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A series of substituted (2*E*)-3-(2-fluoro-4-phenoxyphenyl)-1-phenylprop-2-en-1-ones

Deepak Chopra,[a]* T. P. Mohan,[b] B. Vishalakshi[b] and T. N. Guru Row[a]

[a]Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and [b]Department of Chemistry, Mangalore University, Bangalore 574 199, Karnataka, India
Correspondence e-mail: deepak@sscu.iisc.ernet.in

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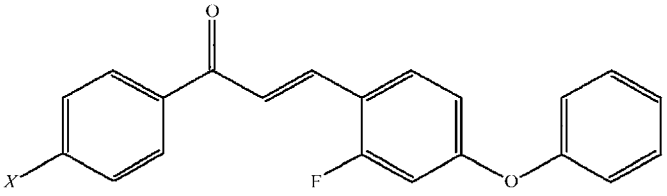
In the molecular structures of a series of substituted chalcones, namely (2*E*)-3-(2-fluoro-4-phenoxyphenyl)-1-phenylprop-2-en-1-one, $C_{21}H_{15}FO_2$, (I), (2*E*)-3-(2-fluoro-4-phenoxyphenyl)-1-(4-fluorophenyl)prop-2-en-1-one, $C_{21}H_{14}F_2O_2$, (II), (2*E*)-1-(4-chlorophenyl)-3-(2-fluoro-4-phenoxyphenyl)prop-2-en-1-one, $C_{21}H_{14}ClFO_2$, (III), (2*E*)-3-(2-fluoro-4-phenoxyphenyl)-1-(4-methylphenyl)prop-2-en-1-one, $C_{22}H_{17}FO_2$, (IV), and (2*E*)-3-(2-fluoro-4-phenoxyphenyl)-1-(4-methoxyphenyl)prop-2-en-1-one, $C_{22}H_{17}FO_3$, (V), the configuration of the keto group with respect to the olefinic double bond is *s-cis*. The molecules pack utilizing weak C—H$\cdots$O and C—H$\cdots\pi$ intermolecular contacts. Identical packing motifs involving C—H$\cdots$O interactions, forming both chains and dimers, along with C—H$\cdots\pi$ dimers and π–π aromatic interactions are observed in the fluoro, chloro and methyl derivatives.

Comment

Chalcones are an important class of organic compounds, belonging to the biogenetic precursors of the flavonoids in higher plants, and these find potential applications in widespread biological activities, *viz*. antibacterial, antifungal and anti-inflammatory drugs (Ahluwalia *et al.*, 1986; Achenbach *et al.*, 1988; Bhat *et al.*, 1972; Masaoud *et al.* 1995; Mathew *et al.*, 1984; Oganesyan *et al.*, 1986; Sinz *et al.*, 1999; Thuy *et al.*, 1998). The essential feature of chalcone derivatives is the carbonyl functional group, which has a pivotal role in the observed antibacterial activity of such compounds. In addition, antimicrobial, antiviral, antitumoral, antimutagenic, antimitotic, anti-inflammatory, anti-ulcerative and hepatoprotective activities and applications of therapeutic effects have also been reported (Arty *et al.*, 2000; Batt *et al.*, 1993; Hu *et al.*, 1994; Iwata *et al.*, 1995; Lorimer & Perry, 1994; Mahmood *et al.*, 1997; Markham *et al.*, 1987; Min *et al.*, 1996; Pappano *et al.*, 1985, 1990, 1994; Sogawa *et al.*, 1993, 1994; Williams *et al.*, 1995; Makita *et al.*, 1996). Since some chalcones are found to inhibit various enzymes, involving the reaction of reactive oxygen species (5-lipoxygenase, 12-lipoxygenase, cyclooxygenase, *etc*.), the majority of their pharmacological properties are supposed to be related to their antioxidative effect (Forejtnikova *et al.*, 2005; Sogawa *et al.*, 1994). In the literature, there are reports on structure–activity correlations carried out on such classes of compounds (Debattista *et al.*, 1986; Dhar, 1961). These compounds have also been used as starting (precursor) materials in many synthetic reactions (Awad *et al.*, 1960; Carrie & Rochard, 1963; Coudert *et al.*, 1988; Nel *et al.*, 1998). Substituted chalcones have been synthesized and used as fluorescent probes (Jiang *et al.*, 1994). In particular, chalcone derivatives with typical *D-p-A* mode have been reported to possess intense fluorescence properties. For example, dimethylaminochalcone has been reported to be a potential chemosensor (Di Cesare & Lakowicz, 2002). These have also been found to exhibit potential nonlinear optical properties (Wang & Wu, 1994; Fichou *et al.*, 1988). Differently substituted chalcones have been investigated in detail in the solid state to understand the structural features associated with these compounds (Lokaj *et al.*, 2001; Low *et al.*, 2002; Sathiya Moorthi, Chinnakali, Nanjundan, Radhika *et al.*, 2005, and references therein; Moorthi *et al.*, 2005; Sathiya Moorthi, Chinnakali, Nanjundan, Selvam *et al.*, 2005; Ravishankar *et al.*, 2005; Schmalle *et al.*, 1990; Wang *et al.*, 2004). The phenomenon of polymorphism has also been explored in such compounds, a very important case being bavachalcone (Ravikumar *et al.*, 2005), a bioactive compound extracted from the seed of *Psoralea corylifolia*, which has been widely applied for the cure of bleeding, vitiligo and psoriasis, leucoderma, bone fracture and osteoporosis (Kondo *et al.*, 1990; Miura *et al.*, 1996; Yin *et al.*, 2004).



(I) X=H
(II) X=F
(III) X=Cl
(IV) X=Me
(V) X=OMe

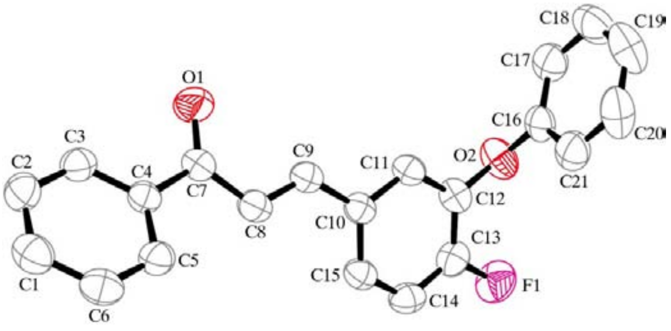


Figure 1
The molecular structure of (I), drawn with 50% probability displacement ellipsoids and showing the atom-labeling scheme.

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In order to understand the geometrical features and the underlying intermolecular interactions which hold the assembly of molecules in the crystalline lattice, we have studied a series of substituted chalcones, (I)–(V), and analyzed the conformational and packing features. Weak intermolecular C—H$\cdots$O, C—H$\cdots\pi$ and π–π interactions generate different packing motifs in the crystal structure.

Figs. 1–5 depict the structures of all the molecules. Compound (I) (Fig. 1) crystallizes in the monoclinic space group $P2_1/n$ with $Z = 4$. Compounds (II)–(V) (Figs. 2–5) crystallize in the triclinic centrosymmetric space group $P\overline{1}$. The H atom of the central propenone group is *trans* with respect to the C8═C9 double bond. The dihedral angles between the C10–C15 and C1–C6 aromatic rings (*A* and *B*, respectively), and the dihedral angles between these rings and the O1═C7—C8═C9 group, are given in Table 11. An analysis of the weighted least-squares plane through the central C4/C7/O1/C8/C9/C10 chain shows that atoms C8 and C9 are displaced by the distances given in Table 12.

The C8═C9 double-bond lengths in all of the compounds are in agreement with the expected values reported in the literature (Allen *et al.*, 1987; Allen, 2002; Sathiya Moorthi, Chinnakali, Nanjundan, Radhika *et al.*, 2005, and references therein; Moorthi *et al.*, 2005; Sathiya Moorthi, Chinnakali, Nanjundan, Selvam *et al.*, 2005; Tables 1, 3, 5, 7 and 9). However, the C8—C9—C10 angle deviates from an ideal value of 120° as a result of the steric interaction between atoms H15 on ring *A* and H8 attached to the C8═C9 double bond (Sathiya Moorthi, Chinnakali, Nanjundan, Radhika *et*

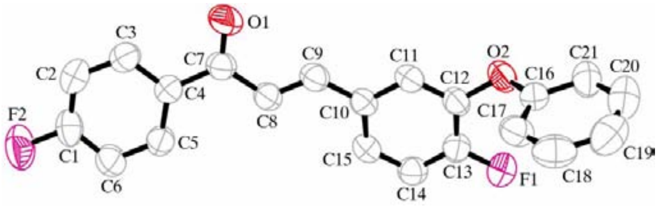

Figure 2
The molecular structure of (II), drawn with 50% probability displacement ellipsoids and showing the atom-labeling scheme.

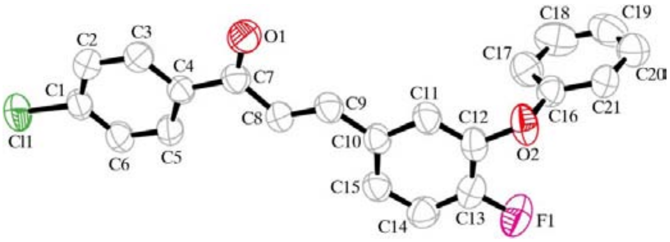

Figure 3
The molecular structure of (III), drawn with 50% probability displacement ellipsoids and showing the atom-labeling scheme.

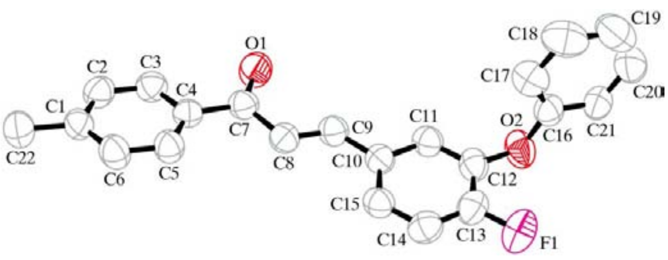

Figure 4
The molecular structure of (IV), drawn with 50% probability displacement ellipsoids and showing the atom-labeling scheme.

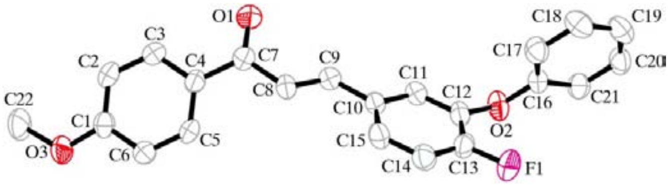

Figure 5
The molecular structure of (V), drawn with 50% probability displacement ellipsoids and showing the atom-labeling scheme.

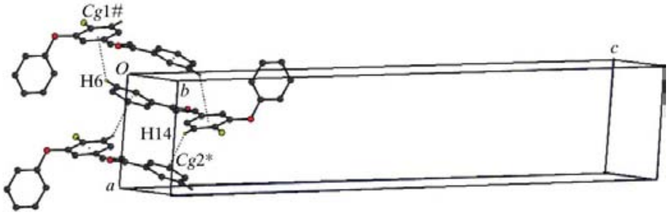

Figure 6
A partial packing diagram for (I), depicting C—H$\cdots\pi$ dimers. Molecules labeled with a hash (#) or asterisk (*) have the symmetry codes ($-x$, $-y + 1$, $-z$) and ($-x + 1$, $-y + 1$, $-z$), respectively. *Cg*1 and *Cg*2 are the centers of gravity of the C10–C15 and C1–C6 aromatic rings, respectively. The dotted lines indicate intermolecular interactions.

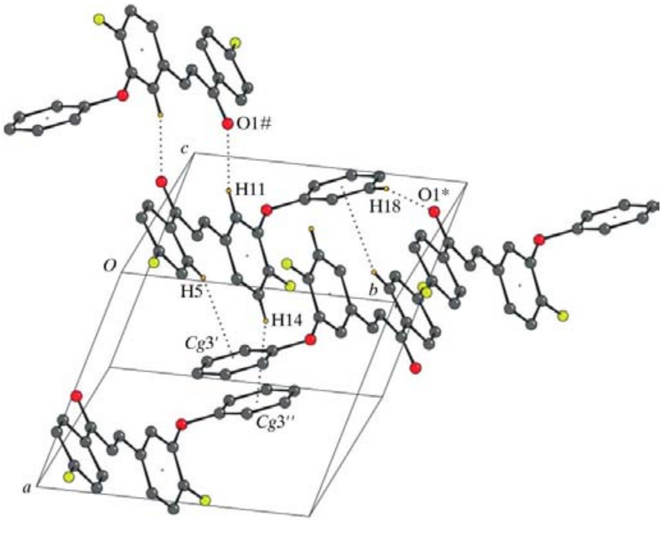

Figure 7
A partial packing diagram for (II), depicting C—H$\cdots$O and C—H$\cdots\pi$ interactions forming dimers and chains. Molecules labeled with a prime (′), double prime (″), hash (#) or asterisk (*) have the symmetry codes ($-x + 1$, $-y + 1$, $-z$), ($x - 1$, y, z), ($-x$, $-y$, $-z + 2$) and (x, $y + 1$, z), respectively. The dotted lines indicate intermolecular interactions. *Cg*3 is the center of gravity of the C16–C21 aromatic ring.

al., 2005, and references therein; Moorthi *et al.*, 2005; Sathiya Moorthi, Chinnakali, Nanjundan, Selvam *et al.*, 2005). This is a common feature observed in all the compounds.

In (I), the intermolecular interactions that characterize the packing of molecules are C—H$\cdots\pi$ interactions involving atoms H6 and H14, forming alternate dimers in the crystalline lattice with rings *A* (whose center of gravity is denoted as *Cg*1) and *B* (whose center of gravity is denoted as *Cg*2), respectively (Fig. 6 and Table 2).

On replacement of the H atom at the *para* position (C1) by an F atom in (II), the electron-donating mesomeric effect of the lone pairs on the F atom on ring *A* leads to extended conjugation, the dihedral angle being 4.29 (8)°. The packing of molecules (Fig. 7 and Table 4) in the crystal structure is *via* two different types of C—H$\cdots$O interaction, one forming chains along the crystallographic *b* axis (involving atom H18) [graph-set symbol *C*(12)] and the other forming dimers (involving atom H11) [graph-set symbol $R_2^2(14)$]. C—H$\cdots\pi$ interactions, involving atom H14 and the C16–C21 ring (*C*), form molecular chains along the *a* axis, leading to the formation of a sheet-like structure.

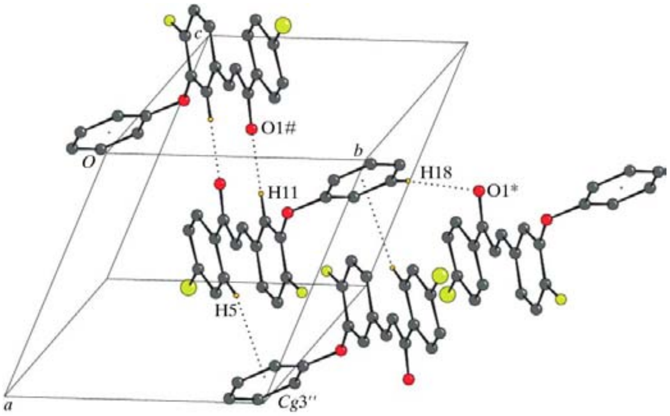

Figure 8
A partial packing diagram for (III), depicting C—H$\cdots$O and C—H$\cdots\pi$ interactions. Molecules labeled with a hash (#), asterisk (*) or double prime (″) have the symmetry codes ($-x$, $-y$, $-z + 2$), (x, $y + 1$, z) and ($-x + 1$, $-y + 1$, $-z$), respectively. The dotted lines indicate intermolecular interactions. *Cg*3 is the center of gravity of the C16–C21 aromatic ring.

On replacement of the F atom by a Cl atom in (III), the crystal packing is unchanged. The same H atoms (H11 and H18) are involved in the formation of identical packing motifs (Fig. 8 and Table 6). Such C—H$\cdots$O interactions generate tetrameric motifs, consisting of $R_2^4(32)$ and $R_2^4(22)$ units. Furthermore, C—H$\cdots\pi$ dimers, involving atom H5 and ring *C*, link consecutive tetrameric units together in the crystal structure.

Similar packing characteristics are observed in the case of the methyl derivative, (IV). Identical C—H$\cdots$O and C—H$\cdots\pi$ intermolecular contacts steer the packing of molecules in the crystal structure (Fig. 9 and Table 8). In addition to these interactions, the crystal structure is further stabilized by aromatic π–π stacking interactions involving rings *A* and *B*; the distances are in the range 3.862 (3)–3.880 (3) Å in the fluoro, chloro and methyl derivatives, respectively.

In the case of (V), the presence of a methoxy group generates different packing motifs, wherein the crystal structure is stabilized by two different C—H$\cdots$O dimers involving atoms H18 and H3 with atoms O3 and O1 [graph-set symbols $R_2^2(32)$ and $R_2^2(12)$], forming dimeric motifs. Furthermore, C—H$\cdots\pi$ dimers, involving atom H21 and ring *B*, reinforce the existing C—H$\cdots$O dimers, providing additional stability (Fig. 10 and Table 10).

It is of interest to note that the packing of molecules in all the crystal structures is dictated by weak C—H$\cdots$O and C—H$\cdots\pi$ intermolecular interactions.

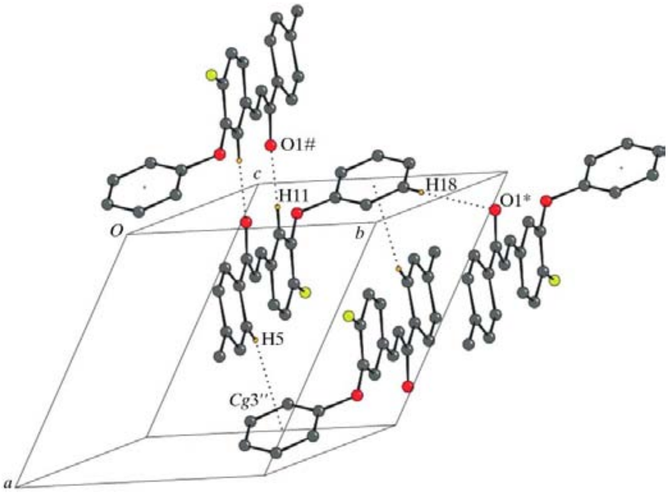

Figure 9
C—H$\cdots$O and C—H$\cdots\pi$ intermolecular interactions highlighting the packing features in (IV). Molecules labeled with a hash (#), asterisk (*) or double prime (″) have the symmetry codes ($-x$, $-y$, $-z + 2$), (x, $y + 1$, z) and ($-x + 1$, $-y + 1$, $-z$), respectively, with *Cg*3 denoting the center of gravity of the C16–C21 aromatic ring.

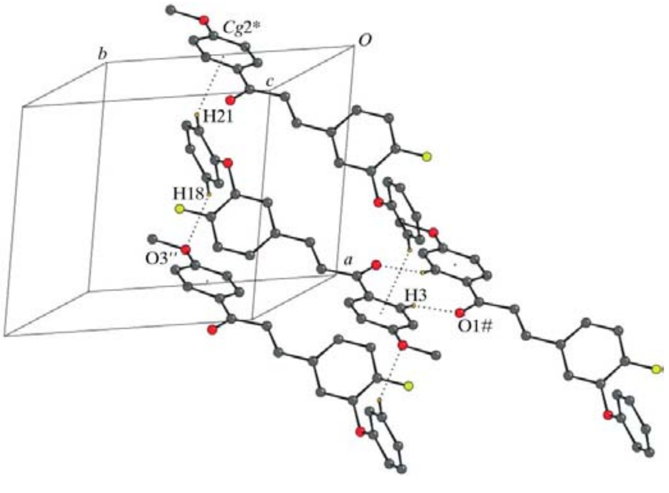

Figure 10
A partial packing diagram for (V), depicting C—H$\cdots$O and C—H$\cdots\pi$ dimers. Molecules labeled with a hash (#), double prime (″) or asterisk (*) have the symmetry codes ($-x + 2$, $-y - 1$, $-z + 1$), ($-x + 2$, $-y$, $-z + 1$) and ($-x + 1$, $-y$, $-z + 1$), respectively. The dotted lines indicate intermolecular interactions. *Cg*2 is the center of gravity of the C1–C6 aromatic ring.

Experimental

To a thoroughly stirred solution of substituted acetophenone (0.01 mol) and 4-fluoro-3-phenoxybenzaldehyde (0.01 mol) in methanol (25 ml), 40% aqueous NaOH (100 ml) was added at room temperature; the mixture was heated for 10 min and left overnight. It was then poured into ice–water and the precipitated product was filtered off. The crude product was recrystallized from hexane. The synthesized product was characterized by melting point measurements, and IR, NMR and mass spectroscopy (Mohan, 2006). The product was taken in 5.0 ml beakers and a series of crystallization experiments were performed in solvents such as acetone, ethyl acetate/hexane, dichloromethane and dichloromethane/hexane. In all cases, the crystals on which the final data were collected were grown from acetone.

Compound (**I**)

Crystal data

$C_{21}H_{15}FO_2$
$M_r = 318.33$
Monoclinic, $P2_1/n$
$a = 7.2869$ (17) Å
$b = 5.9407$ (14) Å
$c = 36.805$ (8) Å
$\beta = 93.724$ (4)°
$V = 1589.9$ (6) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 290$ (2) K
$0.56 \times 0.40 \times 0.24$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.929$, $T_{\max} = 0.978$
11668 measured reflections
3220 independent reflections
2748 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.135$
$S = 1.21$
3220 reflections
277 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I).

C10—C9	1.460 (2)	O2—C16	1.392 (2)
C9—C8	1.327 (3)	C8—C7	1.475 (3)
O1—C7	1.224 (2)	C4—C7	1.489 (2)
O2—C12	1.384 (2)		
C8—C9—C10	128.00 (18)	C5—C4—C7	122.76 (16)
C11—C10—C9—C8	−176.10 (18)	C3—C4—C7—C8	−157.94 (16)
C10—C9—C8—C7	−174.49 (17)	C12—O2—C16—C17	−135.57 (19)
C9—C8—C7—C4	−166.65 (16)	C16—O2—C12—C11	60.7 (2)

Table 2
Hydrogen-bond geometry (Å, °) for (I).

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C6—H6$\cdots$*Cg*1$^{\rm i}$	0.97 (3)	3.02 (2)	3.685 (2)	127 (2)
C14—H14$\cdots$*Cg*2$^{\rm ii}$	0.95 (2)	2.77 (2)	3.429 (2)	128 (2)

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

Compound (**II**)

Crystal data

$C_{21}H_{14}F_2O_2$
$M_r = 336.32$
Triclinic, $P\overline{1}$
$a = 8.164$ (5) Å
$b = 10.036$ (6) Å
$c = 11.136$ (7) Å
$\alpha = 97.362$ (10)°
$\beta = 101.898$ (9)°
$\gamma = 108.731$ (9)°
$V = 826.8$ (9) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 290$ (2) K
$0.23 \times 0.20 \times 0.06$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.944$, $T_{\max} = 0.994$
8638 measured reflections
3291 independent reflections
2205 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.118$
$S = 1.02$
3291 reflections
282 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.12$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (II).

C10—C9	1.463 (2)	C4—C7	1.487 (2)
O2—C16	1.385 (2)	C8—C7	1.478 (2)
O2—C12	1.3895 (19)	C7—O1	1.219 (2)
C9—C8	1.321 (2)		
C8—C9—C10	128.49 (16)	C5—C4—C7	123.47 (14)
C11—C10—C9—C8	−167.27 (17)	C9—C8—C7—C4	−175.44 (15)
C16—O2—C12—C11	110.98 (18)	C3—C4—C7—C8	−176.41 (14)
C10—C9—C8—C7	178.70 (15)	C12—O2—C16—C17	−18.9 (2)

Table 4
Hydrogen-bond geometry (Å, °) for (II).

*Cg*3 is the centroid of the C16–C21 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C11—H11$\cdots$O1$^{\rm i}$	0.95 (2)	2.36 (2)	3.271 (3)	161 (2)
C18—H18$\cdots$O1$^{\rm ii}$	0.90 (2)	2.48 (2)	3.345 (3)	162 (2)
C5—H5$\cdots$*Cg*3$^{\rm iii}$	0.98 (2)	3.12 (2)	3.894 (3)	137 (1)
C14—H14$\cdots$*Cg*3$^{\rm iv}$	0.95 (2)	2.91 (2)	3.780 (3)	153 (1)

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

Compound (**III**)

Crystal data

$C_{21}H_{14}ClFO_2$
$M_r = 352.77$
Triclinic, $P\overline{1}$
$a = 9.045$ (6) Å
$b = 9.833$ (6) Å
$c = 11.474$ (7) Å
$\alpha = 99.109$ (7)°
$\beta = 104.800$ (8)°
$\gamma = 113.697$ (9)°
$V = 863.3$ (9) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.24$ mm^{-1}
$T = 290$ (2) K
$0.14 \times 0.14 \times 0.13$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.921$, $T_{\max} = 0.969$
8889 measured reflections
3395 independent reflections
2672 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.134$
$S = 1.06$
3395 reflections
282 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 5
Selected geometric parameters (Å, °) for (III).

C4—C7	1.493 (3)	O2—C12	1.389 (2)
C8—C9	1.328 (3)	O2—C16	1.391 (2)
C8—C7	1.471 (3)	O1—C7	1.227 (2)
C10—C9	1.454 (3)		
C5—C4—C7	123.88 (18)	C8—C9—C10	129.2 (2)
C9—C8—C7—C4	177.57 (17)	C11—C10—C9—C8	−172.6 (2)
C3—C4—C7—C8	−169.80 (18)	C16—O2—C12—C11	93.7 (2)
C7—C8—C9—C10	177.47 (18)	C12—O2—C16—C17	−14.0 (3)

Table 6
Hydrogen-bond geometry (Å, °) for (III).

*Cg*3 is the centroid of the C16–C21 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1$^{\rm i}$	0.96 (2)	2.35 (2)	3.285 (3)	165 (2)
C18—H18···O1$^{\rm ii}$	0.87 (2)	2.59 (2)	3.381 (4)	151 (2)
C5—H5···*Cg*3$^{\rm iii}$	0.96 (2)	3.13 (3)	3.940 (4)	144 (2)

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

Compound (IV)

Crystal data

$C_{22}H_{17}FO_2$
$M_r = 332.36$
Triclinic, $P\overline{1}$
$a = 9.196$ (5) Å
$b = 9.726$ (5) Å
$c = 11.554$ (7) Å
$\alpha = 98.887$ (9)°
$\beta = 104.304$ (9)°
$\gamma = 113.754$ (8)°
$V = 878.2$ (9) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 290$ (2) K
0.24 × 0.14 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.940$, $T_{\max} = 0.990$
9092 measured reflections
3476 independent reflections
2575 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.124$
$S = 1.03$
3476 reflections
294 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.13$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

Table 7
Selected geometric parameters (Å, °) for (IV).

C10—C9	1.464 (2)	C8—C9	1.324 (2)
C4—C7	1.487 (2)	C8—C7	1.478 (2)
O2—C16	1.387 (2)	C7—O1	1.2326 (19)
O2—C12	1.3911 (19)		
C5—C4—C7	123.77 (14)	C8—C9—C10	128.49 (15)
C7—C8—C9—C10	177.56 (13)	C3—C4—C7—C8	−169.90 (13)
C11—C10—C9—C8	−174.20 (15)	C16—O2—C12—C11	91.19 (18)
C9—C8—C7—C4	178.26 (13)	C12—O2—C16—C17	−11.6 (2)

Table 8
Hydrogen-bond geometry (Å, °) for (IV).

*Cg*3 is the centroid of the C16–C21 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O1$^{\rm i}$	0.95 (2)	2.35 (2)	3.288 (3)	167 (2)
C18—H18···O1$^{\rm ii}$	0.89 (2)	2.64 (2)	3.443 (3)	150 (2)
C5—H5···*Cg*3$^{\rm iii}$	0.95 (2)	3.21 (2)	3.980 (3)	140 (1)

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

Compound (V)

Crystal data

$C_{22}H_{17}FO_3$
$M_r = 348.36$
Triclinic, $P\overline{1}$
$a = 9.645$ (4) Å
$b = 10.111$ (5) Å
$c = 10.537$ (6) Å
$\alpha = 66.706$ (7)°
$\beta = 65.347$ (8)°
$\gamma = 80.949$ (9)°
$V = 857.8$ (7) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 290$ (2) K
0.12 × 0.08 × 0.04 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.946$, $T_{\max} = 0.996$
8983 measured reflections
3419 independent reflections
2219 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.111$
$S = 1.02$
3419 reflections
303 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 9
Selected geometric parameters (Å, °) for (V).

O2—C12	1.387 (2)	C4—C7	1.487 (3)
O2—C16	1.392 (2)	C7—C8	1.479 (3)
C10—C9	1.468 (3)	C9—C8	1.321 (3)
O1—C7	1.220 (2)		
C5—C4—C7	121.99 (18)	C8—C9—C10	127.0 (2)
C16—O2—C12—C11	115.2 (2)	C10—C9—C8—C7	177.1 (2)
C3—C4—C7—C8	169.3 (2)	C4—C7—C8—C9	153.5 (2)
C11—C10—C9—C8	172.8 (2)	C12—O2—C16—C17	−21.4 (3)

Table 10
Hydrogen-bond geometry (Å, °) for (V).

*Cg*2 is the centroid of the C1–C6 ring.

$D-\mathrm{H}\cdots A$	$D-\mathrm{H}$	$\mathrm{H}\cdots A$	$D\cdots A$	$D-\mathrm{H}\cdots A$
$\mathrm{C3-H3}\cdots\mathrm{O1}^{\mathrm{i}}$	0.94 (2)	2.53 (2)	3.427 (3)	160 (2)
$\mathrm{C18-H18}\cdots\mathrm{O3}^{\mathrm{ii}}$	0.97 (3)	2.59 (3)	3.432 (4)	145 (2)
$\mathrm{C21-H21}\cdots Cg2^{\mathrm{iii}}$	0.97 (3)	2.84 (2)	3.548 (4)	130 (1)

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

Table 11
Dihedral angles (°) between the least-squares planes for all five title compounds.

Compound	1/2	2/3	1/3
(I)	25.2 (1)	20.2 (1)	45.4 (1)
(II)	4.3 (1)	14.2 (1)	18.1 (1)
(III)	10.5 (1)	6.6 (1)	10.4 (2)
(IV)	11.1 (1)	15.5 (3)	10.5 (3)
(V)	23.0 (1)	27.5 (1)	49.1 (2)

Notes: plane 1 = C1–C6; plane 2 = O1/C7/C8/C9; plane 3 = C10–C15/F1.

Table 12
Displacement (Å) of atoms C8 and C9 from the weighted least-squares plane formed by the atom chain C4/C7/O1/C8/C9/C10 for compounds (I)–(V).

Compound	C8	C9
(I)	0.078 (2)	0.077 (2)
(II)	0.005 (2)	0.012 (2)
(III)	0.003 (2)	0.024 (2)
(IV)	0.003 (2)	0.020 (2)
(V)	0.120 (3)	0.139 (3)

In all the five title compounds, H atoms were located from difference Fourier maps and refined isotropically. The C—H bond lengths are in the range 0.87 (2)–1.05 (2) Å.

For all compounds, data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV3115). Services for accessing these data are described at the back of the journal.

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