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ISSN 2414-3146

2-Oxo-2*H*-chromen-3-yl 4-fluorobenzoate

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Received 9 February 2017
Accepted 11 April 2017

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

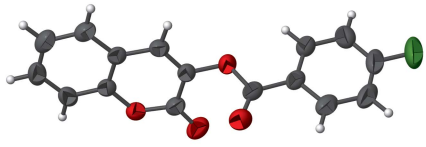
Keywords: crystal structure; chroman; hydrogen bond; π–π interactions.

CCDC reference: 1543492

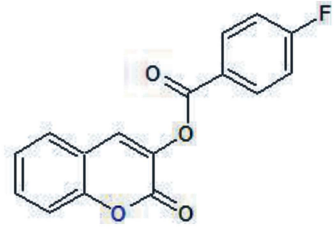
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_9FO_4$, the dihedral angle between the planar coumarin ring system [maximum deviation = 0.027 (1) Å] and the benzene ring is 70.18 (6)°. In the crystal, π–π interactions [shortest centroid–centroid separation = 3.5338 (8) Å] link the molecules into a three-dimensional framework.

3D view



Chemical scheme



Structure description

Coumarin and its derivatives are widely recognized for their multiple biological activities, including anticancer (Lacy & O'Kennedy, 2004) and anti-inflammatory (Todeschini *et al.*, 1998) effects. As part of our studies in this area, we now describe the synthesis and crystal structure of the title compound (Fig. 1).

The coumarin ring system is, as expected, almost planar [maximum deviation = 0.027 (1) Å for atom C1] and is oriented at an angle of 70.18 (6)° with respect to the benzene ring. The C3—C2 [1.3332 (18) Å] and C2—C1 [1.4553 (19) Å] bond lengths are slightly shorter and longer, respectively, than those expected for a C_{ar}—C_{ar} bond. This suggests that the electron density is preferentially located in the C2—C3 bond at the pyrone ring, as seen in other coumarin derivatives (Gomes *et al.*, 2016; Ziki *et al.*, 2017).

In the crystal, weak aromatic π–π stacking interactions are present [$Cg1\cdots Cg2^{i}$ = 3.5337 (8) Å and $Cg2\cdots Cg2^{i}$ = 3.6529 (8) Å, where $Cg1$ and $Cg2$ are the centroids of the coumarin pyran and benzene rings, respectively; symmetry code: (i) $2-x, 1-y, 1-z$]. Together, these lead to a three-dimensional supramolecular network.

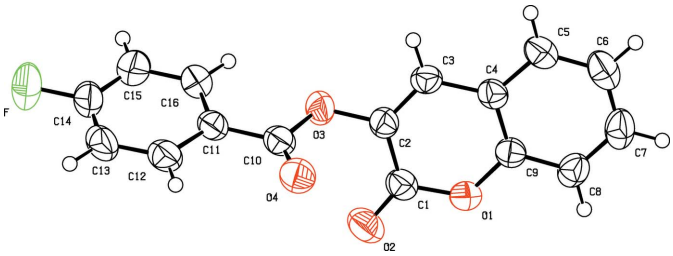


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

To a solution of 4-fluorobenzoyl chloride (6.17 mmol, ≃1 g) in dry tetrahydrofuran (31 ml), was introduced dried triethylamine (3 molar equivalents, ≃2.6 ml). While stirring strongly, 6.17 mmol (1 g) of chroman-2,3-dione was added in small portions over 30 min. The reaction mixture was then refluxed for 4 h and poured into a separating funnel containing 40 ml of chloroform. The solution was acidified with dilute hydrochloric acid until the pH was 2–3. The organic layer was extracted, washed with water until neutral, dried over $MgSO_4$ and the solvent removed. The resulting precipitate (crude product) was washed with petroleum ether and dissolved in a minimum amount of chloroform by heating under agitation. To this hot mixture, hexane was added until the formation of a new precipitate started; this dissolved in the resulting mixture upon heating. Upon cooling, colourless crystals of the title compound precipitated in a yield of 80%, m.p. 452–454 K.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_9FO_4$
M_r	284.23
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	6.8116 (2), 7.2402 (2), 13.4826 (3)
α, β, γ (°)	96.943 (2), 90.862 (2), 106.139 (2)
V (Å^3)	633.21 (3)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.00
Crystal size (mm)	0.36 × 0.26 × 0.16
Data collection	
Diffractometer	Agilent SuperNova, Dual, Cu at zero, AtlasS2
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.788, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12676, 2358, 2112
R_{int}	0.017
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.608
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.100, 1.09
No. of reflections	2358
No. of parameters	191
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.19

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Acknowledgements

The authors thank the Spectropole Service of the Faculty of Sciences (Aix-Marseille, France) for the use of the diffractometer and the NMR and MS spectrometers.

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full crystallographic data

IUCrData (2017). **2**, x170550 [https://doi.org/10.1107/S2414314617005508]

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Crystal data

$C_{16}H_9FO_4$
M_r = 284.23
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.8116 (2) Å
b = 7.2402 (2) Å
c = 13.4826 (3) Å
α = 96.943 (2)°
β = 90.862 (2)°
γ = 106.139 (2)°
V = 633.21 (3) Å^3
Z = 2
$F(000)$ = 292
D_x = 1.491 Mg m^{-3}
Melting point: 452 K
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 7530 reflections
θ = 6.6–69.4°
μ = 1.00 mm^{-1}
T = 293 K
Prism, colourless
0.36 × 0.26 × 0.16 mm

Data collection

Agilent SuperNova, Dual, Cu at zero, AtlasS2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 5.3048 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2014)
T_{min} = 0.788, T_{max} = 1.000
12676 measured reflections
2358 independent reflections
2112 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 69.5°, θ_{min} = 6.4°
h = −8→8
k = −8→8
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.100
S = 1.09
2358 reflections
191 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 0.1315P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: SHELXL2013 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0047 (8)

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	0.28843 (13)	0.32990 (14)	0.54636 (7)	0.0486 (3)
O3	0.13270 (16)	0.55441 (14)	0.77556 (7)	0.0526 (3)
C3	−0.06635 (19)	0.37512 (18)	0.63231 (9)	0.0431 (3)
H3	−0.1840	0.3938	0.6607	0.052*
C2	0.1136 (2)	0.44106 (18)	0.68358 (10)	0.0439 (3)
C4	−0.07706 (19)	0.27525 (17)	0.53310 (10)	0.0414 (3)
C8	0.1081 (2)	0.1647 (2)	0.39686 (10)	0.0526 (4)
H8	0.2313	0.1564	0.3706	0.063*
O2	0.47036 (15)	0.47719 (17)	0.68475 (8)	0.0660 (3)
C9	0.1037 (2)	0.25624 (18)	0.49220 (9)	0.0424 (3)
O4	0.19944 (16)	0.32095 (15)	0.85538 (8)	0.0599 (3)
C5	−0.2591 (2)	0.1964 (2)	0.47421 (11)	0.0509 (3)
H5	−0.3823	0.2080	0.4991	0.061*
C1	0.3038 (2)	0.4204 (2)	0.64253 (10)	0.0470 (3)
C11	0.22979 (19)	0.6336 (2)	0.94604 (10)	0.0471 (3)
C10	0.1894 (2)	0.4839 (2)	0.85772 (10)	0.0473 (3)
F	0.34415 (19)	1.0344 (2)	1.19588 (8)	0.1091 (5)
C6	−0.2564 (3)	0.1018 (2)	0.37969 (12)	0.0602 (4)
H6	−0.3781	0.0482	0.3413	0.072*
C16	0.2040 (2)	0.8158 (2)	0.94244 (11)	0.0550 (4)
H16	0.1610	0.8470	0.8825	0.066*
C15	0.2415 (2)	0.9517 (3)	1.02675 (13)	0.0663 (4)
H15	0.2234	1.0739	1.0247	0.080*
C7	−0.0734 (3)	0.0859 (2)	0.34129 (11)	0.0602 (4)
H7	−0.0733	0.0213	0.2773	0.072*
C12	0.2947 (2)	0.5883 (3)	1.03582 (11)	0.0577 (4)
H12	0.3111	0.4658	1.0390	0.069*
C13	0.3350 (2)	0.7236 (3)	1.12031 (11)	0.0692 (5)
H13	0.3807	0.6949	1.1804	0.083*
C14	0.3060 (2)	0.9012 (3)	1.11331 (12)	0.0703 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0441 (5)	0.0577 (6)	0.0475 (5)	0.0211 (4)	0.0023 (4)	0.0047 (4)
O3	0.0689 (6)	0.0553 (6)	0.0386 (5)	0.0281 (5)	−0.0071 (4)	0.0014 (4)
C3	0.0447 (7)	0.0472 (7)	0.0427 (7)	0.0198 (6)	0.0045 (5)	0.0100 (6)
C2	0.0523 (7)	0.0439 (7)	0.0386 (7)	0.0190 (6)	−0.0022 (5)	0.0053 (5)
C4	0.0465 (7)	0.0389 (6)	0.0412 (7)	0.0148 (5)	−0.0020 (5)	0.0092 (5)

C8	0.0717 (9)	0.0509 (8)	0.0431 (7)	0.0297 (7)	0.0051 (7)	0.0073 (6)
O2	0.0463 (6)	0.0844 (8)	0.0644 (7)	0.0155 (5)	−0.0097 (5)	0.0067 (6)
C9	0.0493 (7)	0.0395 (6)	0.0420 (7)	0.0174 (5)	−0.0010 (5)	0.0084 (5)
O4	0.0687 (7)	0.0593 (6)	0.0574 (6)	0.0261 (5)	−0.0023 (5)	0.0121 (5)
C5	0.0484 (7)	0.0501 (8)	0.0546 (8)	0.0136 (6)	−0.0070 (6)	0.0104 (6)
C1	0.0470 (7)	0.0487 (7)	0.0471 (7)	0.0155 (6)	−0.0033 (6)	0.0085 (6)
C11	0.0377 (6)	0.0651 (9)	0.0397 (7)	0.0162 (6)	0.0005 (5)	0.0075 (6)
C10	0.0421 (7)	0.0588 (8)	0.0438 (7)	0.0175 (6)	0.0001 (5)	0.0101 (6)
F	0.1002 (9)	0.1531 (12)	0.0620 (7)	0.0413 (8)	−0.0124 (6)	−0.0444 (7)
C6	0.0707 (10)	0.0504 (8)	0.0558 (9)	0.0126 (7)	−0.0200 (7)	0.0063 (7)
C16	0.0528 (8)	0.0656 (9)	0.0471 (8)	0.0201 (7)	−0.0054 (6)	0.0017 (7)
C15	0.0600 (9)	0.0731 (10)	0.0630 (10)	0.0225 (8)	−0.0029 (7)	−0.0103 (8)
C7	0.0944 (12)	0.0479 (8)	0.0418 (8)	0.0275 (8)	−0.0080 (8)	0.0027 (6)
C12	0.0478 (8)	0.0856 (11)	0.0455 (8)	0.0253 (7)	0.0033 (6)	0.0156 (7)
C13	0.0516 (9)	0.1223 (16)	0.0361 (8)	0.0295 (9)	0.0000 (6)	0.0087 (8)
C14	0.0496 (8)	0.1086 (14)	0.0460 (9)	0.0234 (9)	−0.0019 (7)	−0.0175 (9)

Geometric parameters (Å, º)

O1—C1	1.3688 (16)	C5—H5	0.9300
O1—C9	1.3804 (15)	C11—C16	1.385 (2)
O3—C10	1.3676 (16)	C11—C12	1.389 (2)
O3—C2	1.3849 (15)	C11—C10	1.4765 (19)
C3—C2	1.3332 (18)	F—C14	1.3525 (18)
C3—C4	1.4322 (17)	C6—C7	1.386 (2)
C3—H3	0.9300	C6—H6	0.9300
C2—C1	1.4553 (19)	C16—C15	1.381 (2)
C4—C9	1.3914 (18)	C16—H16	0.9300
C4—C5	1.4005 (18)	C15—C14	1.366 (3)
C8—C7	1.376 (2)	C15—H15	0.9300
C8—C9	1.3779 (18)	C7—H7	0.9300
C8—H8	0.9300	C12—C13	1.380 (2)
O2—C1	1.2018 (16)	C12—H12	0.9300
O4—C10	1.1974 (17)	C13—C14	1.368 (3)
C5—C6	1.375 (2)	C13—H13	0.9300
C1—O1—C9	122.55 (10)	C12—C11—C10	118.42 (14)
C10—O3—C2	118.12 (10)	O4—C10—O3	122.73 (13)
C2—C3—C4	119.50 (12)	O4—C10—C11	126.62 (13)
C2—C3—H3	120.2	O3—C10—C11	110.64 (12)
C4—C3—H3	120.2	C5—C6—C7	120.37 (14)
C3—C2—O3	120.88 (12)	C5—C6—H6	119.8
C3—C2—C1	122.92 (12)	C7—C6—H6	119.8
O3—C2—C1	115.77 (11)	C15—C16—C11	120.84 (15)
C9—C4—C5	117.83 (12)	C15—C16—H16	119.6
C9—C4—C3	118.33 (11)	C11—C16—H16	119.6
C5—C4—C3	123.83 (12)	C14—C15—C16	117.96 (17)
C7—C8—C9	118.66 (14)	C14—C15—H15	121.0

C7—C8—H8	120.7	C16—C15—H15	121.0
C9—C8—H8	120.7	C8—C7—C6	120.63 (14)
C8—C9—O1	116.95 (12)	C8—C7—H7	119.7
C8—C9—C4	122.25 (12)	C6—C7—H7	119.7
O1—C9—C4	120.80 (11)	C13—C12—C11	120.52 (16)
C6—C5—C4	120.23 (14)	C13—C12—H12	119.7
C6—C5—H5	119.9	C11—C12—H12	119.7
C4—C5—H5	119.9	C14—C13—C12	118.18 (15)
O2—C1—O1	118.12 (13)	C14—C13—H13	120.9
O2—C1—C2	126.03 (13)	C12—C13—H13	120.9
O1—C1—C2	115.84 (11)	F—C14—C15	118.14 (19)
C16—C11—C12	119.20 (14)	F—C14—C13	118.57 (16)
C16—C11—C10	122.38 (12)	C15—C14—C13	123.29 (15)
C4—C3—C2—O3	173.63 (11)	O3—C2—C1—O1	−171.86 (10)
C4—C3—C2—C1	1.4 (2)	C2—O3—C10—O4	−9.0 (2)
C10—O3—C2—C3	117.23 (14)	C2—O3—C10—C11	172.17 (11)
C10—O3—C2—C1	−70.06 (16)	C16—C11—C10—O4	−176.64 (14)
C2—C3—C4—C9	−2.15 (18)	C12—C11—C10—O4	3.0 (2)
C2—C3—C4—C5	178.32 (12)	C16—C11—C10—O3	2.11 (18)
C7—C8—C9—O1	178.67 (11)	C12—C11—C10—O3	−178.30 (12)
C7—C8—C9—C4	−1.5 (2)	C4—C5—C6—C7	−0.9 (2)
C1—O1—C9—C8	−178.64 (11)	C12—C11—C16—C15	−0.2 (2)
C1—O1—C9—C4	1.50 (18)	C10—C11—C16—C15	179.41 (13)
C5—C4—C9—C8	0.44 (19)	C11—C16—C15—C14	0.5 (2)
C3—C4—C9—C8	−179.12 (12)	C9—C8—C7—C6	1.3 (2)
C5—C4—C9—O1	−179.71 (11)	C5—C6—C7—C8	−0.2 (2)
C3—C4—C9—O1	0.73 (18)	C16—C11—C12—C13	−0.6 (2)
C9—C4—C5—C6	0.8 (2)	C10—C11—C12—C13	179.79 (13)
C3—C4—C5—C6	−179.72 (12)	C11—C12—C13—C14	1.1 (2)
C9—O1—C1—O2	178.72 (12)	C16—C15—C14—F	179.56 (14)
C9—O1—C1—C2	−2.18 (18)	C16—C15—C14—C13	0.0 (3)
C3—C2—C1—O2	179.71 (14)	C12—C13—C14—F	179.68 (14)
O3—C2—C1—O2	7.2 (2)	C12—C13—C14—C15	−0.8 (3)
C3—C2—C1—O1	0.69 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O3	0.93	2.37	2.7031 (17)	101