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(*E*)-3-[4-(Difluoromethoxy)-3-hydroxyphenyl]-1-phenylprop-2-en-1-one

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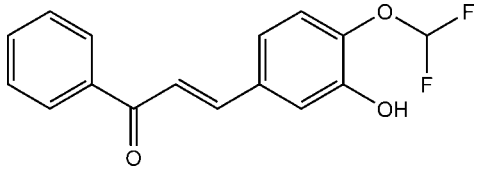
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.120; data-to-parameter ratio = 17.4.

In the title compound, $C_{16}H_{12}F_2O_3$, the plane of the phenyl ring makes a dihedral angle of 3.22 (8)° with that of the benzene ring. The molecule has an *E* conformation about the C=C bond. In the crystal, molecules are linked *via* pairs of O—H···O hydrogen bonds, forming inversion dimers which are further consolidated by a pair of C—H···O hydrogen bonds. The dimers are linked *via* C—H···O hydrogen bonds, forming columns along the *b*-axis direction.

Related literature

For the biological activity of chalcones, see: Di Carlo *et al.* (1999); Lin *et al.* (2002). For a related structure, see: Ranjith *et al.* (2010).



Experimental

Crystal data

$C_{16}H_{12}F_2O_3$
M_r = 290.26
Monoclinic, $P2_1/n$
a = 17.1880 (11) Å
b = 4.1124 (3) Å
c = 19.6699 (13) Å
β = 106.289 (4)°
V = 1334.54 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.966, T_{max} = 0.977
12412 measured reflections
3328 independent reflections
2456 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.120
S = 1.03
3328 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3^{i}	0.82	1.96	2.7722 (16)	172
C4—H4···O3^{i}	0.93	2.48	3.1940 (19)	134
C1—H1···O1^{ii}	0.98	2.40	3.3022 (19)	152

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank the TBI X-ray facility, CAS in Crystallography and Biophysics, University of Madras, India, for the data collection. TS and DV thank the UGC (SAP–CAS) for the departmental facilties. TS also thanks the DST Inspire program for financial assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2584).

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supporting information

Acta Cryst. (2013). E**69**, o812 [https://doi.org/10.1107/S1600536813011288]

(*E*)-3-[4-(Difluoromethoxy)-3-hydroxyphenyl]-1-phenylprop-2-en-1-one

Thothadri Srinivasan, Govindaraj Senthilkumar, Kaliaperumal Neelakandan, Haridoss Manikandan and Devadasan Velmurugan

S1. Comment

Chalcones are a major classes of natural products with widespread distribution in fruits, vegetables, spices, tea and soy based foodstuff and have recently been the subject of great interest for their interesting pharmacological activities (Di Carlo *et al.*, 1999). Chalcones and flavonoids have been reported to be anti-tuberculosis agents (Lin *et al.*, 2002). Against this background and in order to obtain detailed information on molecular conformations in the solid state of similar compounds, we report herein on the synthesis and crystal structure of the title compound.

In the title compound, Fig. 1, the phenyl ring (C2-C7) makes a dihedral angle of 3.22 (8)° with the benzene ring (C11-C16). The hydroxyl oxygen atom, O2, deviates by -0.0243 (12) Å from the benzene ring mean plane.

In the crystal, molecules are linked via a pair of O-H···O hydrogen bonds forming inversion dimers which are further consolidated by a pair of C-H···O hydrogen bonds (Fig. 2 and Table 1). The dimers are linked via C-H···O hydrogen bonds forming columns along the b axis direction (Table 1).

S2. Experimental

A mixture of 3-hydroxy-4-difluoromethoxybezaldehyde (2 mmol), acetophenone (2 mmol) and sodiumhydroxide (2 mmol) in ethanol were placed in a conical flask and exposed to ultrasound irradiation. The reaction mixture was monitored by TLC. After completion of the reaction, the mixture was acidified with dilute HCl and kept in the fridge overnight. The product that separated out was washed with distilled water and recrystallized from ethanol [Yield = 96%; M.p. = 409-411 K]. Colourless block-like crystals, suitable for X-ray diffraction analysis, were obtained by slow evaporation of a solution of the title compound in hexane at room temperature.

S3. Refinement

H atoms were placed in calculated positions, with O-H = 0.82 Å and C—H = 0.93-0.98 Å, and refined in a riding model with U_{iso}(H) = 1.5U_{eq}(O) and = 1.2U_{eq}(C).

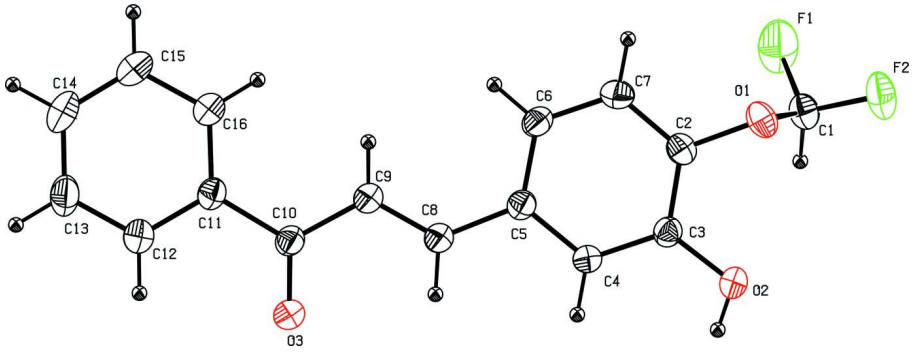

Figure 1

The molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

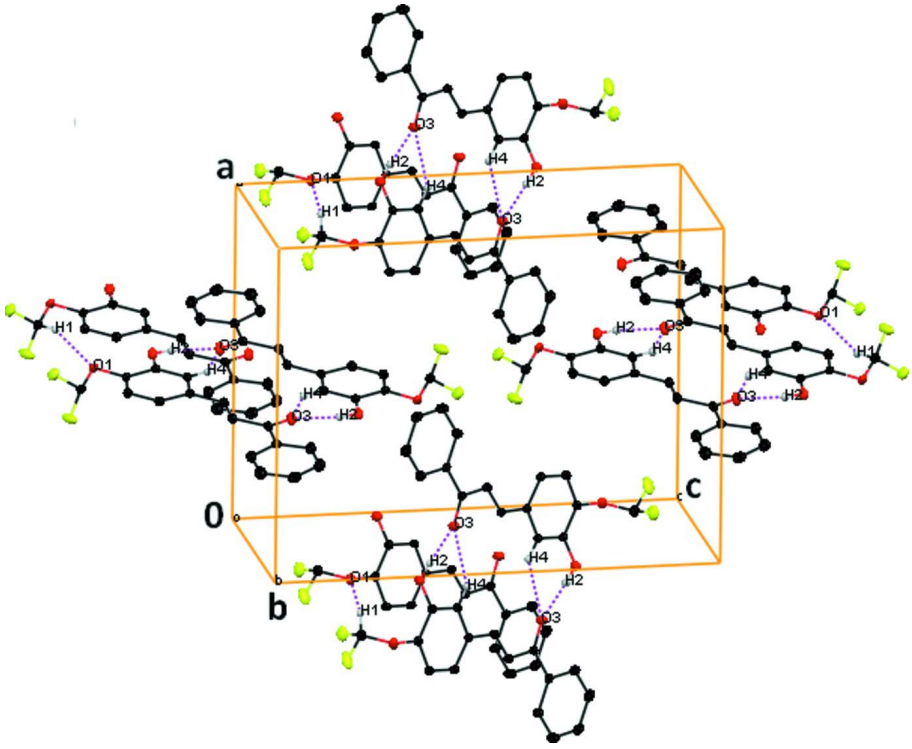

Figure 2

The crystal packing of the title compound viewed along the *b* axis. Hydrogen bonds are shown as dashed lines (see Table 1 for details; H-atoms not involved in hydrogen bonding have been omitted for clarity).

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Crystal data

$C_{16}H_{12}F_2O_3$
$M_r = 290.26$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 17.1880\ (11)$ Å
$b = 4.1124\ (3)$ Å
$c = 19.6699\ (13)$ Å
$\beta = 106.289\ (4)°$
$V = 1334.54\ (16)$ Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.445$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3328 reflections

θ = 1.4–28.3°
μ = 0.12 mm^{-1}
T = 293 K

Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.966, T_{max} = 0.977

12412 measured reflections
3328 independent reflections
2456 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 28.3°, θ_{min} = 1.4°
h = −22→21
k = −5→5
l = −26→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.120
S = 1.03
3328 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.3218P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 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. #== # 8. Refinement Data #== Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) 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}
C1	0.58013 (9)	0.9403 (4)	0.34909 (8)	0.0501 (4)
H1	0.5553	0.7323	0.3305	0.060*
C2	0.57744 (8)	1.0801 (3)	0.23416 (7)	0.0408 (3)
C3	0.52043 (8)	0.8962 (3)	0.18585 (7)	0.0406 (3)
C4	0.53484 (8)	0.8222 (4)	0.12164 (7)	0.0421 (3)
H4	0.4969	0.7001	0.0885	0.051*
C5	0.60481 (7)	0.9264 (3)	0.10571 (7)	0.0399 (3)
C6	0.66097 (8)	1.1133 (4)	0.15554 (7)	0.0459 (3)
H6	0.7078	1.1872	0.1457	0.055*
C7	0.64685 (9)	1.1882 (4)	0.21944 (8)	0.0478 (3)
H7	0.6843	1.3120	0.2527	0.057*
C8	0.61704 (8)	0.8271 (4)	0.03816 (7)	0.0443 (3)

H8	0.5763	0.6990	0.0094	0.053*
C9	0.67854 (8)	0.8960 (4)	0.01260 (7)	0.0471 (3)
H9	0.7203	1.0275	0.0388	0.057*
C10	0.68243 (8)	0.7696 (4)	−0.05624 (7)	0.0434 (3)
C11	0.75269 (8)	0.8529 (3)	−0.08315 (7)	0.0429 (3)
C12	0.75121 (10)	0.7547 (4)	−0.15079 (8)	0.0587 (4)
H12	0.7062	0.6441	−0.1785	0.070*
C13	0.81561 (11)	0.8191 (5)	−0.17761 (9)	0.0662 (5)
H13	0.8143	0.7488	−0.2229	0.079*
C14	0.88160 (10)	0.9868 (5)	−0.13757 (10)	0.0655 (5)
H14	0.9247	1.0326	−0.1560	0.079*
C15	0.88416 (10)	1.0869 (5)	−0.07064 (10)	0.0710 (5)
H15	0.9291	1.1997	−0.0435	0.085*
C16	0.81987 (9)	1.0202 (4)	−0.04329 (8)	0.0576 (4)
H16	0.8219	1.0885	0.0022	0.069*
O1	0.56344 (6)	1.1715 (2)	0.29861 (5)	0.0490 (3)
O2	0.45380 (6)	0.7923 (3)	0.20384 (5)	0.0547 (3)
H2	0.4253	0.6830	0.1715	0.082*
O3	0.62832 (6)	0.5935 (3)	−0.09091 (6)	0.0615 (3)
F1	0.65995 (7)	0.9098 (4)	0.37665 (6)	0.1057 (5)
F2	0.55249 (7)	1.0449 (3)	0.40166 (5)	0.0779 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0568 (8)	0.0515 (8)	0.0449 (7)	0.0069 (7)	0.0190 (6)	−0.0019 (6)
C2	0.0461 (7)	0.0362 (7)	0.0401 (6)	0.0061 (6)	0.0120 (5)	−0.0010 (5)
C3	0.0368 (6)	0.0450 (7)	0.0406 (6)	0.0024 (6)	0.0120 (5)	0.0022 (6)
C4	0.0366 (6)	0.0507 (8)	0.0384 (6)	−0.0055 (6)	0.0097 (5)	−0.0029 (6)
C5	0.0374 (6)	0.0424 (7)	0.0404 (6)	0.0006 (5)	0.0116 (5)	0.0038 (5)
C6	0.0398 (7)	0.0487 (8)	0.0508 (8)	−0.0063 (6)	0.0153 (6)	0.0009 (6)
C7	0.0480 (7)	0.0440 (8)	0.0486 (7)	−0.0067 (6)	0.0090 (6)	−0.0057 (6)
C8	0.0409 (7)	0.0521 (8)	0.0406 (7)	−0.0057 (6)	0.0124 (5)	0.0000 (6)
C9	0.0429 (7)	0.0560 (9)	0.0441 (7)	−0.0083 (6)	0.0150 (6)	−0.0016 (6)
C10	0.0395 (7)	0.0498 (8)	0.0419 (7)	−0.0032 (6)	0.0128 (5)	0.0042 (6)
C11	0.0420 (7)	0.0457 (8)	0.0436 (7)	0.0005 (6)	0.0165 (5)	0.0058 (6)
C12	0.0577 (9)	0.0730 (11)	0.0498 (8)	−0.0128 (8)	0.0224 (7)	−0.0035 (8)
C13	0.0730 (11)	0.0793 (12)	0.0581 (9)	−0.0036 (10)	0.0377 (8)	0.0019 (9)
C14	0.0554 (9)	0.0719 (11)	0.0814 (12)	0.0000 (8)	0.0394 (9)	0.0111 (10)
C15	0.0479 (9)	0.0908 (14)	0.0791 (12)	−0.0185 (9)	0.0258 (8)	−0.0072 (10)
C16	0.0483 (8)	0.0727 (11)	0.0551 (8)	−0.0111 (8)	0.0200 (7)	−0.0068 (8)
O1	0.0638 (6)	0.0415 (5)	0.0433 (5)	0.0093 (5)	0.0178 (4)	−0.0050 (4)
O2	0.0452 (5)	0.0768 (8)	0.0471 (5)	−0.0116 (5)	0.0214 (4)	−0.0088 (5)
O3	0.0530 (6)	0.0835 (8)	0.0511 (6)	−0.0251 (6)	0.0199 (5)	−0.0131 (6)
F1	0.0658 (7)	0.1779 (15)	0.0725 (7)	0.0482 (8)	0.0180 (5)	0.0333 (8)
F2	0.0913 (8)	0.0979 (8)	0.0562 (6)	0.0185 (6)	0.0402 (5)	−0.0023 (5)

Geometric parameters (Å, º)

C1—F2	1.3250 (16)	C8—H8	0.9300
C1—F1	1.3322 (18)	C9—C10	1.4693 (19)
C1—O1	1.3461 (18)	C9—H9	0.9300
C1—H1	0.9800	C10—O3	1.2244 (16)
C2—C7	1.378 (2)	C10—C11	1.4880 (17)
C2—C3	1.3830 (19)	C11—C16	1.383 (2)
C2—O1	1.4063 (16)	C11—C12	1.384 (2)
C3—O2	1.3589 (15)	C12—C13	1.379 (2)
C3—C4	1.3873 (18)	C12—H12	0.9300
C4—C5	1.3925 (17)	C13—C14	1.372 (3)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.3974 (19)	C14—C15	1.368 (3)
C5—C8	1.4605 (18)	C14—H14	0.9300
C6—C7	1.380 (2)	C15—C16	1.385 (2)
C6—H6	0.9300	C15—H15	0.9300
C7—H7	0.9300	C16—H16	0.9300
C8—C9	1.3223 (18)	O2—H2	0.8200
F2—C1—F1	105.47 (13)	C8—C9—C10	121.50 (13)
F2—C1—O1	107.31 (12)	C8—C9—H9	119.3
F1—C1—O1	110.42 (14)	C10—C9—H9	119.3
F2—C1—H1	111.1	O3—C10—C9	119.92 (12)
F1—C1—H1	111.1	O3—C10—C11	120.23 (12)
O1—C1—H1	111.1	C9—C10—C11	119.85 (12)
C7—C2—C3	121.35 (12)	C16—C11—C12	118.51 (13)
C7—C2—O1	118.71 (12)	C16—C11—C10	122.99 (13)
C3—C2—O1	119.88 (12)	C12—C11—C10	118.50 (13)
O2—C3—C2	118.64 (12)	C13—C12—C11	120.78 (15)
O2—C3—C4	123.15 (12)	C13—C12—H12	119.6
C2—C3—C4	118.20 (12)	C11—C12—H12	119.6
C3—C4—C5	121.55 (12)	C14—C13—C12	120.00 (16)
C3—C4—H4	119.2	C14—C13—H13	120.0
C5—C4—H4	119.2	C12—C13—H13	120.0
C4—C5—C6	118.80 (12)	C15—C14—C13	120.14 (15)
C4—C5—C8	118.20 (12)	C15—C14—H14	119.9
C6—C5—C8	122.99 (12)	C13—C14—H14	119.9
C7—C6—C5	119.88 (12)	C14—C15—C16	120.01 (16)
C7—C6—H6	120.1	C14—C15—H15	120.0
C5—C6—H6	120.1	C16—C15—H15	120.0
C2—C7—C6	120.21 (13)	C11—C16—C15	120.55 (15)
C2—C7—H7	119.9	C11—C16—H16	119.7
C6—C7—H7	119.9	C15—C16—H16	119.7
C9—C8—C5	128.35 (13)	C1—O1—C2	114.90 (10)
C9—C8—H8	115.8	C3—O2—H2	109.5
C5—C8—H8	115.8		

C7—C2—C3—O2	178.98 (13)	C8—C9—C10—C11	179.99 (14)
O1—C2—C3—O2	−3.68 (19)	O3—C10—C11—C16	−172.66 (15)
C7—C2—C3—C4	0.0 (2)	C9—C10—C11—C16	6.6 (2)
O1—C2—C3—C4	177.37 (12)	O3—C10—C11—C12	6.5 (2)
O2—C3—C4—C5	−178.42 (13)	C9—C10—C11—C12	−174.29 (14)
C2—C3—C4—C5	0.5 (2)	C16—C11—C12—C13	0.7 (3)
C3—C4—C5—C6	−0.8 (2)	C10—C11—C12—C13	−178.48 (15)
C3—C4—C5—C8	177.55 (13)	C11—C12—C13—C14	−1.1 (3)
C4—C5—C6—C7	0.7 (2)	C12—C13—C14—C15	0.9 (3)
C8—C5—C6—C7	−177.60 (14)	C13—C14—C15—C16	−0.3 (3)
C3—C2—C7—C6	−0.2 (2)	C12—C11—C16—C15	−0.1 (3)
O1—C2—C7—C6	−177.53 (12)	C10—C11—C16—C15	179.00 (16)
C5—C6—C7—C2	−0.2 (2)	C14—C15—C16—C11	−0.1 (3)
C4—C5—C8—C9	−179.68 (15)	F2—C1—O1—C2	−170.69 (12)
C6—C5—C8—C9	−1.4 (3)	F1—C1—O1—C2	74.84 (16)
C5—C8—C9—C10	178.56 (14)	C7—C2—O1—C1	−101.79 (16)
C8—C9—C10—O3	−0.8 (2)	C3—C2—O1—C1	80.80 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3^{i}	0.82	1.96	2.7722 (16)	172
C4—H4···O3^{i}	0.93	2.48	3.1940 (19)	134
C1—H1···O1^{ii}	0.98	2.40	3.3022 (19)	152

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*, *y*−1, *z*.