(2E,2′E)-3,3′-(1,4-Phenylene)bis[1-(4-fluorophenyl)-prop-2-en-1-one]

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The title bis-chalcone compound, $C_{24}H_{16}F_2O_2$, crystallizes with one half-molecule in the asymmetric unit. The molecule lies about an inversion centre at the centroid of the central benzene ring. The olefinic double bonds adopt E conformations. In the crystal, C—H⋯O hydrogen bonds form sheets of molecules in the ac plane and C—H⋯F hydrogen bonds form zigzag chains along the a-axis direction. These combine to generate a three-dimensional network of molecules stacked along the c-axis direction.

3D view
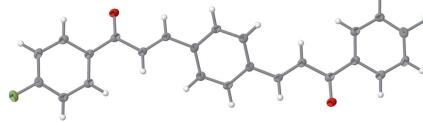
Chemical scheme
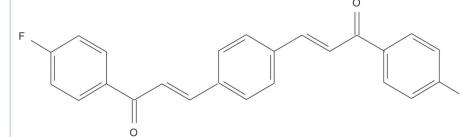
Structure description

Numerous studies have shown that bis-chalcones possess multiple pharmacological properties (Nowakowska, 2007). Materials with large non-linear optical (NLO) susceptibilities are of current interest in the area of harmonic generation and optical modulation. There is also considerable interest in the synthesis of new NLO materials because of their potential applications in technologies such as optical computing and optical communication (Chidan et al., 2015) and because such materials play an important role in fields such as photonics and optoelectronics (Kumar et al. 2013). As a part of our ongoing work on such molecules (Naveen et al., 2017; Rajendraprasad et al., 2017), we report here the crystal structure of the title bis-chalcone derivative.

The title compound crystallizes with one half-molecule in the asymmetric unit and its structure is shown in Fig. 1. The molecule lies about an inversion centre at the centroid of
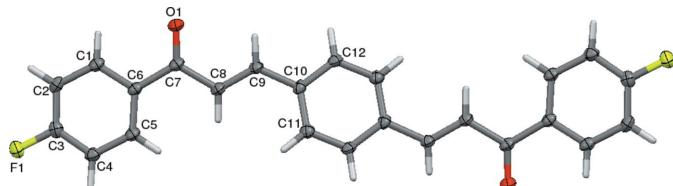
Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme. Labelled atoms are related to unlabelled atoms by the symmetry operation $(-x + 1, -y + 2, -z + 2)$. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

the central C10–C12/C10^i–C12^i, benzene ring [symmetry code: (i) $-x + 1, -y + 2, -z + 2$]. The olefinic double bond adopts an *E* conformation. The *trans* conformation of the C=C double bond in the central enone group is confirmed by the C10—C9—C8=C7 torsion angle of $-178.3 (2)°$. This value is larger than that reported previously for the related compound (2*E*,2′*E*)-1,1′-(1,4-phenylene)bis(3-(3-chlorophenyl)prop-2-en-1-one) (Rajendraprasad *et al.*, 2017).

In the crystal, C5—H5*A*···O1 and C8—H8*A*···O1 hydrogen bonds link molecules into sheets in the *ac* plane, Fig. 2, while C2—H2*A*···F1 hydrogen bonds form zigzag chains along the *a*-axis direction (Table 1). These contacts combine to generate a three-dimensional network with molecules stacked along the *c*-axis direction, Fig. 3.

Synthesis and crystallization

Terephthalaldehyde (1.06 g, 0.01 mol) was mixed with 4-fluoroacetophenone (2.76 g, 0.01 mol) and dissolved in methanol (30 ml). To this, 3 ml of NaOH (50%) was added. The reaction mixture was stirred for 6 h. The resulting crude solid was filtered, washed successively with distilled water and

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···F1^i	0.95	2.50	3.392 (3)	155
C5—H5A···O1^{ii}	0.95	2.59	3.515 (3)	166
C8—H8A···O1^{ii}	0.95	2.63	3.577 (3)	171

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{16}F_2O_2$
M_r	374.37
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	20.405 (6), 3.8237 (11), 11.233 (3)
β (°)	93.989 (5)
V (Å^3)	874.3 (4)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	$0.22 \times 0.15 \times 0.13$
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Multi-scan (*NUMABS*; Rigaku, 1999)
T_{min}, T_{max}	0.977, 0.987
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6328, 1963, 1379
R_{int}	0.039
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.647
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.069, 0.203, 1.09
No. of reflections	1963
No. of parameters	127
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.33, −0.37

Computer programs: *CrystalClear SM-Expert* (Rigaku, 2011), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

finally recrystallized from methanol (90%) to give the pure bis-chalcone. Single crystals suitable for X-ray diffraction studies were grown by the slow evaporation of an acetone-methanol (1:1) solution (m.p. 402–405 K).

Refinement

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

Figure 2
A sheet of molecules in the *ac* plane viewed along the *b* axis, with C—H···O hydrogen bonds drawn as dashed lines.
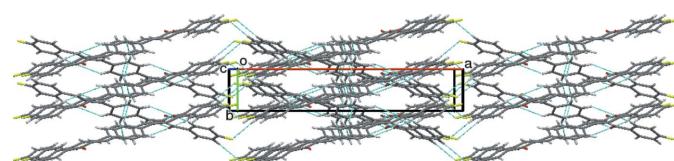
Figure 3
Packing of the molecules viewed along the *c* axis, showing the zigzag arrangement of molecules and layered stacking.

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full crystallographic data

(2*E*,2′*E*)-3,3′-(1,4-Phenylene)bis[1-(4-fluorophenyl)prop-2-en-1-one]

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(2*E*,2′*E*)-3,3′-(1,4-Phenylene)bis[1-(4-fluorophenyl)prop-2-en-1-one]

Crystal data

$C_{24}H_{16}F_2O_2$	$F(000) = 388$
$M_r = 374.37$	$D_x = 1.422$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1379 reflections
$a = 20.405$ (6) Å	$\theta = 2.0$–27.4°
$b = 3.8237$ (11) Å	$\mu = 0.10$ mm^{-1}
$c = 11.233$ (3) Å	$T = 100$ K
$\beta = 93.989$ (5)°	Rectangle, white
$V = 874.3$ (4) Å^3	$0.22 \times 0.15 \times 0.13$ mm
$Z = 2$	

Data collection

Rigaku Saturn724+ diffractometer	6328 measured reflections
Radiation source: fine-focus sealed tube	1963 independent reflections
Graphite monochromator	1379 reflections with $I > 2\sigma(I)$
Detector resolution: 18.4 pixels mm^{-1}	$R_{int} = 0.039$
profile data from ω–scans	$\theta_{max} = 27.4°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (NUMABS; Rigaku, 1999)	$h = -25 \rightarrow 26$
$T_{min} = 0.977$, $T_{max} = 0.987$	$k = -4 \rightarrow 4$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.069$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.203$	$w = 1/[\sigma^2(F_o^2) + (0.1169P)^2 + 0.1724P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	
1963 reflections	
127 parameters	$(\Delta/\sigma)_{max} < 0.001$
0 restraints	$\Delta\rho_{max} = 0.33$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.37$ e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
F1	0.02041 (7)	0.3368 (4)	0.88439 (14)	0.0309 (5)
O1	0.29100 (8)	0.6037 (5)	0.66903 (15)	0.0296 (6)
C1	0.16568 (12)	0.4078 (6)	0.7220 (2)	0.0217 (7)
C2	0.10182 (12)	0.3342 (6)	0.7470 (2)	0.0239 (8)
C3	0.08291 (11)	0.4157 (6)	0.8586 (2)	0.0223 (7)
C4	0.12399 (11)	0.5737 (6)	0.9452 (2)	0.0227 (8)
C5	0.18808 (11)	0.6453 (6)	0.9197 (2)	0.0215 (7)
C6	0.20983 (11)	0.5648 (6)	0.8074 (2)	0.0195 (7)
C7	0.27745 (12)	0.6434 (6)	0.7722 (2)	0.0218 (7)
C8	0.32763 (12)	0.7663 (6)	0.8654 (2)	0.0213 (7)
C9	0.39052 (11)	0.7978 (6)	0.8397 (2)	0.0204 (7)
C10	0.44537 (11)	0.9062 (6)	0.9229 (2)	0.0203 (7)
C11	0.43665 (12)	1.0668 (6)	1.0322 (2)	0.0219 (7)
C12	0.50979 (12)	0.8414 (6)	0.8921 (2)	0.0207 (7)
H1A	0.18000	0.35110	0.64570	0.0260*
H2A	0.07190	0.23040	0.68880	0.0290*
H4A	0.10870	0.63200	1.02070	0.0270*
H5A	0.21740	0.74970	0.97870	0.0260*
H8A	0.31510	0.82230	0.94300	0.0260*
H9A	0.40050	0.74510	0.76030	0.0250*
H11A	0.39350	1.11350	1.05480	0.0260*
H12A	0.51670	0.73270	0.81800	0.0250*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0202 (8)	0.0400 (9)	0.0327 (9)	−0.0027 (6)	0.0033 (6)	0.0003 (7)
O1	0.0271 (10)	0.0471 (12)	0.0147 (9)	−0.0049 (8)	0.0016 (7)	−0.0024 (8)
C1	0.0273 (13)	0.0205 (12)	0.0170 (12)	0.0007 (9)	−0.0015 (9)	0.0007 (9)
C2	0.0233 (13)	0.0247 (13)	0.0225 (13)	−0.0004 (10)	−0.0068 (9)	−0.0015 (10)
C3	0.0203 (12)	0.0194 (12)	0.0271 (14)	0.0010 (9)	0.0004 (9)	0.0044 (9)
C4	0.0243 (13)	0.0256 (14)	0.0180 (12)	0.0020 (10)	0.0008 (9)	0.0004 (9)
C5	0.0249 (13)	0.0230 (12)	0.0161 (12)	0.0004 (10)	−0.0013 (9)	0.0012 (9)
C6	0.0236 (12)	0.0180 (12)	0.0163 (12)	0.0009 (9)	−0.0020 (9)	0.0010 (8)
C7	0.0264 (13)	0.0226 (12)	0.0159 (12)	0.0002 (10)	−0.0019 (9)	0.0006 (9)
C8	0.0224 (12)	0.0253 (13)	0.0157 (12)	−0.0008 (9)	−0.0016 (9)	−0.0006 (9)

C9	0.0226 (13)	0.0241 (13)	0.0144 (12)	−0.0013 (9)	−0.0001 (9)	0.0003 (9)
C10	0.0213 (12)	0.0217 (12)	0.0175 (12)	−0.0029 (9)	−0.0017 (9)	0.0036 (9)
C11	0.0226 (12)	0.0238 (13)	0.0191 (12)	−0.0008 (9)	0.0007 (9)	0.0025 (9)
C12	0.0258 (13)	0.0227 (12)	0.0136 (12)	−0.0013 (9)	0.0009 (9)	0.0000 (9)

Geometric parameters (Å, º)

F1—C3	1.361 (3)	C10—C11	1.395 (3)
O1—C7	1.220 (3)	C10—C12	1.404 (3)
C1—C2	1.381 (3)	C11—C12^{i}	1.383 (3)
C1—C6	1.404 (3)	C1—H1A	0.9500
C2—C3	1.373 (3)	C2—H2A	0.9500
C3—C4	1.379 (3)	C4—H4A	0.9500
C4—C5	1.385 (3)	C5—H5A	0.9500
C5—C6	1.400 (3)	C8—H8A	0.9500
C6—C7	1.492 (3)	C9—H9A	0.9500
C7—C8	1.489 (3)	C11—H11A	0.9500
C8—C9	1.340 (3)	C12—H12A	0.9500
C9—C10	1.467 (3)		
C2—C1—C6	121.2 (2)	C10—C11—C12^{i}	120.6 (2)
C1—C2—C3	117.9 (2)	C10—C12—C11^{i}	121.1 (2)
F1—C3—C2	118.1 (2)	C2—C1—H1A	119.00
F1—C3—C4	118.7 (2)	C6—C1—H1A	119.00
C2—C3—C4	123.2 (2)	C1—C2—H2A	121.00
C3—C4—C5	118.5 (2)	C3—C2—H2A	121.00
C4—C5—C6	120.4 (2)	C3—C4—H4A	121.00
C1—C6—C5	118.7 (2)	C5—C4—H4A	121.00
C1—C6—C7	117.8 (2)	C4—C5—H5A	120.00
C5—C6—C7	123.5 (2)	C6—C5—H5A	120.00
O1—C7—C6	120.1 (2)	C7—C8—H8A	120.00
O1—C7—C8	121.2 (2)	C9—C8—H8A	120.00
C6—C7—C8	118.7 (2)	C8—C9—H9A	117.00
C7—C8—C9	119.9 (2)	C10—C9—H9A	117.00
C8—C9—C10	126.0 (2)	C10—C11—H11A	120.00
C9—C10—C11	123.1 (2)	C12^{i}—C11—H11A	120.00
C9—C10—C12	118.6 (2)	C10—C12—H12A	119.00
C11—C10—C12	118.3 (2)	C11^{i}—C12—H12A	119.00
C6—C1—C2—C3	−0.7 (3)	C5—C6—C7—O1	−171.6 (2)
C2—C1—C6—C5	0.3 (3)	C5—C6—C7—C8	9.1 (3)
C2—C1—C6—C7	−178.6 (2)	O1—C7—C8—C9	−7.5 (4)
C1—C2—C3—F1	−178.6 (2)	C6—C7—C8—C9	171.9 (2)
C1—C2—C3—C4	1.5 (4)	C7—C8—C9—C10	−178.3 (2)
F1—C3—C4—C5	178.3 (2)	C8—C9—C10—C11	−15.2 (4)
C2—C3—C4—C5	−1.8 (4)	C8—C9—C10—C12	163.9 (2)
C3—C4—C5—C6	1.3 (3)	C9—C10—C11—C12^{i}	179.0 (2)
C4—C5—C6—C1	−0.6 (3)	C12—C10—C11—C12^{i}	−0.1 (3)

C4—C5—C6—C7	178.2 (2)	C9—C10—C12—C11ⁱ	−179.1 (2)
C1—C6—C7—O1	7.2 (3)	C11—C10—C12—C11ⁱ	0.1 (3)
C1—C6—C7—C8	−172.1 (2)	C10—C11—C12ⁱ—C10ⁱ	0.1 (3)

Symmetry code: (i) $-x+1, -y+2, -z+2$.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···F1ⁱⁱ	0.95	2.50	3.392 (3)	155
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Symmetry codes: (ii) $-x, y-1/2, -z+3/2$; (iii) $x, -y+3/2, z+1/2$.