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Cinnamyl 8-methoxy-2-oxo-2*H*-chromene-3-carboxylate

Cui-Lian Xu,[a]* Shan-Yu Liu,[b] Cai-Xia Wang[a] and Ming-Qin Zhao[b]

[a]College of Sciences, Henan Agricultural University, Zhengzhou 450002, People's Republic of China, and [b]College of Tobacco Science, Henan Agricultural University, Zhengzhou 450002, People's Republic of China
Correspondence e-mail: xucuilian666@126.com

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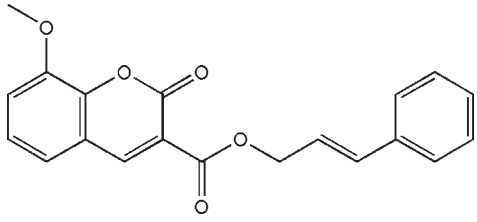
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.065; wR factor = 0.155; data-to-parameter ratio = 12.4.

In the crystal structure of the title compound, $C_{20}H_{16}O_5$, the molecule assumes an *E* configuration with the benzene ring and chromenecarboxyl group located on opposite ends of the C=C double bond. The chromene ring system and benzene ring are oriented at a dihedral angle of 74.66 (12)°. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure.

Related literature

For applications of coumarins and related compounds, see: Hoult & Paya (1996); Yu *et al.* (2003, 2007); Finn *et al.* (2004).



Experimental

Crystal data

$C_{20}H_{16}O_5$ M_r = 336.33
Monoclinic, $P2_1/c$
a = 19.226 (4) Å
b = 9.5483 (19) Å
c = 9.0046 (18) Å
β = 90.97 (3)°
V = 1652.8 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.20 × 0.17 × 0.17 mm

Data collection

Bruker SMART CCD area detector diffractometer
Absorption correction: none
4903 measured reflections
2834 independent reflections
2157 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.155
S = 1.15
2834 reflections
228 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 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
C4—H4A···O3^{i}	0.93	2.51	3.429 (3)	170
C17—H17A···O4^{ii}	0.93	2.44	3.294 (4)	153

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: XU2613).

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supporting information

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Cinnamyl 8-methoxy-2-oxo-2*H*-chromene-3-carboxylate

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S1. Comment

Coumarins and related compounds, kinds of plant-derived compounds, have diverse biological activities, including anti-HIV, anti-bacterial, anti-inflammatory, anti-proliferative and antioxidant properties (Hoult & Paya, 1996; Yu *et al.*, 2003; Finn *et al.*, 2004; Yu *et al.*, 2007). It thus appeared of interest to synthesize the compounds with coumarin-skeleton. As part of work, we have synthesized the title compound (I) and report its crystal structure here.

The title molecule crystallizes in the E conformation, with an C12-C13-C14-C15 torsion angle of -179.5 (3)°. The 8-methoxy-2*H*-chromen-2-one ring and the C15-benzene ring make a dihedral of 74.66 (12)°.

In the crystal structure, an intramolecular C—H···O hydrogen bond is observed and helps to stablize the conformation of the molecule.

S2. Experimental

A solution of cinnamic alcohol (7.2 mmol) dissolved in dried methyl dichloride (DCM) (25 ml) was added dropwise to a solution of 2-oxo-2*H*-chromene-3-acyl chloride (7.2 mmol) dissolved in DCM (25 ml) and triethylamine (1 ml) at room temperature. The reaction mixture was stirred for 24 h, monitored by TLC. The reaction mixture was neutralized with 5% HCl and washed by saturated $NaHCO_3$ solution and brine, respectively. The organic phase is dried over Na_2SO_4 and evaporated under the reduced pressure. The resulting residue was purified by column chromatography (EtOAc:petroleum ether) to give the purified compound.

S3. Refinement

All H atoms were positioned geometrically and refined as riding with C—H = 0.93 (aromatic), 0.97 (methylene) and 0.96 Å (methyl), $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for the others.

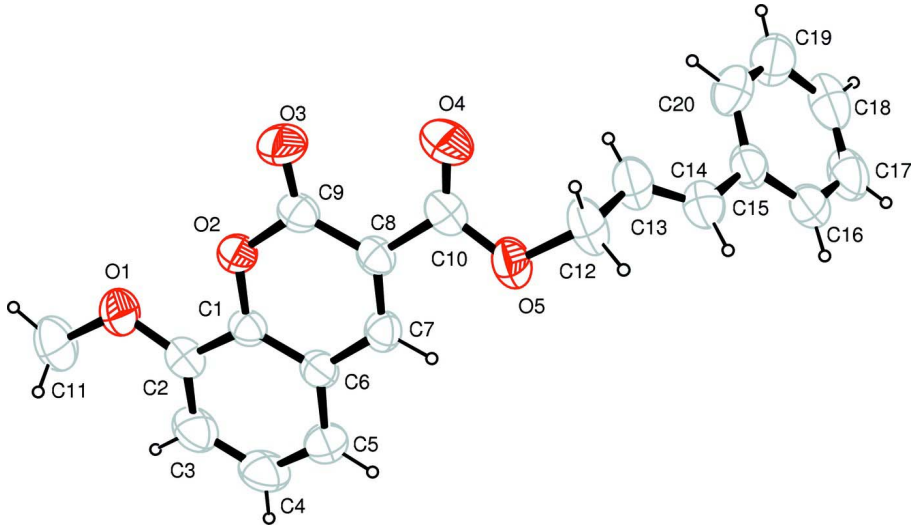

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

cinnamyl 8-methoxy-2-oxo-2*H*-chromene-3-carboxylate

Crystal data

$C_{20}H_{16}O_5$
$M_r = 336.33$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 19.226\ (4)$ Å
$b = 9.5483\ (19)$ Å
$c = 9.0046\ (18)$ Å
$\beta = 90.97\ (3)°$
$V = 1652.8\ (6)$ Å^3
$Z = 4$
$F(000) = 704$
$D_x = 1.352$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2834 reflections
$\theta = 3.1–24.2°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, colorless
0.20 × 0.17 × 0.17 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
4903 measured reflections
2834 independent reflections
2157 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.1°$
$h = -22\rightarrow22$
$k = -11\rightarrow11$
$l = 0\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.155$
$S = 1.15$
2834 reflections
228 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
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2 + 0.2012P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.024 (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. 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 $F^2 > \sigma(F^2)$ 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	0.04001 (9)	0.88854 (18)	0.70962 (19)	0.0610 (5)
O2	0.11713 (7)	0.72996 (15)	0.54027 (17)	0.0463 (4)
O3	0.15130 (10)	0.51497 (17)	0.4995 (2)	0.0705 (6)
O4	0.25790 (11)	0.4732 (2)	0.3000 (3)	0.0885 (7)
O5	0.28952 (8)	0.66346 (19)	0.17300 (19)	0.0617 (5)
C1	0.12199 (11)	0.8722 (2)	0.5210 (2)	0.0414 (6)
C2	0.08113 (11)	0.9568 (3)	0.6115 (3)	0.0497 (6)
C3	0.08597 (14)	1.0996 (3)	0.5946 (3)	0.0652 (8)
H3A	0.0593	1.1578	0.6536	0.078*
C4	0.13001 (15)	1.1585 (3)	0.4910 (3)	0.0710 (8)
H4A	0.1323	1.2554	0.4816	0.085*
C5	0.17013 (13)	1.0757 (3)	0.4023 (3)	0.0605 (7)
H5A	0.1992	1.1160	0.3328	0.073*
C6	0.16698 (11)	0.9293 (2)	0.4174 (3)	0.0445 (6)
C7	0.20856 (11)	0.8339 (2)	0.3351 (2)	0.0450 (6)
H7A	0.2383	0.8693	0.2638	0.054*
C8	0.20641 (10)	0.6940 (2)	0.3568 (2)	0.0429 (6)
C9	0.15868 (11)	0.6355 (2)	0.4665 (3)	0.0460 (6)
C10	0.25260 (12)	0.5959 (3)	0.2770 (3)	0.0525 (6)
C11	−0.00432 (15)	0.9739 (3)	0.7994 (3)	0.0743 (8)
H11A	−0.0314	0.9150	0.8626	0.112*
H11B	−0.0349	1.0278	0.7362	0.112*
H11C	0.0237	1.0360	0.8592	0.112*
C12	0.33877 (13)	0.5785 (3)	0.0894 (3)	0.0697 (8)
H12A	0.3470	0.6224	−0.0059	0.084*
H12B	0.3189	0.4867	0.0709	0.084*
C13	0.40569 (13)	0.5629 (3)	0.1714 (3)	0.0646 (7)
H13A	0.4053	0.5142	0.2608	0.078*
C14	0.46490 (14)	0.6123 (3)	0.1279 (3)	0.0639 (7)
H14A	0.4640	0.6613	0.0386	0.077*
C15	0.53343 (12)	0.6000 (3)	0.2031 (3)	0.0560 (7)
C16	0.58725 (14)	0.6863 (3)	0.1617 (4)	0.0741 (9)

H16A	0.5804	0.7481	0.0830	0.089*
C17	0.65113 (15)	0.6830 (3)	0.2345 (5)	0.0831 (10)
H17A	0.6861	0.7445	0.2067	0.100*
C18	0.66314 (14)	0.5891 (3)	0.3481 (4)	0.0741 (9)
H18A	0.7059	0.5874	0.3980	0.089*
C19	0.61138 (15)	0.4981 (4)	0.3866 (3)	0.0763 (9)
H19A	0.6195	0.4320	0.4608	0.092*
C20	0.54716 (14)	0.5041 (3)	0.3158 (3)	0.0694 (8)
H20A	0.5123	0.4426	0.3443	0.083*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0600 (10)	0.0639 (11)	0.0597 (11)	0.0079 (9)	0.0171 (8)	−0.0059 (9)
O2	0.0487 (9)	0.0410 (9)	0.0494 (10)	0.0015 (7)	0.0053 (7)	−0.0008 (7)
O3	0.0869 (14)	0.0375 (10)	0.0877 (14)	0.0016 (9)	0.0227 (10)	0.0041 (9)
O4	0.1028 (16)	0.0562 (13)	0.1075 (17)	0.0296 (11)	0.0322 (13)	0.0052 (11)
O5	0.0522 (10)	0.0719 (12)	0.0615 (12)	0.0113 (9)	0.0125 (8)	−0.0069 (9)
C1	0.0391 (12)	0.0364 (12)	0.0486 (14)	0.0004 (10)	−0.0026 (10)	−0.0021 (10)
C2	0.0444 (13)	0.0505 (15)	0.0541 (15)	0.0053 (11)	−0.0019 (11)	−0.0051 (12)
C3	0.0668 (17)	0.0523 (16)	0.0767 (19)	0.0122 (13)	0.0061 (14)	−0.0112 (14)
C4	0.0784 (19)	0.0382 (14)	0.096 (2)	0.0072 (13)	0.0005 (17)	−0.0028 (15)
C5	0.0590 (16)	0.0446 (14)	0.0783 (19)	−0.0029 (12)	0.0079 (13)	0.0063 (13)
C6	0.0394 (12)	0.0392 (12)	0.0550 (15)	0.0021 (10)	−0.0010 (10)	−0.0012 (11)
C7	0.0370 (12)	0.0503 (14)	0.0477 (14)	−0.0026 (10)	0.0007 (9)	0.0011 (11)
C8	0.0364 (12)	0.0444 (13)	0.0476 (14)	0.0026 (10)	−0.0039 (10)	−0.0026 (10)
C9	0.0472 (13)	0.0401 (14)	0.0506 (15)	0.0028 (10)	−0.0024 (10)	−0.0044 (11)
C10	0.0470 (14)	0.0573 (16)	0.0531 (16)	0.0102 (12)	−0.0031 (11)	−0.0058 (13)
C11	0.0676 (18)	0.090 (2)	0.0663 (19)	0.0167 (16)	0.0152 (14)	−0.0173 (16)
C12	0.0520 (16)	0.093 (2)	0.0642 (18)	0.0203 (14)	0.0082 (13)	−0.0162 (16)
C13	0.0517 (15)	0.0826 (19)	0.0598 (17)	0.0113 (14)	0.0072 (12)	−0.0058 (15)
C14	0.0605 (16)	0.0681 (17)	0.0635 (17)	0.0112 (14)	0.0127 (13)	−0.0002 (14)
C15	0.0484 (14)	0.0513 (14)	0.0690 (18)	0.0042 (12)	0.0139 (12)	−0.0074 (13)
C16	0.0617 (18)	0.0521 (16)	0.109 (2)	0.0106 (14)	0.0295 (16)	0.0079 (16)
C17	0.0542 (18)	0.0535 (17)	0.143 (3)	−0.0068 (14)	0.0329 (18)	−0.012 (2)
C18	0.0490 (16)	0.078 (2)	0.096 (2)	0.0006 (15)	0.0078 (15)	−0.0310 (19)
C19	0.0669 (18)	0.091 (2)	0.071 (2)	0.0004 (17)	0.0080 (15)	0.0048 (16)
C20	0.0549 (16)	0.0741 (19)	0.080 (2)	−0.0136 (14)	0.0122 (14)	0.0070 (16)

Geometric parameters (Å, º)

O1—C2	1.362 (3)	C11—H11A	0.9600
O1—C11	1.438 (3)	C11—H11B	0.9600
O2—C1	1.373 (3)	C11—H11C	0.9600
O2—C9	1.382 (3)	C12—C13	1.480 (4)
O3—C9	1.198 (3)	C12—H12A	0.9700
O4—C10	1.194 (3)	C12—H12B	0.9700
O5—C10	1.349 (3)	C13—C14	1.299 (4)

O5—C12	1.465 (3)	C13—H13A	0.9300
C1—C6	1.394 (3)	C14—C15	1.476 (4)
C1—C2	1.398 (3)	C14—H14A	0.9300
C2—C3	1.375 (4)	C15—C16	1.379 (3)
C3—C4	1.389 (4)	C15—C20	1.389 (4)
C3—H3A	0.9300	C16—C17	1.383 (4)
C4—C5	1.370 (4)	C16—H16A	0.9300
C4—H4A	0.9300	C17—C18	1.377 (4)
C5—C6	1.405 (3)	C17—H17A	0.9300
C5—H5A	0.9300	C18—C19	1.370 (4)
C6—C7	1.428 (3)	C18—H18A	0.9300
C7—C8	1.351 (3)	C19—C20	1.381 (4)
C7—H7A	0.9300	C19—H19A	0.9300
C8—C9	1.469 (3)	C20—H20A	0.9300
C8—C10	1.485 (3)		

C2—O1—C11	116.7 (2)	H11A—C11—H11B	109.5
C1—O2—C9	122.93 (18)	O1—C11—H11C	109.5
C10—O5—C12	116.4 (2)	H11A—C11—H11C	109.5
O2—C1—C6	121.0 (2)	H11B—C11—H11C	109.5
O2—C1—C2	117.3 (2)	O5—C12—C13	111.3 (2)
C6—C1—C2	121.7 (2)	O5—C12—H12A	109.4
O1—C2—C3	126.0 (2)	C13—C12—H12A	109.4
O1—C2—C1	116.1 (2)	O5—C12—H12B	109.4
C3—C2—C1	117.9 (2)	C13—C12—H12B	109.4
C2—C3—C4	121.3 (3)	H12A—C12—H12B	108.0
C2—C3—H3A	119.4	C14—C13—C12	124.9 (3)
C4—C3—H3A	119.4	C14—C13—H13A	117.6
C5—C4—C3	120.9 (2)	C12—C13—H13A	117.6
C5—C4—H4A	119.6	C13—C14—C15	127.8 (3)
C3—C4—H4A	119.6	C13—C14—H14A	116.1
C4—C5—C6	119.5 (2)	C15—C14—H14A	116.1
C4—C5—H5A	120.3	C16—C15—C20	117.2 (3)
C6—C5—H5A	120.3	C16—C15—C14	119.8 (3)
C1—C6—C5	118.8 (2)	C20—C15—C14	123.0 (2)
C1—C6—C7	117.2 (2)	C15—C16—C17	121.5 (3)
C5—C6—C7	124.0 (2)	C15—C16—H16A	119.2
C8—C7—C6	122.5 (2)	C17—C16—H16A	119.2
C8—C7—H7A	118.7	C18—C17—C16	120.2 (3)
C6—C7—H7A	118.7	C18—C17—H17A	119.9
C7—C8—C9	119.6 (2)	C16—C17—H17A	119.9
C7—C8—C10	122.2 (2)	C19—C18—C17	119.2 (3)
C9—C8—C10	118.1 (2)	C19—C18—H18A	120.4
O3—C9—O2	115.8 (2)	C17—C18—H18A	120.4
O3—C9—C8	127.6 (2)	C18—C19—C20	120.3 (3)
O2—C9—C8	116.62 (19)	C18—C19—H19A	119.9
O4—C10—O5	123.1 (2)	C20—C19—H19A	119.9
O4—C10—C8	125.8 (3)	C19—C20—C15	121.5 (3)

O5—C10—C8	111.2 (2)	C19—C20—H20A	119.3
O1—C11—H11A	109.5	C15—C20—H20A	119.3
O1—C11—H11B	109.5		

C9—O2—C1—C6	4.0 (3)	C7—C8—C9—O3	−179.1 (2)
C9—O2—C1—C2	−174.71 (19)	C10—C8—C9—O3	−1.1 (3)
C11—O1—C2—C3	2.6 (4)	C7—C8—C9—O2	1.1 (3)
C11—O1—C2—C1	−177.7 (2)	C10—C8—C9—O2	179.11 (18)
O2—C1—C2—O1	−0.4 (3)	C12—O5—C10—O4	−1.3 (3)
C6—C1—C2—O1	−179.15 (19)	C12—O5—C10—C8	178.20 (18)
O2—C1—C2—C3	179.3 (2)	C7—C8—C10—O4	172.9 (3)
C6—C1—C2—C3	0.6 (3)	C9—C8—C10—O4	−5.0 (4)
O1—C2—C3—C4	179.8 (2)	C7—C8—C10—O5	−6.5 (3)
C1—C2—C3—C4	0.0 (4)	C9—C8—C10—O5	175.55 (18)
C2—C3—C4—C5	−0.1 (4)	C10—O5—C12—C13	−83.6 (3)
C3—C4—C5—C6	−0.5 (4)	O5—C12—C13—C14	−115.1 (3)
O2—C1—C6—C5	−179.8 (2)	C12—C13—C14—C15	−179.5 (3)
C2—C1—C6—C5	−1.2 (3)	C13—C14—C15—C16	−164.1 (3)
O2—C1—C6—C7	−1.3 (3)	C13—C14—C15—C20	15.9 (4)
C2—C1—C6—C7	177.37 (19)	C20—C15—C16—C17	−3.4 (4)
C4—C5—C6—C1	1.1 (4)	C14—C15—C16—C17	176.6 (3)
C4—C5—C6—C7	−177.4 (2)	C15—C16—C17—C18	2.2 (4)
C1—C6—C7—C8	−1.3 (3)	C16—C17—C18—C19	0.7 (4)
C5—C6—C7—C8	177.1 (2)	C17—C18—C19—C20	−2.3 (4)
C6—C7—C8—C9	1.4 (3)	C18—C19—C20—C15	0.9 (4)
C6—C7—C8—C10	−176.50 (19)	C16—C15—C20—C19	1.9 (4)
C1—O2—C9—O3	176.3 (2)	C14—C15—C20—C19	−178.2 (3)
C1—O2—C9—C8	−3.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4*A*···O3^{i}	0.93	2.51	3.429 (3)	170
C17—H17*A*···O4^{ii}	0.93	2.44	3.294 (4)	153

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