	0.72688 (18)	0.0650 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0468 (7)	0.0646 (8)	0.0492 (7)	−0.0038 (5)	−0.0054 (5)	−0.0045 (6)
O4	0.0752 (9)	0.0610 (8)	0.0452 (7)	0.0067 (6)	−0.0063 (6)	−0.0045 (6)
O1	0.0644 (9)	0.0749 (9)	0.0638 (9)	−0.0119 (7)	−0.0162 (7)	−0.0064 (7)
C8	0.0605 (11)	0.0560 (10)	0.0428 (9)	0.0012 (8)	−0.0036 (8)	0.0004 (7)
C11	0.0512 (10)	0.0528 (9)	0.0500 (10)	−0.0037 (7)	−0.0073 (8)	0.0024 (7)
O3	0.0836 (10)	0.0595 (8)	0.0517 (8)	0.0027 (7)	−0.0062 (7)	−0.0043 (6)
C6	0.0390 (8)	0.0595 (10)	0.0498 (10)	−0.0045 (7)	−0.0047 (7)	−0.0052 (8)
C9	0.0579 (11)	0.0524 (9)	0.0478 (10)	−0.0040 (8)	−0.0078 (8)	0.0025 (8)
C12	0.0527 (10)	0.0478 (9)	0.0467 (9)	−0.0050 (7)	−0.0075 (8)	0.0040 (7)
C13	0.0605 (12)	0.0576 (10)	0.0505 (10)	−0.0026 (8)	−0.0033 (9)	−0.0026 (8)
C10	0.0484 (10)	0.0640 (11)	0.0513 (10)	0.0006 (8)	0.0000 (8)	0.0003 (8)

C7	0.0478 (9)	0.0599 (11)	0.0459 (9)	−0.0007 (7)	−0.0022 (7)	−0.0015 (8)
C1	0.0512 (10)	0.0630 (11)	0.0520 (10)	−0.0079 (8)	−0.0064 (8)	−0.0046 (8)
C5	0.0575 (11)	0.0640 (11)	0.0630 (12)	−0.0052 (8)	−0.0077 (9)	−0.0019 (9)
C3	0.0591 (12)	0.0822 (14)	0.0702 (14)	−0.0132 (10)	−0.0087 (10)	−0.0273 (12)
C16	0.0545 (11)	0.0672 (12)	0.0698 (13)	−0.0037 (9)	−0.0120 (10)	−0.0018 (10)
C2	0.0588 (11)	0.0835 (14)	0.0515 (11)	−0.0140 (10)	−0.0076 (9)	−0.0113 (10)
C15	0.0754 (14)	0.0645 (12)	0.0673 (13)	−0.0085 (10)	−0.0254 (11)	−0.0033 (10)
C4	0.0674 (13)	0.0594 (12)	0.0905 (17)	−0.0071 (9)	−0.0110 (11)	−0.0156 (11)
C14	0.0832 (15)	0.0563 (11)	0.0547 (11)	−0.0026 (9)	−0.0102 (10)	−0.0061 (9)

Geometric parameters (Å, º)

O2—C9	1.372 (2)	C13—H13	0.98 (3)
O2—C12	1.381 (2)	C10—H10	0.95 (2)
O4—C7	1.374 (2)	C1—C2	1.390 (3)
O4—C8	1.386 (2)	C1—H1	1.01 (2)
O1—C9	1.202 (2)	C5—C4	1.398 (3)
C8—C10	1.331 (3)	C5—H5	0.96 (2)
C8—C9	1.462 (3)	C3—C2	1.361 (3)
C11—C12	1.392 (3)	C3—C4	1.375 (4)
C11—C16	1.398 (3)	C3—H3	0.96 (2)
C11—C10	1.440 (3)	C16—C15	1.384 (3)
O3—C7	1.191 (2)	C16—H16	0.95 (3)
C6—C5	1.383 (3)	C2—H2	0.99 (2)
C6—C1	1.387 (3)	C15—C14	1.378 (3)
C6—C7	1.481 (3)	C15—H15	0.99 (3)
C12—C13	1.384 (3)	C4—H4	0.90 (3)
C13—C14	1.374 (3)	C14—H14	0.92 (2)
C9—O2—C12	122.54 (14)	O4—C7—C6	111.59 (15)
C7—O4—C8	115.88 (14)	C6—C1—C2	119.8 (2)
C10—C8—O4	122.46 (17)	C6—C1—H1	120.5 (12)
C10—C8—C9	122.72 (17)	C2—C1—H1	119.7 (13)
O4—C8—C9	114.61 (17)	C6—C5—C4	119.3 (2)
C12—C11—C16	117.93 (18)	C6—C5—H5	120.7 (13)
C12—C11—C10	118.13 (17)	C4—C5—H5	120.0 (13)
C16—C11—C10	123.92 (18)	C2—C3—C4	120.8 (2)
C5—C6—C1	120.07 (18)	C2—C3—H3	120.4 (14)
C5—C6—C7	122.07 (18)	C4—C3—H3	118.8 (14)
C1—C6—C7	117.85 (17)	C15—C16—C11	120.0 (2)
O1—C9—O2	118.14 (17)	C15—C16—H16	121.2 (14)
O1—C9—C8	125.95 (18)	C11—C16—H16	118.8 (14)
O2—C9—C8	115.89 (17)	C3—C2—C1	120.1 (2)
O2—C12—C13	116.78 (16)	C3—C2—H2	120.4 (13)
O2—C12—C11	120.95 (16)	C1—C2—H2	119.5 (13)
C13—C12—C11	122.27 (18)	C14—C15—C16	120.4 (2)
C14—C13—C12	118.38 (19)	C14—C15—H15	121.3 (15)
C14—C13—H13	123.6 (14)	C16—C15—H15	118.3 (15)

C12—C13—H13	117.9 (14)	C3—C4—C5	119.9 (2)
C8—C10—C11	119.75 (17)	C3—C4—H4	122.2 (19)
C8—C10—H10	119.1 (14)	C5—C4—H4	117.5 (19)
C11—C10—H10	121.1 (14)	C13—C14—C15	120.99 (19)
O3—C7—O4	121.79 (17)	C13—C14—H14	117.2 (15)
O3—C7—C6	126.62 (18)	C15—C14—H14	121.8 (15)
C7—O4—C8—C10	−107.8 (2)	C8—O4—C7—O3	9.9 (3)
C7—O4—C8—C9	77.3 (2)	C8—O4—C7—C6	−170.95 (15)
C12—O2—C9—O1	178.33 (15)	C5—C6—C7—O3	−176.40 (19)
C12—O2—C9—C8	−0.2 (3)	C1—C6—C7—O3	2.6 (3)
C10—C8—C9—O1	−176.93 (19)	C5—C6—C7—O4	4.5 (2)
O4—C8—C9—O1	−2.0 (3)	C1—C6—C7—O4	−176.48 (15)
C10—C8—C9—O2	1.4 (3)	C5—C6—C1—C2	−0.6 (3)
O4—C8—C9—O2	176.31 (15)	C7—C6—C1—C2	−179.64 (16)
C9—O2—C12—C13	178.71 (15)	C1—C6—C5—C4	0.1 (3)
C9—O2—C12—C11	−1.4 (3)	C7—C6—C5—C4	179.10 (18)
C16—C11—C12—O2	−179.87 (16)	C12—C11—C16—C15	−0.2 (3)
C10—C11—C12—O2	1.7 (3)	C10—C11—C16—C15	178.12 (19)
C16—C11—C12—C13	0.0 (3)	C4—C3—C2—C1	0.3 (3)
C10—C11—C12—C13	−178.40 (17)	C6—C1—C2—C3	0.4 (3)
O2—C12—C13—C14	−179.91 (16)	C11—C16—C15—C14	0.2 (3)
C11—C12—C13—C14	0.2 (3)	C2—C3—C4—C5	−0.8 (3)
O4—C8—C10—C11	−175.59 (16)	C6—C5—C4—C3	0.6 (3)
C9—C8—C10—C11	−1.1 (3)	C12—C13—C14—C15	−0.2 (3)
C12—C11—C10—C8	−0.5 (3)	C16—C15—C14—C13	0.0 (3)
C16—C11—C10—C8	−178.80 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···*Cg*3^{i}	1.00 (2)	2.94 (2)	3.841 (2)	150.5 (2)
C9—O1···*Cg*1^{ii}	1.20 (1)	3.15 (1)	3.464 (2)	95 (1)

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*+1, −*y*+1, −*z*.