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A cocrystal of 1-(4-methoxyphenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one and (*E*)-3-(3-chloro-4,5-dimethoxyphenyl)-1-(4-methoxyphenyl)-2-propen-1-one (0.92/0.08)

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.001 Å
Disorder in main residue
R factor = 0.043
wR factor = 0.128
Data-to-parameter ratio = 30.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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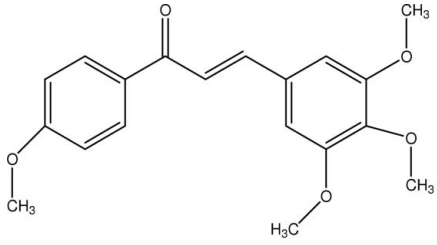
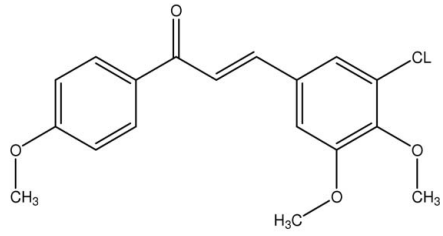
In the title compound, $0.92C_{19}H_{20}O_5{\cdot}0.08C_{18}H_{17}ClO_4$, C—H···O and C—H···π interactions link the molecules into a three-dimensional network.

Comment

Chalcone derivatives offer a wide range of biological activity, including antimitotic potency, and are amenable to chemical modification and/or functionalization in order to improve their characteristics. A series of dimethoxy and trimethoxychalcone derivatives, with various patterns of fluorination, are important compounds for their influence on nitric oxide production in lipopolysaccharide-stimulated murine RAW 264.7 cells (Rojas *et al.*, 2002). They are also potential novel cancer chemopreventive agents (Bertl *et al.*, 2004).



(I)



(II)

Among the many organic compounds reported for their nonlinear optical (NLO) properties, chalcone derivatives are noticeable for their excellent blue light transmittance and good crystallizability. It is observed that substitution of the methoxy group on either side of the benzene rings greatly influences the non-centrosymmetric crystal packing (Fichou *et al.*, 1988; Kitaoka *et al.*, 1990; Zhao *et al.*, 2000). We report here the synthesis and structure of the title compound, a cocrystal of (I) and (II). The title compound is found to crystallize in a centrosymmetric space group and therefore has no second-order NLO properties.

The bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and comparable to those reported for similar structures (Jeyabharathi *et al.*, 2002; Sathiya Moorthi, Chinnakali, Nanjundan, Radhika *et al.*, 2005; Sathiya Moorthi, Chinnakali, Nanjundan, Santhi & Fun, 2005; Sathiya Moorthi, Chinnakali, Nanjundan, Selvam *et al.*, 2005; Sathiya Moorthi, Chinnakali, Nanjundan, Unnithan *et al.*, 2005; Ravishankar *et al.*, 2005).

The short H1···H8 (2.19 Å) contact causes the bond angles C1—C6—C7 [122.10 (7)°] and C6—C7—C8 [117.12 (7)°] to deviate significantly from 120°. In addition, the short H8···H15 (2.11 Å) contact produces a slight widening of the

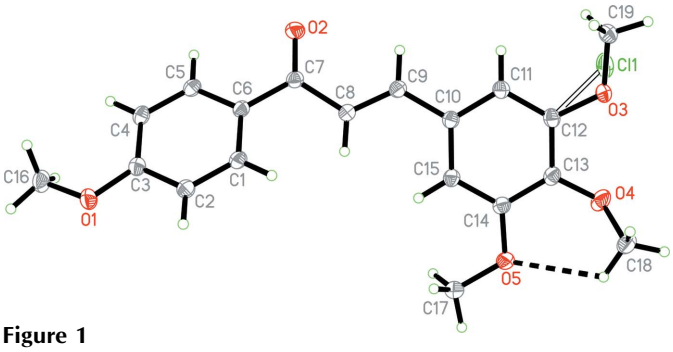


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. The dashed line indicates a hydrogen bond. The minor disorder component is indicated by a hollow bond.

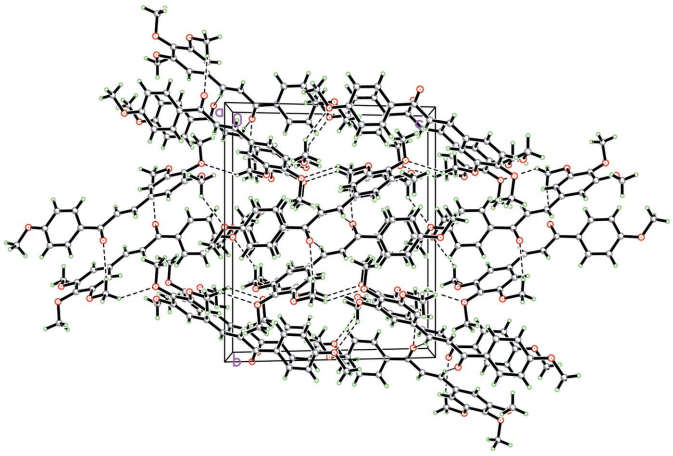


Figure 2
The crystal packing of the title compound, viewed down the *a* axis. Hydrogen bonds are shown as dashed lines. Cl atoms have been omitted for clarity.

C9—C10—C15 angle to 121.29 (7)°. These result in the molecule being twisted about the C6—C7 bond for (I) and (II), with the torsion angles C5—C6–C7—C8 = −157.78 (7)°, C6—C7—C8—C9 = −167.79 (8)°, C7—C8—C9—C10 = −178.01 (7)° and C8—C9—C10—C11 = −177.19 (8)°.

The enone unit (O2/C7–C9) and the two benzene rings C1–C6 and C10–C15 of the chalcone are planar, with maximum deviations of 0.054 (1), 0.017 (1) and 0.016 (1) Å for atoms C7, C5 and C15, respectively. The enone unit makes dihedral angles of 21.7 (1) and 14.4 (1)° with the C1–C6 and C10–C15 benzene rings, respectively. The dihedral angle between the two benzene rings is 35.72 (4)°.

Three methoxy groups in (I) and (II) are almost coplanar with the attached rings, with torsion angles C16—O1—C3—C4 of 6.0 (1)°, C19—O3—C12—C11 of 11.0 (1)° and C17—O5—C14—C15 of 3.4 (1)°. The fourth methoxy group at C13 is twisted away from the attached ring, with a torsion angle C18—O4—C13—C14 of −64.4 (1)°.

In the crystal structure, all intra- and intermolecular hydrogen bonds involve O atoms. While atom O5 is involved in the C18—H18*B*···O5 intramolecular interaction, atoms O1, O2 and O4 are involved in intermolecular C—H···O interactions (Table 1). These interactions form a three-dimensional network. The crystal packing is further stabilized by a weak intermolecular C—H···π interaction involving the C1–C6 ring (centroid *Cg*1).

Experimental

The title compound was obtained by the condensation of 4-methoxyacetophenone (0.01 mol) and 3,4,5-trimethoxybenzaldehyde (0.01 mol) in ethanol (60 ml) in the presence of NaOH (5 ml, 30%). After stirring for 2 h, the contents of the flask were poured into ice-cold water and allowed to stand for 24 h. The resulting crude solid compound was collected by filtration, dried and recrystallized twice from acetone. Crystals suitable for X-ray diffraction study were grown by slow evaporation of an acetone solution over a period of 10 d. Our intention was to the synthesize (I), but the presence of chlorine in 3,4,5-trimethoxybenzaldehyde (one of the starting materials) in the form of 3-chloro-4,5-dimethoxybenzaldehyde resulted also in the formation of (II). The presence of the Cl atom was confirmed by chemical analysis and NMR data. Analysis found: C 68.84, H 6.30, O 24.09%; calculated for $C_{18.92}H_{19.77}Cl_{0.08}O_{4.92}$: C 69.13, H 6.06, O 23.94%.

Crystal data

$0.92C_{19}H_{20}O_5{\cdot}0.08C_{18}H_{17}ClO_4$
M_r = 328.69
Monoclinic, $P2_1/n$
a = 7.4777 (2) Å
b = 16.1092 (4) Å
c = 13.7416 (3) Å
β = 104.043 (1)°
V = 1605.84 (7) Å^3
Z = 4
D_x = 1.360 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6138 reflections
θ = 2.5–35.0°
μ = 0.11 mm^{-1}
T = 120.0 (1) K
Block, yellow
0.65 × 0.45 × 0.40 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.829, T_{max} = 0.958
32466 measured reflections
7035 independent reflections
5757 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 35.0°
$h = -11 \rightarrow 11$
$k = -25 \rightarrow 26$
$l = -21 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.128
S = 1.05
7035 reflections
231 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 0.3474P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···O2^{i}	0.93	2.44	3.3472 (11)	164
C17—H17*B*···O2^{ii}	0.96	2.50	3.4408 (12)	166
C17—H17*C*···O4^{iii}	0.96	2.54	3.2349 (12)	129
C18—H18*B*···O5	0.96	2.39	2.9411 (12)	116
C19—H19*C*···O1^{iv}	0.96	2.49	3.3110 (13)	143
C16—H16*C*···*Cg*1^{v}	0.96	2.76	3.5927 (11)	145

Symmetry codes: (i) $-x+2, -y, -z$; (ii) $-x+1, -y, -z$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $x+1, y, z+1$; (v) $-x+1, -y, -z-1$. Note: *Cg*1 is the centroid of ring C1–C6.

H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.96 Å. The U_{iso}(H) values were constrained to be

$1.5U_{eq}$(H) of the carrier atom for methyl H atoms and $1.2U_{eq}$(H) for the remaining H atoms. The ratio of (I) and (II) in the cocrystal was obtained by refinement as 0.924 (3):0.076 (3).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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