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(*E*)-2-(3,5-Dimethoxybenzylidene)indan-1-one

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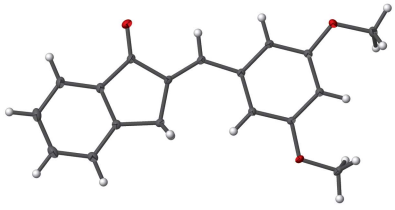
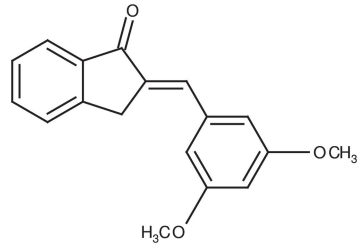
The title chalcone, $C_{18}H_{16}O_3$, was prepared by a solventless base-promoted Claisen–Schmidt condensation and, upon recrystallization from ethanol, obtained in 56% yield. The dihedral angle between the indanone ring system and the benzene ring is 2.54 (4) ° and the C atoms of the methoxy groups deviate from the benzene ring by 0.087 (1) and 0.114 (1) Å. In the crystal, π-stacking is the predominant intermolecular force, with the molecules stacking into columns running parallel to the *b* axis of the unit cell.

3D view



Chemical scheme



Structure description

The chalcone family of compounds possess an aromatic α,β-unsaturated ketone functionality and can readily be formed by base-promoted condensation–dehydrations of an aromatic aldehyde and an aromatic ketone. They are important pharmacophore scaffolds and can possess anti-inflammatory, anti-fungal, anti-cancer, and anti-malarial biological activities (Singh *et al.*, 2015, 2014; Berthelette *et al.*, 1997). Additionally, the aromatic groups can be functionalized so as to produce other biological effects. The indanone family of compounds are biologically active compounds that are involved in steroid hormone biosynthesis and arachidonic acid metabolism pathways (Berthelette *et al.*, 1997). In addition, indanone derivatives serve as scaffolds for a variety of heterocycles (Sloop *et al.*, 2002, 2012).

The combination of these two potential pharmacophores using greener and more efficient synthesis pathways en route to a series of highly functionalized indanone-based chalcones is now being studied by our research group. The solvent-free Claisen–Schmidt reaction undertaken in Fig. 1 minimizes reaction toxicity, limits waste production and enables easier product isolation in many cases.

In the title molecule (Fig. 2), the dihedral angle between the indanone ring system and the benzene ring is 2.54 (4) ° and the C‘7 and C18 atoms of the methoxy groups deviate

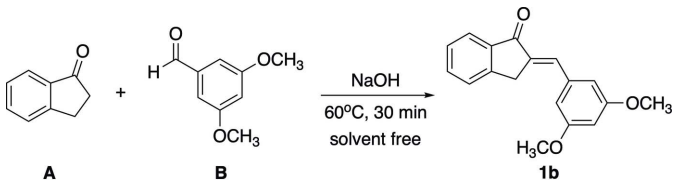


Figure 1
Green synthesis scheme for indanone-based chalcones

from the benzene ring by 0.087 (1) and 0.114 (1) Å, respectively. No unusual bond lengths or angles are noted after a routine *Mogul* geometry check (Bruno *et al.*, 2004).

The predominant supramolecular feature of this structure (Fig. 3) are slipped stacking interactions. This consists of ring-over-atom pairings between the indanone ring and the 3-position of the dimethoxyphenyl ring of a neighboring molecule and generates a relatively close contact of 2.7 Å for the methylene H atoms of the indanone ring to the adjacent molecule.

Structurally characterized 1b is consistent with known structures of similar indaneones. A search of the Cambridge Structural Database (Version 5.41, update of November 2019; Groom *et al.*, 2016) gave 35 hits with a similar core structure. A defined three-dimensional parameter search on the distance between the carbonyl O atom and the phenyl ring gave a clear indication of the stereochemistry of the double bond. The title compound adopts the more common *E* isomer – along with 33 of the other structures published – indicated by an O—C distances 4.2 to 4.5 Å. Only two examples of *Z* isomers (O—C of 3.2 to 3.4 Å) exist [POWZUX (Zhou *et al.*, 2009) and HAVLAR (Mori & Maeda, 1994)]. The latter has seven structure determinations as part of a light-driven solid-state isomerization study (Harada *et al.*, 2009).

Synthesis and crystallization

A 25 mL beaker equipped with a stir bar was charged with 3,5-dimethoxybenzaldehyde (0.50 g, 3.0 mmol) and warmed to 60°C. To the liquified aldehyde was added 1-indanone (0.40 g, 3.0 mmol) and solid NaOH (0.20 g, 3.8 mmol). The reaction mixture was stirred for 30 minutes at 60°C. The resulting reaction mixture was neutralized with 4 mL of 1 *M* HCl, the resulting residue was washed with several 1 mL aliquots of distilled water and the crude product (0.80 g, 95% yield) isolated *via* vacuum filtration. Recrystallization from 95% ethanol solution *via* slow evaporation afforded the target chalcone, (*E*)-2-(3,5-dimethoxybenzylidenyl)-1-indanone (1b) as colorless needles, (0.47 g, 56% yield). Melting range: 174–175°C. IR, ^{1}H and ^{13}C NMR spectroscopy and single-crystal X-ray analysis (see supporting information) confirmed the product identity.

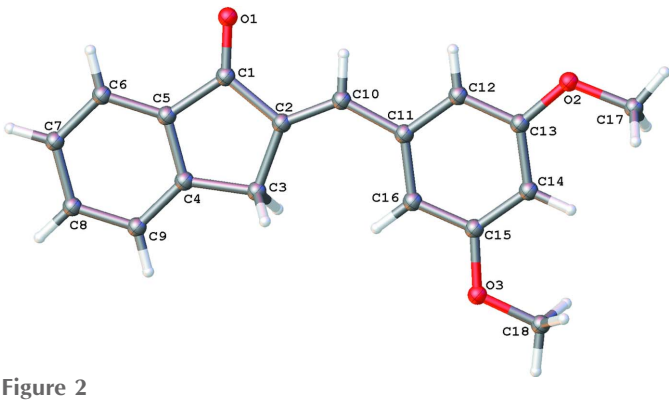


Figure 2
Displacement ellipsoid plot of 1b. Ellipsoids are drawn at the 50% probability level.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{16}O_3$
M_r	280.31
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	7.7611 (4), 7.2894 (4), 24.0331 (13)
β (°)	93.5573 (12)
V (Å^3)	1357.02 (13)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.39 × 0.12 × 0.05
Data collection	
Diffractometer	Bruker-Nonius X8 Kappa APEXII
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.95, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30838, 5231, 4087
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.772
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.123, 1.02
No. of reflections	5231
No. of parameters	192
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.61, −0.24

Computer programs: *Instrument Service*, *APEX3* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016/6* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

Refinement

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

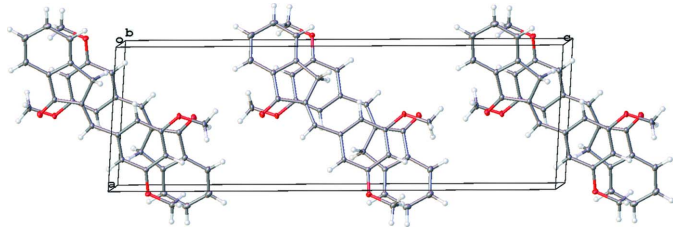


Figure 3
Packing diagram of 1b viewed along the *b* axis.

Acknowledgements

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Funding information

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full crystallographic data

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Crystal data

$C_{18}H_{16}O_3$
M_r = 280.31
Monoclinic, $P2_1/c$
a = 7.7611 (4) Å
b = 7.2894 (4) Å
c = 24.0331 (13) Å
β = 93.5573 (12)°
V = 1357.02 (13) Å^3
Z = 4
$F(000)$ = 592
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 242 reflections
θ = 3.0–33.1°
μ = 0.09 mm^{-1}
T = 100 K
Needle, colourless
0.39 × 0.12 × 0.05 mm

Data collection

Bruker-Nonius X8 Kappa APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.95, T_{max} = 0.99
30838 measured reflections
5231 independent reflections
4087 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 33.3°, θ_{min} = 2.6°
h = −11→11
k = −11→11
l = −37→37

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.123
S = 1.02
5231 reflections
192 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
$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2 + 0.2851P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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 hydrogen atoms were seen in the difference map of later refinements, but were placed at calculated positions and refined using a riding model, setting isotropic displacement parameters to 1.2 or 1.5 times that of the parent atom for ring H atoms and methyl groups respectively.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.47855 (9)	0.17474 (11)	0.33135 (3)	0.01698 (15)
O2	1.03362 (8)	0.10918 (10)	0.58143 (3)	0.01475 (14)
O3	0.48827 (9)	0.32457 (10)	0.64602 (3)	0.01472 (14)
C1	0.37431 (12)	0.23058 (12)	0.36368 (4)	0.01126 (16)
C2	0.40164 (11)	0.24728 (12)	0.42550 (4)	0.01007 (15)
C3	0.23699 (11)	0.31671 (12)	0.44870 (4)	0.01069 (16)
H3A	0.191109	0.227118	0.474981	0.013*
H3B	0.256199	0.435489	0.468075	0.013*
C4	0.11611 (11)	0.33843 (12)	0.39744 (4)	0.01048 (16)
C5	0.19615 (12)	0.29325 (12)	0.34906 (4)	0.01129 (16)
C6	0.11094 (12)	0.30860 (13)	0.29642 (4)	0.01457 (18)
H6	0.168019	0.279979	0.263676	0.017*
C7	−0.05951 (13)	0.36685 (14)	0.29328 (4)	0.01710 (19)
H7	−0.120329	0.378944	0.257935	0.021*
C8	−0.14259 (12)	0.40795 (13)	0.34182 (4)	0.01618 (18)
H8	−0.260325	0.444422	0.339062	0.019*
C9	−0.05538 (12)	0.39621 (12)	0.39403 (4)	0.01333 (17)
H9	−0.111664	0.427002	0.426756	0.016*
C10	0.55633 (12)	0.20333 (12)	0.44993 (4)	0.01049 (16)
H10	0.63798	0.160868	0.425033	0.013*
C11	0.62038 (11)	0.20978 (12)	0.50842 (4)	0.00944 (15)
C12	0.79305 (11)	0.15891 (12)	0.52012 (4)	0.01034 (15)
H12	0.86179	0.122965	0.490626	0.012*
C13	0.86425 (11)	0.16081 (12)	0.57468 (4)	0.01032 (15)
C14	0.76564 (11)	0.21385 (12)	0.61855 (4)	0.01067 (16)
H14	0.813948	0.214673	0.655848	0.013*
C15	0.59412 (11)	0.26573 (12)	0.60630 (4)	0.01004 (15)
C16	0.52038 (11)	0.26358 (12)	0.55215 (4)	0.01045 (15)
H16	0.40318	0.298291	0.544834	0.013*
C17	1.10933 (12)	0.09692 (13)	0.63704 (4)	0.01411 (17)
H17A	1.229061	0.054895	0.636091	0.021*
H17B	1.043717	0.009564	0.658384	0.021*
H17C	1.107152	0.217881	0.654776	0.021*
C18	0.54517 (13)	0.30032 (15)	0.70314 (4)	0.01596 (18)
H18A	0.57173	0.170604	0.710108	0.024*
H18B	0.453887	0.339485	0.726921	0.024*
H18C	0.648975	0.374224	0.711613	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0152 (3)	0.0258 (4)	0.0101 (3)	0.0022 (3)	0.0025 (2)	−0.0029 (3)
O2	0.0093 (3)	0.0234 (3)	0.0114 (3)	0.0048 (2)	−0.0004 (2)	−0.0017 (2)
O3	0.0125 (3)	0.0249 (4)	0.0069 (3)	0.0053 (3)	0.0018 (2)	−0.0017 (2)
C1	0.0120 (4)	0.0128 (4)	0.0089 (4)	−0.0009 (3)	0.0004 (3)	−0.0003 (3)
C2	0.0117 (4)	0.0111 (4)	0.0074 (3)	−0.0006 (3)	0.0009 (3)	−0.0002 (3)
C3	0.0112 (4)	0.0120 (4)	0.0090 (3)	0.0004 (3)	0.0015 (3)	−0.0008 (3)
C4	0.0108 (4)	0.0096 (3)	0.0110 (4)	−0.0010 (3)	0.0002 (3)	0.0002 (3)
C5	0.0119 (4)	0.0119 (4)	0.0099 (4)	−0.0007 (3)	−0.0007 (3)	0.0004 (3)
C6	0.0155 (4)	0.0170 (4)	0.0109 (4)	−0.0010 (3)	−0.0017 (3)	0.0006 (3)
C7	0.0164 (4)	0.0177 (4)	0.0165 (4)	−0.0009 (3)	−0.0053 (3)	0.0025 (3)
C8	0.0121 (4)	0.0142 (4)	0.0218 (5)	0.0007 (3)	−0.0021 (3)	0.0023 (3)
C9	0.0116 (4)	0.0126 (4)	0.0159 (4)	0.0006 (3)	0.0012 (3)	0.0014 (3)
C10	0.0117 (4)	0.0116 (4)	0.0083 (3)	0.0002 (3)	0.0012 (3)	−0.0007 (3)
C11	0.0100 (4)	0.0097 (3)	0.0086 (3)	−0.0002 (3)	0.0006 (3)	−0.0004 (3)
C12	0.0110 (4)	0.0116 (4)	0.0086 (3)	0.0012 (3)	0.0016 (3)	−0.0005 (3)
C13	0.0089 (4)	0.0112 (4)	0.0109 (4)	0.0009 (3)	0.0007 (3)	−0.0002 (3)
C14	0.0103 (4)	0.0125 (4)	0.0092 (4)	0.0011 (3)	0.0004 (3)	−0.0005 (3)
C15	0.0101 (4)	0.0121 (4)	0.0080 (4)	0.0006 (3)	0.0017 (3)	−0.0010 (3)
C16	0.0094 (4)	0.0127 (4)	0.0093 (4)	0.0011 (3)	0.0003 (3)	−0.0004 (3)
C17	0.0123 (4)	0.0165 (4)	0.0132 (4)	0.0014 (3)	−0.0025 (3)	−0.0011 (3)
C18	0.0172 (4)	0.0238 (5)	0.0071 (4)	0.0020 (3)	0.0022 (3)	−0.0007 (3)

Geometric parameters (Å, º)

O1—C1	1.2255 (11)	C8—H8	0.95
O2—C13	1.3673 (11)	C9—H9	0.95
O2—C17	1.4289 (11)	C10—C11	1.4623 (12)
O3—C15	1.3665 (11)	C10—H10	0.95
O3—C18	1.4267 (11)	C11—C16	1.4006 (12)
C1—C5	1.4777 (13)	C11—C12	1.4020 (12)
C1—C2	1.4929 (12)	C12—C13	1.3912 (12)
C2—C10	1.3421 (12)	C12—H12	0.95
C2—C3	1.5127 (13)	C13—C14	1.3952 (12)
C3—C4	1.5098 (13)	C14—C15	1.3975 (12)
C3—H3A	0.99	C14—H14	0.95
C3—H3B	0.99	C15—C16	1.3890 (12)
C4—C5	1.3913 (12)	C16—H16	0.95
C4—C9	1.3935 (12)	C17—H17A	0.98
C5—C6	1.3951 (12)	C17—H17B	0.98
C6—C7	1.3869 (14)	C17—H17C	0.98
C6—H6	0.95	C18—H18A	0.98
C7—C8	1.3999 (15)	C18—H18B	0.98
C7—H7	0.95	C18—H18C	0.98
C8—C9	1.3908 (13)		

C13—O2—C17	117.69 (7)	C2—C10—H10	114.6
C15—O3—C18	118.00 (7)	C11—C10—H10	114.6
O1—C1—C5	126.64 (8)	C16—C11—C12	119.40 (8)
O1—C1—C2	126.83 (8)	C16—C11—C10	123.99 (8)
C5—C1—C2	106.53 (7)	C12—C11—C10	116.61 (8)
C10—C2—C1	118.94 (8)	C13—C12—C11	120.34 (8)
C10—C2—C3	132.21 (8)	C13—C12—H12	119.8
C1—C2—C3	108.84 (7)	C11—C12—H12	119.8
C4—C3—C2	103.34 (7)	O2—C13—C12	115.58 (8)
C4—C3—H3A	111.1	O2—C13—C14	123.71 (8)
C2—C3—H3A	111.1	C12—C13—C14	120.70 (8)
C4—C3—H3B	111.1	C13—C14—C15	118.42 (8)
C2—C3—H3B	111.1	C13—C14—H14	120.8
H3A—C3—H3B	109.1	C15—C14—H14	120.8
C5—C4—C9	119.79 (8)	O3—C15—C16	115.30 (8)
C5—C4—C3	111.71 (8)	O3—C15—C14	122.96 (8)
C9—C4—C3	128.50 (8)	C16—C15—C14	121.73 (8)
C4—C5—C6	121.84 (8)	C15—C16—C11	119.40 (8)
C4—C5—C1	109.53 (8)	C15—C16—H16	120.3
C6—C5—C1	128.63 (8)	C11—C16—H16	120.3
C7—C6—C5	118.09 (9)	O2—C17—H17A	109.5
C7—C6—H6	121.0	O2—C17—H17B	109.5
C5—C6—H6	121.0	H17A—C17—H17B	109.5
C6—C7—C8	120.48 (9)	O2—C17—H17C	109.5
C6—C7—H7	119.8	H17A—C17—H17C	109.5
C8—C7—H7	119.8	H17B—C17—H17C	109.5
C9—C8—C7	121.00 (9)	O3—C18—H18A	109.5
C9—C8—H8	119.5	O3—C18—H18B	109.5
C7—C8—H8	119.5	H18A—C18—H18B	109.5
C8—C9—C4	118.77 (9)	O3—C18—H18C	109.5
C8—C9—H9	120.6	H18A—C18—H18C	109.5
C4—C9—H9	120.6	H18B—C18—H18C	109.5
C2—C10—C11	130.87 (8)		

O1—C1—C2—C10	2.20 (14)	C3—C4—C9—C8	179.35 (9)
C5—C1—C2—C10	−178.24 (8)	C1—C2—C10—C11	178.55 (9)
O1—C1—C2—C3	−178.27 (9)	C3—C2—C10—C11	−0.85 (17)
C5—C1—C2—C3	1.28 (9)	C2—C10—C11—C16	1.67 (15)
C10—C2—C3—C4	179.41 (10)	C2—C10—C11—C12	−178.08 (9)
C1—C2—C3—C4	−0.03 (9)	C16—C11—C12—C13	0.36 (13)
C2—C3—C4—C5	−1.36 (9)	C10—C11—C12—C13	−179.87 (8)
C2—C3—C4—C9	179.02 (9)	C17—O2—C13—C12	−175.86 (8)
C9—C4—C5—C6	1.74 (14)	C17—O2—C13—C14	4.54 (13)
C3—C4—C5—C6	−177.92 (8)	C11—C12—C13—O2	−179.86 (8)
C9—C4—C5—C1	−178.10 (8)	C11—C12—C13—C14	−0.25 (13)
C3—C4—C5—C1	2.24 (10)	O2—C13—C14—C15	179.29 (8)
O1—C1—C5—C4	177.40 (9)	C12—C13—C14—C15	−0.29 (13)
C2—C1—C5—C4	−2.16 (10)	C18—O3—C15—C16	169.35 (8)

O1—C1—C5—C6	−2.42 (16)	C18—O3—C15—C14	−11.69 (13)
C2—C1—C5—C6	178.02 (9)	C13—C14—C15—O3	−178.18 (8)
C4—C5—C6—C7	−1.47 (14)	C13—C14—C15—C16	0.72 (13)
C1—C5—C6—C7	178.33 (9)	O3—C15—C16—C11	178.37 (8)
C5—C6—C7—C8	−0.26 (14)	C14—C15—C16—C11	−0.61 (13)
C6—C7—C8—C9	1.73 (15)	C12—C11—C16—C15	0.06 (13)
C7—C8—C9—C4	−1.46 (14)	C10—C11—C16—C15	−179.69 (8)
C5—C4—C9—C8	−0.24 (13)		