IUCrData

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(3*E*)-3-[(2*E*)-3-(4-Methoxyphenyl)prop-2-enylidene]-2,3-dihydro-4*H*-chromen-4-one

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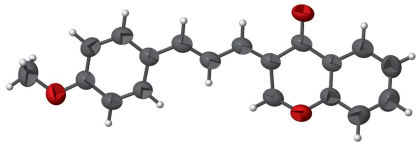
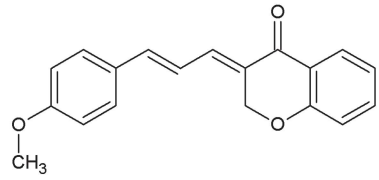
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{19}H_{16}O_3$, the dihedral angle between the chromanone moiety and the methoxy phenyl ring is 16.47 (1)°. In the crystal, the molecules are linked by pairs of C—H···O hydrogen bonds, generating $R_2^2(14)$ inversion dimers; further C—H···O hydrogen bonds connect the dimers into [100] double chains.

3D view



Chemical scheme



Structure description

A large number of naturally occurring chalcones are polyhydroxylated in the aryl rings (Jasinski *et al.*, 2010). The radical quenching properties of the phenolic groups present in many chalcones have raised interest in using these compounds or chalcone-rich plant extracts as drugs or food preservatives (Dhar, 1981). As part of our studies in this area, we now describe the synthesis and structure of the title compound (Fig. 1).

The tetrahydro-4*H*-pyran-4-one ring adopts a sofa conformation with the methylene group (atom C12) displaced from the other atoms. The C—O and C═O distances in the chromanone moiety are typical of those in previously reported structures (Gopaul *et al.*, 2012). The dihedral angle between the the methoxy phenyl ring and chromanone ring system (all atoms) is 16.47 (1)°.

In the crystal, the molecules are linked by C—H···O hydrogen bonds (Table 1). Inversion dimers featuring R_2^2 (14) loops arise from the very weak C8—H8···O2 bonds and the dimers are linked into [100] double chains by the C17—H17···O3 bonds (Figs. 2 and 3).

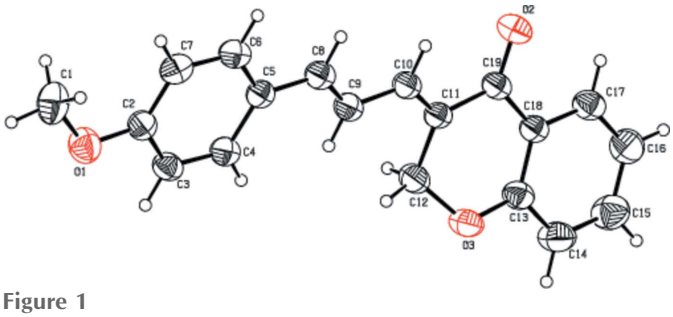


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

Synthesis and crystallization

In a 250 ml round-bottomed flask, a mixture of 4-chromanone (1.4 g, 0.010 mol) and methoxy cinnamaldehyde (1.5 g, 0.010 mol) was added to absolute alcohol and stirred for five minutes. Then, a solution of NaOH (0.3 g, 10 ml) was added and stirred for 2 h. The mixture was kept overnight at room temperature and then dumped into crushed ice, leading to a precipitate of the title compound, which was isolated by filtration and washed with distilled water several times to remove any trace of NaOH remaining in the product. The crude chalcone derivative was recrystallized twice from ethyl methylketone solution to give colourless blocks of the title compound (yield: 80%; mp: 135°C).

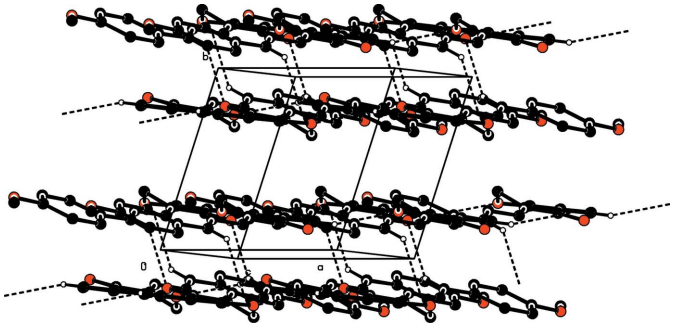


Figure 2
The packing of the molecules in the crystal structure. The dashed lines indicate hydrogen bonds.

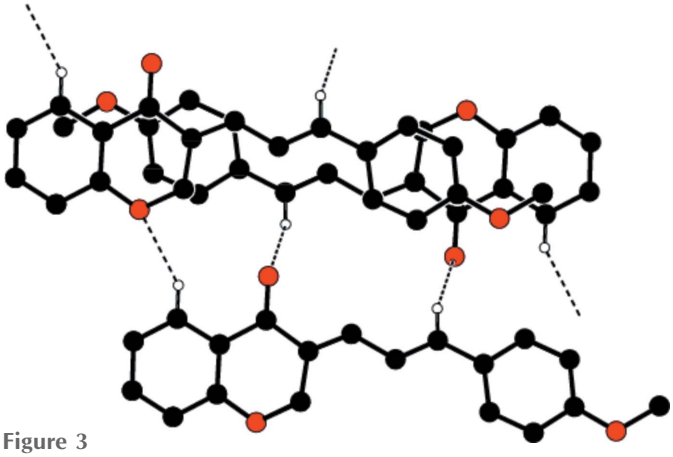


Figure 3
A partial view of the packing of the title compound showing the hydrogen bonds as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2^{i}	0.93	2.59	3.362 (5)	141
C17—H17···O3^{ii}	0.93	2.53	3.342 (5)	146

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{16}O_3$
M_r	292.32
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	6.8573 (12), 7.4073 (14), 15.550 (3)
α, β, γ (°)	87.843 (6), 78.892 (5), 72.347 (5)
V (Å^3)	738.4 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.15 × 0.15 × 0.10
Data collection	
Diffractometer	Bruker APEXIII CMOS
Absorption correction	Multi-scan (*SADABS*)
T_{min}, T_{max}	0.987, 0.991
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20521, 2589, 1609
R_{int}	0.092
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.080, 0.251, 1.12
No. of reflections	2580
No. of parameters	201
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.25

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *ORTEP-3 for Windows* (Farrugia, 2012).

Refinement

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

Acknowledgements

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References

Bruker (2016). *APEX3*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dhar, D. N. (1981). *The Chemistry of Chalcones and Related Compounds*. New York: John Wiley.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Gopaul, K., Shaikh, M. M., Koorbanally, N. A., Ramjugernath, D. & Omondi, B. (2012). *Acta Cryst.* E**68**, o1972.

Jasinski, J. P., Pek, A. E., Narayana, B., Kamath, P. K. & Yathirajan, H. S. (2010). *Acta Cryst.* E**66**, o1995.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2018). **3**, x180829 [https://doi.org/10.1107/S2414314618008295]

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Crystal data

$C_{19}H_{16}O_3$
M_r = 292.32
Triclinic, $P\overline{1}$
a = 6.8573 (12) Å
b = 7.4073 (14) Å
c = 15.550 (3) Å
α = 87.843 (6)°
β = 78.892 (5)°
γ = 72.347 (5)°
V = 738.4 (2) Å^3
Z = 2
F(000) = 308
D_x = 1.315 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7704 reflections
θ = 3.2–26.7°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.15 × 0.15 × 0.10 mm

Data collection

Bruker APEXIII CMOS diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS)
T_{min} = 0.987, T_{max} = 0.991
20521 measured reflections
2589 independent reflections
1609 reflections with $I > 2\sigma(I)$
R_{int} = 0.092
θ_{max} = 25.0°, θ_{min} = 2.9°
h = −8→8
k = −8→8
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.080
$wR(F^2)$ = 0.251
S = 1.12
2580 reflections
201 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0879P)^2 + 0.7561P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.09 (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. H atoms were positioned geometrically and treated as riding on their parent atoms and refined with, C—H distance of 0.93–0.97 Å, with U_{iso}(H)= 1.5 U_{eq}(c-methyl),and U_{iso}(H)= 1.2Ueq(C) for other H atom.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.3016 (8)	0.3018 (7)	0.8572 (3)	0.0924 (15)
H1A	−0.2325	0.3744	0.8833	0.139*
H1B	−0.2454	0.1701	0.8693	0.139*
H1C	−0.4481	0.3440	0.8814	0.139*
C2	−0.0725 (6)	0.2889 (5)	0.7197 (3)	0.0597 (10)
C3	−0.0532 (6)	0.3314 (5)	0.6320 (3)	0.0628 (10)
H3	−0.1725	0.3833	0.6085	0.075*
C4	0.1369 (6)	0.2990 (5)	0.5794 (3)	0.0605 (10)
H4	0.1451	0.3291	0.5205	0.073*
C5	0.3203 (6)	0.2214 (5)	0.6116 (2)	0.0539 (9)
C8	0.5250 (6)	0.1928 (5)	0.5585 (3)	0.0596 (10)
H8	0.6378	0.1365	0.5855	0.072*
C9	0.5699 (6)	0.2387 (5)	0.4746 (3)	0.0593 (10)
H9	0.4594	0.2937	0.4462	0.071*
C10	0.7755 (6)	0.2092 (5)	0.4260 (3)	0.0567 (10)
H10	0.8838	0.1566	0.4560	0.068*
C11	0.8297 (5)	0.2488 (5)	0.3421 (2)	0.0524 (9)
C19	1.0486 (6)	0.2170 (5)	0.3014 (3)	0.0555 (10)
C18	1.0927 (5)	0.2306 (5)	0.2055 (2)	0.0537 (9)
C17	1.2938 (6)	0.2108 (6)	0.1601 (3)	0.0682 (11)
H17	1.4000	0.1982	0.1913	0.082*
C16	1.3383 (8)	0.2095 (7)	0.0702 (3)	0.0831 (13)
H16	1.4731	0.1976	0.0405	0.100*
C15	1.1807 (9)	0.2259 (8)	0.0246 (3)	0.0949 (16)
H15	1.2105	0.2234	−0.0363	0.114*
C7	0.1057 (7)	0.2083 (5)	0.7534 (3)	0.0651 (11)
H7	0.0958	0.1757	0.8120	0.078*
C6	0.2986 (6)	0.1764 (5)	0.6996 (3)	0.0629 (11)
H6	0.4177	0.1230	0.7230	0.076*
C14	0.9823 (8)	0.2457 (8)	0.0671 (3)	0.0875 (14)
H14	0.8775	0.2567	0.0353	0.105*
C13	0.9370 (6)	0.2496 (6)	0.1575 (3)	0.0635 (10)
C12	0.6740 (6)	0.3353 (6)	0.2856 (3)	0.0693 (11)
H12A	0.5441	0.3102	0.3106	0.083*
H12B	0.6476	0.4715	0.2862	0.083*
O1	−0.2702 (5)	0.3267 (5)	0.7659 (2)	0.0835 (10)
O2	1.1894 (4)	0.1790 (4)	0.3435 (2)	0.0788 (10)

O3	0.7373 (4)	0.2668 (4)	0.19599 (18)	0.0742 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.100 (4)	0.077 (3)	0.083 (3)	−0.016 (3)	0.006 (3)	0.005 (3)
C2	0.061 (2)	0.050 (2)	0.070 (3)	−0.0191 (17)	−0.0130 (19)	−0.0013 (18)
C3	0.060 (2)	0.055 (2)	0.076 (3)	−0.0138 (17)	−0.024 (2)	0.0037 (19)
C4	0.063 (2)	0.054 (2)	0.067 (2)	−0.0182 (18)	−0.0183 (19)	0.0042 (18)
C5	0.060 (2)	0.0371 (18)	0.069 (2)	−0.0184 (16)	−0.0165 (18)	−0.0016 (16)
C8	0.063 (2)	0.044 (2)	0.077 (3)	−0.0185 (17)	−0.0219 (19)	0.0022 (18)
C9	0.058 (2)	0.043 (2)	0.079 (3)	−0.0128 (16)	−0.0227 (19)	−0.0006 (18)
C10	0.056 (2)	0.0421 (19)	0.074 (3)	−0.0129 (16)	−0.0214 (18)	−0.0018 (17)
C11	0.055 (2)	0.0378 (18)	0.068 (2)	−0.0145 (15)	−0.0211 (17)	−0.0020 (16)
C19	0.054 (2)	0.0365 (18)	0.082 (3)	−0.0156 (15)	−0.0239 (19)	0.0010 (16)
C18	0.056 (2)	0.0395 (18)	0.071 (2)	−0.0181 (15)	−0.0192 (18)	0.0013 (16)
C17	0.068 (3)	0.059 (2)	0.086 (3)	−0.0256 (19)	−0.023 (2)	0.004 (2)
C16	0.078 (3)	0.090 (3)	0.086 (3)	−0.037 (3)	−0.008 (2)	0.002 (3)
C15	0.100 (4)	0.120 (4)	0.073 (3)	−0.047 (3)	−0.014 (3)	0.005 (3)
C7	0.083 (3)	0.057 (2)	0.062 (2)	−0.029 (2)	−0.019 (2)	0.0067 (18)
C6	0.071 (3)	0.049 (2)	0.075 (3)	−0.0195 (18)	−0.028 (2)	0.0075 (18)
C14	0.088 (3)	0.112 (4)	0.075 (3)	−0.039 (3)	−0.029 (3)	0.006 (3)
C13	0.061 (2)	0.062 (2)	0.074 (3)	−0.0238 (18)	−0.020 (2)	0.0042 (19)
C12	0.058 (2)	0.070 (3)	0.081 (3)	−0.0162 (19)	−0.021 (2)	0.001 (2)
O1	0.074 (2)	0.090 (2)	0.080 (2)	−0.0201 (16)	−0.0068 (16)	0.0035 (16)
O2	0.0637 (18)	0.090 (2)	0.087 (2)	−0.0200 (15)	−0.0310 (15)	0.0082 (16)
O3	0.0652 (18)	0.092 (2)	0.0743 (19)	−0.0280 (15)	−0.0279 (14)	0.0066 (15)

Geometric parameters (Å, º)

C1—O1	1.408 (5)	C11—C12	1.492 (5)
C1—H1A	0.9600	C19—O2	1.227 (4)
C1—H1B	0.9600	C19—C18	1.469 (5)
C1—H1C	0.9600	C18—C13	1.386 (5)
C2—O1	1.357 (5)	C18—C17	1.390 (5)
C2—C3	1.378 (5)	C17—C16	1.373 (6)
C2—C7	1.380 (6)	C17—H17	0.9300
C3—C4	1.356 (5)	C16—C15	1.376 (7)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.395 (5)	C15—C14	1.360 (6)
C4—H4	0.9300	C15—H15	0.9300
C5—C6	1.386 (5)	C7—C6	1.380 (5)
C5—C8	1.444 (5)	C7—H7	0.9300
C8—C9	1.337 (5)	C6—H6	0.9300
C8—H8	0.9300	C14—C13	1.380 (6)
C9—C10	1.421 (5)	C14—H14	0.9300
C9—H9	0.9300	C13—O3	1.354 (4)
C10—C11	1.334 (5)	C12—O3	1.439 (5)

C10—H10	0.9300	C12—H12A	0.9700
C11—C19	1.462 (5)	C12—H12B	0.9700

O1—C1—H1A	109.5	C13—C18—C17	118.3 (4)
O1—C1—H1B	109.5	C13—C18—C19	120.5 (3)
H1A—C1—H1B	109.5	C17—C18—C19	121.0 (3)
O1—C1—H1C	109.5	C16—C17—C18	121.1 (4)
H1A—C1—H1C	109.5	C16—C17—H17	119.4
H1B—C1—H1C	109.5	C18—C17—H17	119.4
O1—C2—C3	116.0 (3)	C17—C16—C15	119.1 (4)
O1—C2—C7	125.1 (4)	C17—C16—H16	120.5
C3—C2—C7	118.9 (4)	C15—C16—H16	120.5
C4—C3—C2	121.2 (4)	C14—C15—C16	121.2 (5)
C4—C3—H3	119.4	C14—C15—H15	119.4
C2—C3—H3	119.4	C16—C15—H15	119.4
C3—C4—C5	121.5 (4)	C2—C7—C6	119.7 (4)
C3—C4—H4	119.3	C2—C7—H7	120.1
C5—C4—H4	119.3	C6—C7—H7	120.1
C6—C5—C4	116.8 (4)	C7—C6—C5	122.0 (4)
C6—C5—C8	120.2 (3)	C7—C6—H6	119.0
C4—C5—C8	123.0 (4)	C5—C6—H6	119.0
C9—C8—C5	127.0 (4)	C15—C14—C13	119.7 (4)
C9—C8—H8	116.5	C15—C14—H14	120.1
C5—C8—H8	116.5	C13—C14—H14	120.1
C8—C9—C10	124.4 (4)	O3—C13—C14	116.9 (4)
C8—C9—H9	117.8	O3—C13—C18	122.5 (4)
C10—C9—H9	117.8	C14—C13—C18	120.6 (4)
C11—C10—C9	127.0 (4)	O3—C12—C11	114.2 (3)
C11—C10—H10	116.5	O3—C12—H12A	108.7
C9—C10—H10	116.5	C11—C12—H12A	108.7
C10—C11—C19	121.0 (3)	O3—C12—H12B	108.7
C10—C11—C12	122.8 (3)	C11—C12—H12B	108.7
C19—C11—C12	116.1 (3)	H12A—C12—H12B	107.6
O2—C19—C11	123.0 (4)	C2—O1—C1	119.0 (4)
O2—C19—C18	121.0 (3)	C13—O3—C12	116.3 (3)
C11—C19—C18	116.0 (3)		

O1—C2—C3—C4	−179.8 (3)	C18—C17—C16—C15	−0.8 (7)
C7—C2—C3—C4	−1.3 (6)	C17—C16—C15—C14	0.8 (8)
C2—C3—C4—C5	0.0 (6)	O1—C2—C7—C6	179.9 (3)
C3—C4—C5—C6	1.0 (5)	C3—C2—C7—C6	1.6 (6)
C3—C4—C5—C8	−177.1 (3)	C2—C7—C6—C5	−0.6 (6)
C6—C5—C8—C9	−176.1 (3)	C4—C5—C6—C7	−0.8 (5)
C4—C5—C8—C9	2.0 (6)	C8—C5—C6—C7	177.4 (3)
C5—C8—C9—C10	179.1 (3)	C16—C15—C14—C13	0.0 (8)
C8—C9—C10—C11	178.7 (3)	C15—C14—C13—O3	−179.1 (4)
C9—C10—C11—C19	177.7 (3)	C15—C14—C13—C18	−0.9 (7)
C9—C10—C11—C12	0.6 (6)	C17—C18—C13—O3	179.0 (3)

C10—C11—C19—O2	−10.5 (5)	C19—C18—C13—O3	3.7 (5)
C12—C11—C19—O2	166.8 (3)	C17—C18—C13—C14	1.0 (6)
C10—C11—C19—C18	168.7 (3)	C19—C18—C13—C14	−174.3 (4)
C12—C11—C19—C18	−14.0 (4)	C10—C11—C12—O3	−143.7 (3)
O2—C19—C18—C13	171.7 (3)	C19—C11—C12—O3	39.0 (5)
C11—C19—C18—C13	−7.5 (5)	C3—C2—O1—C1	−174.7 (4)
O2—C19—C18—C17	−3.5 (5)	C7—C2—O1—C1	7.0 (6)
C11—C19—C18—C17	177.3 (3)	C14—C13—O3—C12	−159.3 (4)
C13—C18—C17—C16	−0.2 (6)	C18—C13—O3—C12	22.6 (5)
C19—C18—C17—C16	175.2 (4)	C11—C12—O3—C13	−43.5 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2^{i}	0.93	2.59	3.362 (5)	141
C17—H17···O3^{ii}	0.93	2.53	3.342 (5)	146

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