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The 0.893/0.104/0.003 cocrystal of 1-(4-methylphenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one, 3-(3-chloro-4,5-dimethoxyphenyl)-1-(4-methylphenyl)-prop-2-en-1-one and 3-(3,5-dichloro-4-methoxyphenyl)-1-(4-methylphenyl)prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
Disorder in main residue
R factor = 0.046
wR factor = 0.148
Data-to-parameter ratio = 30.6

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

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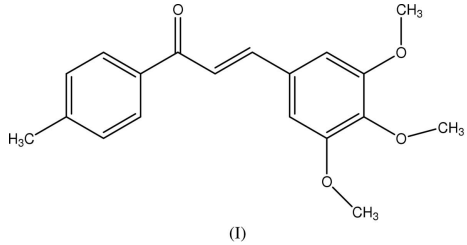
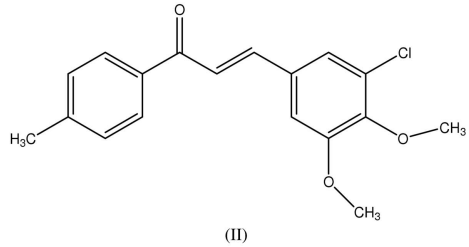
In the title cocrystal, $0.893C_{19}H_{20}O_4{\cdot}0.104C_{18}H_{17}ClO_3{\cdot}0.003C_{17}H_{14}Cl_2O$, the crystal packing is stabilized by weak intermolecular C—H···O and C—H···π interactions.

Comment

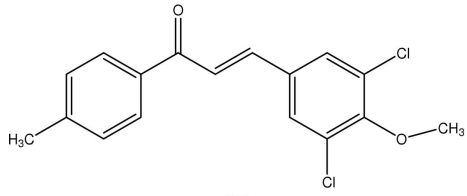
The interest in chalcone derivatives in several disciplines stems from their biological (Boeck *et al.*, 2005) and pharmacological activities, such as antiprotozoal (Nielsen *et al.*, 1998; Li *et al.*, 1995; Liu *et al.*, 2001), anti-inflammatory (Hsieh *et al.*, 1998) and nitric oxide inhibitory activities (Rojas *et al.*, 2002). Furthermore, it has been noted that derivatives of chalcones exhibit extremely high and fast non-linearity (Fichou *et al.*, 1988; Uchida *et al.*, 1998; Goto *et al.*, 1991; Patil *et al.*, 2006*a*,*b*). In view of these features associated with chalcones, we and others have undertaken a number of theoretical and structural studies of such compounds (Patil *et al.*, 2006*a*,*b*; Radha Krishna *et al.*, 2005; Sathiya Moorthi *et al.*, 2005; Uchida *et al.*, 1995), and we report here the structure of the title cocrystal, (I) (Fig. 1).



(I)



(II)



(III)

Crystals of the title cocrystal do not exhibit second-order non-linear optical properties as they crystallize in a centrosymmetric space group. Our intention was to synthesize (I),

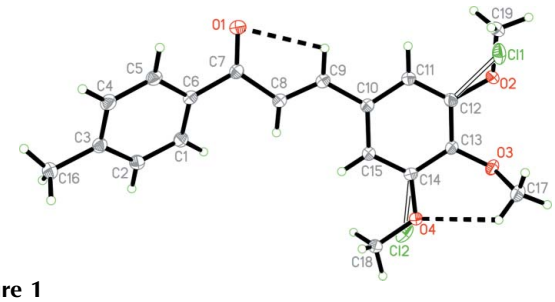


Figure 1
The molecular structure of the title cocrystal, showing 50% probability displacement ellipsoids and the atomic numbering. The dashed line indicates a hydrogen bond. The Cl atoms and their open bonds are minor disorder components.

but the presence of chlorine in 3,4,5-trimethoxybenzaldehyde (one of the starting materials) in the form of 3-chloro-4,5-dimethoxybenzaldehyde led to the formation of (II) and (III) as well. Bond lengths and angles in the title cocrystal show normal values (Allen *et al.*, 1987) and are comparable with related structures (Ng *et al.*, 2006; Patil *et al.*, 2006*a*,*b*). The enone group and the two benzene rings of the chalcone are both planar, the largest deviations being 0.045 (1) Å from O1/C7–C9, 0.012 (1) Å from C1–C6, and 0.004 (1) Å from C10–C15 for atoms C8, C1 and C15, respectively.

The short H1*A*···H8*A* (2.25 Å) contact causes the bond angles C1—C6—C7 [122.04 (8)°] and C6—C7—C8 [117.37 (8)°] to deviate significantly from 120°. Similarly, the short H8*A*···H15*A* (2.20 Å) contact produces a slight widening of the C9—C10—C15 angle to 121.99 (8)°. These contacts lead to the molecule being twisted about the C6—C7 bond. The least-squares plane through the enone group makes dihedral angles of 36.00 (3) and 4.91 (3)° with the C1–C6 and C10–C15 benzene rings, respectively. The dihedral angle between benzene rings C1–C6 and C10–C15 is 32.26 (3)°.

The methoxy groups at C12 and C14 are almost coplanar with the attached rings, with torsion angles C19—O2—C12—C11 = 14.91 (1)° and C18—O4—C14—C15 = 0.1 (2)°, while the methoxy group at C17 is twisted away from the attached ring [C17–O3—C13—C14 = −81.19 (1)°].

In the crystal structure, atom O1 is involved in both intra- and intermolecular hydrogen bonding (Table 1). The intramolecular C9—H9*A*···O1 and C17—H17*B*···O4 interactions generate *S*(5) and *S*(6) ring motifs, respectively (Bernstein *et al.*, 1995). The closest intermolecular H···Cl contact of 2.803 (10) Å for H16*B*···Cl2 [C16···Cl2 = 3.679 (10) Å, C16—H16*B* = 0.96 Å and C16—H16*B*···Cl2 = 152°] probably falls outside the range for a significant C—H···Cl hydrogen bond, but there is a weak intermolecular C16—H16*A*···O1 interaction (Table 1) between molecules arranged along [00$\bar{1}$] (Fig. 2). In addition, the crystal packing is further stabilized by weak intermolecular C—H···π interactions involving the C1–C6 and C10–C15 rings.

Experimental

Compound (I) was synthesized by the Claisen–Schmidt condensation of 3,4,5-trimethoxybenzaldehyde (0.01 mol) with 4-methylacetophenone (0.01 mol) in ethanol (60 ml) in the presence of NaOH (2 ml, 30%). After stirring for 3 h, the contents of the flask were poured into ice-cold water (250 ml) and left to stand for 24 h. The resulting crude solid was collected by filtration, dried and purified by repeated recrystallization from acetone. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of an acetone solution at room temperature over a period of 7 d. The presence of Cl in the minor cocrystal components was consistent with the chemical analysis. Analysis found: C 72.81, H 06.02, O 19.97%; calculated for $C_{18.89}H_{19.66}Cl_{0.11}O_{3.89}$: C 72.52, H 06.34, O 19.89%.

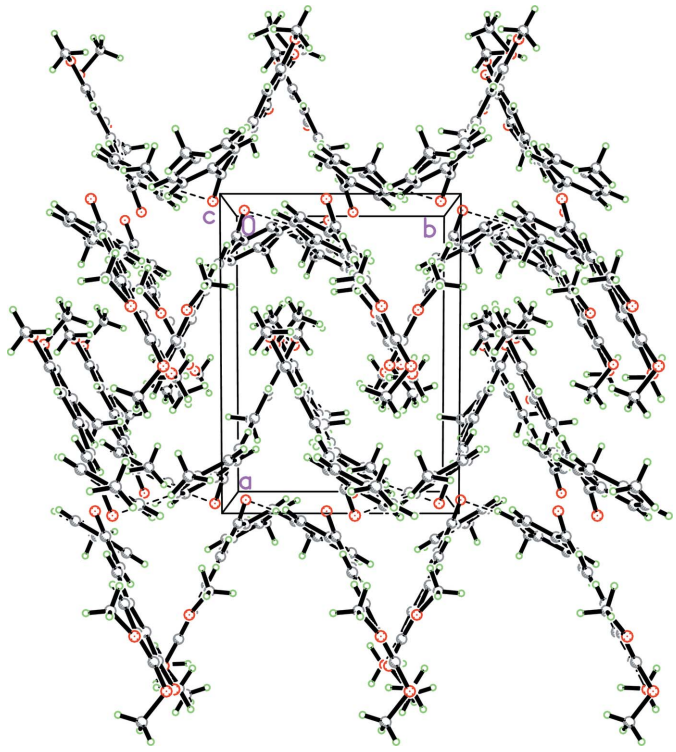


Figure 2
The crystal structure of the major component of the title cocrystal, viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

Crystal data

$0.893C_{19}H_{20}O_4{\cdot}0.104C_{18}H_{17}ClO_3{\cdot}0.003C_{17}H_{14}Cl_2O$
M_r = 312.84
Monoclinic, $P2_1/c$
a = 12.0743 (2) Å
b = 8.9473 (2) Å
c = 15.0565 (3) Å
β = 97.683 (1)°
V = 1611.99 (6) Å^3
Z = 4
D_x = 1.289 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100.0 (1) K
Block, yellow
0.58 × 0.55 × 0.39 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.872, T_{max} = 0.959
24407 measured reflections
7095 independent reflections
5116 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 35.0°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0757P)^2 + 0.394P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.148$	$\Delta\rho_{max} = 0.56$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.25$ e Å^{-3}
7095 reflections	
232 parameters	
H-atom parameters constrained	

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

*Cg*1 and *Cg*2 are the centroids of the C1–C6 and C10–C15 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9*A*···O1	0.93	2.50	2.833 (1)	101
C11—H11*A*···O1^{i}	0.93	2.60	3.424 (1)	148
C16—H16*A*···O1^{ii}	0.96	2.43	3.299 (1)	151
C17—H17*B*···O4	0.96	2.60	3.103 (2)	113
C2—H2*A*···*Cg*2^{iii}	0.93	2.99	3.716 (1)	137
C16—H16*C*···*Cg*1^{iv}	0.96	2.86	3.465 (1)	122
C17—H17*A*···*Cg*1^{v}	0.96	2.97	3.583 (1)	123

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $-x, -y+1, -z-1$; (v) $-x+1, -y+1, -z$.

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 $1.5U_{eq}$(carrier atom) for methyl H atoms and $1.2U_{eq}$(carrier atom) for the remaining H atoms. The ratio of the three disorder components was obtained by refinement as 0.893 (4):0.104 (4):0.003 (4).

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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