IUCrData

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(*E*)-3-(2,3-Dichlorophenyl)-1-(4-fluorophenyl)prop-2-en-1-one

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Keywords: crystal structure; chalcone derivative; prop-2-en-1-one; hydrogen bonding.

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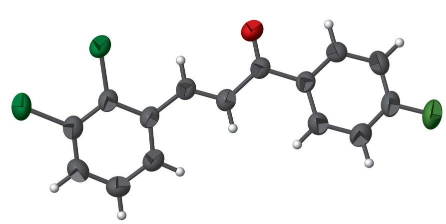
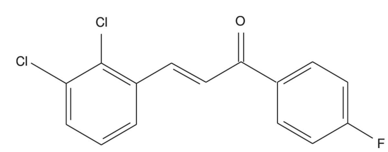
Structural data: full structural data are available from iucrdata.iucr.org

In the title chalcone derivative, $C_{15}H_9Cl_2FO$, the dihedral angle between the aromatic rings is 19.13 (15)° and the double bond adopts an *E* conformation. In the crystal, molecules are connected by weak C—H···O hydrogen bonds, forming a chain propagating along the [001] direction.

3D view



Chemical scheme



Structure description

Chalcones are compounds that contain an α, β-unsaturated carbonyl function. The classical route for the synthesis of chalcones involves the Claisen–Schmidt condensation of an aromatic aldehyde and an aromatic ketone in the presence of aqueous alkaline bases (Jadav *et al.*, 2015). As part of our studies in this area, we herein report the synthesis and crystal structure of the title compound (Fig. 1).

The dihedral angle between the fluorophenyl and the dichlorophenyl rings is 19.35 (15)°. The *trans* conformation of the C7=C8 double bond in the central enone group is confirmed by the C7—C8=C9—C10 torsion angle value of −177.3 (2)°. The major twist in the molecule occurs about the C1—C7 bond, as indicated by the C2—C1—C7—C8 torsion angle of −18.5 (4)°.

In the crystal, the molecules are connected *via* weak C—H···O hydrogen bonds (Table 1), forming a *C*(7) chain propagating along the [001] direction (Fig. 2).

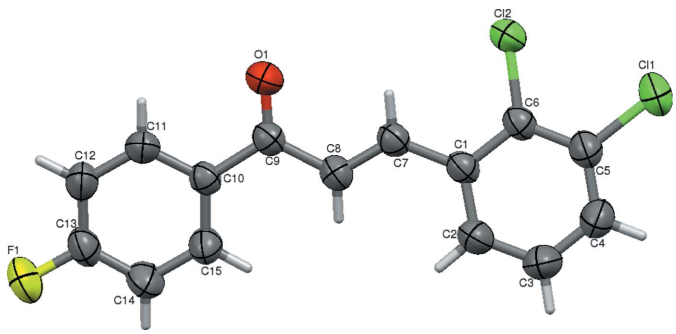


Figure 1
A view of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

A mixture of 2,3-dichlorobenzaldehyde (0.05 mmol), 1-(4-fluorophenyl)ethanone (0.05 mmol) and sodium hydroxide (0.05 mmol) in 80% ethyl alcohol (25 ml) was stirred at room temperature for 3 h. The progress of the reaction was monitored by TLC. After the completion of the reaction, the mixture was poured in to ice-cold water and kept in the refrigerator for 18 h. The solid formed was filtered, and washed with cold acetic acid (5%). It was then recrystallized from dichloromethane solution (with 3–4 drops of acetonitrile added) to get the title compound in the form of green blocks, m.p. 105°C.

Refinement

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

Acknowledgements

The authors are grateful to the Institution of Excellence, Vijnana Bhavana, University of Mysore, India, for providing the single-crystal X-ray diffractometer facility.

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1^{i}	0.93	2.54	3.465 (4)	177

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{9}Cl_{2}FO$
M_r	295.12
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	15.662 (2), 8.1130 (14), 10.9108 (18)
β (°)	106.288 (6)
V (Å^3)	1330.8 (4)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	4.40
Crystal size (mm)	0.28 × 0.26 × 0.25
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.372, 0.406
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9531, 2163, 1881
R_{int}	0.053
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.584
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.065, 0.210, 1.06
No. of reflections	2163
No. of parameters	173
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.51, −0.52

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

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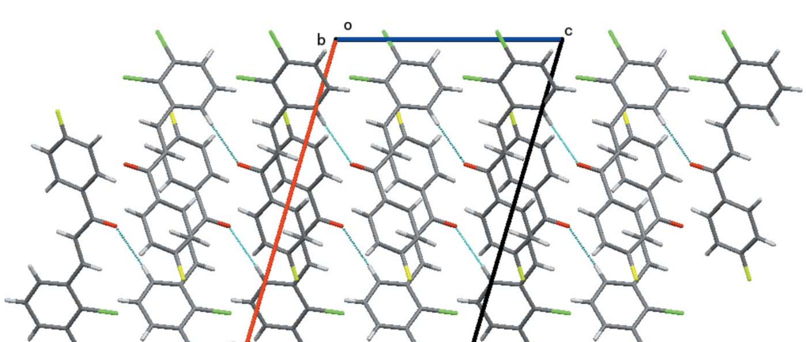

Figure 2
A view along the *b* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1).

full crystallographic data

IUCrData (2016). **1**, x161800 [https://doi.org/10.1107/S2414314616018009]

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Crystal data

$C_{15}H_9Cl_2FO$
M_r = 295.12
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 15.662 (2) Å
b = 8.1130 (14) Å
c = 10.9108 (18) Å
β = 106.288 (6)°
V = 1330.8 (4) Å^3
Z = 4
$F(000)$ = 600
D_x = 1.473 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 1881 reflections
θ = 6.2–64.2°
μ = 4.40 mm^{-1}
T = 296 K
Block, green
0.28 × 0.26 × 0.25 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.372, T_{max} = 0.406
9531 measured reflections
2163 independent reflections
1881 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 64.2°, θ_{min} = 6.2°
$h = -18 \rightarrow 18$
$k = -9 \rightarrow 9$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.210
S = 1.06
2163 reflections
173 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1575P)^2 + 0.2475P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 2008), $FC^*=KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Extinction coefficient: 0.017 (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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.02367 (6)	0.65394 (16)	0.20294 (10)	0.0936 (5)
Cl2	0.12132 (5)	0.46410 (11)	0.11655 (7)	0.0673 (4)
F1	0.76924 (12)	0.0730 (3)	0.6363 (2)	0.0855 (8)
O1	0.40095 (14)	0.1717 (3)	0.2333 (2)	0.0638 (8)
C1	0.21279 (17)	0.4408 (3)	0.3681 (3)	0.0466 (9)
C2	0.2207 (2)	0.4739 (5)	0.4950 (3)	0.0633 (11)
C3	0.1557 (2)	0.5621 (5)	0.5317 (4)	0.0747 (14)
C4	0.0817 (2)	0.6185 (4)	0.4421 (4)	0.0676 (11)
C5	0.07215 (18)	0.5874 (4)	0.3154 (3)	0.0568 (10)
C6	0.13620 (16)	0.4998 (3)	0.2767 (3)	0.0471 (8)
C7	0.27948 (17)	0.3469 (3)	0.3273 (3)	0.0476 (9)
C8	0.36255 (17)	0.3194 (3)	0.3957 (3)	0.0496 (9)
C9	0.42414 (17)	0.2226 (3)	0.3428 (3)	0.0458 (8)
C10	0.51619 (16)	0.1877 (3)	0.4247 (3)	0.0433 (8)
C11	0.56881 (19)	0.0816 (4)	0.3774 (3)	0.0549 (10)
C12	0.6542 (2)	0.0431 (4)	0.4488 (4)	0.0628 (11)
C13	0.68611 (17)	0.1113 (4)	0.5673 (3)	0.0577 (10)
C14	0.6357 (2)	0.2145 (4)	0.6183 (3)	0.0646 (11)
C15	0.55032 (18)	0.2513 (4)	0.5467 (3)	0.0563 (10)
H2	0.27050	0.43640	0.55720	0.0760*
H3	0.16260	0.58280	0.61780	0.0890*
H4	0.03840	0.67750	0.46680	0.0810*
H7	0.26200	0.30220	0.24560	0.0570*
H8	0.38250	0.36150	0.47810	0.0590*
H11	0.54630	0.03580	0.29660	0.0660*
H12	0.68930	−0.02790	0.41670	0.0750*
H14	0.65870	0.25870	0.69960	0.0770*
H15	0.51510	0.31980	0.58070	0.0680*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0664 (6)	0.1289 (10)	0.0747 (8)	0.0475 (5)	0.0020 (5)	−0.0026 (5)
Cl2	0.0556 (6)	0.0992 (7)	0.0429 (6)	0.0174 (3)	0.0071 (4)	0.0060 (4)
F1	0.0460 (10)	0.1197 (17)	0.0812 (16)	0.0191 (10)	0.0020 (9)	0.0054 (12)
O1	0.0558 (12)	0.0856 (14)	0.0449 (13)	0.0090 (10)	0.0059 (10)	−0.0047 (11)
C1	0.0388 (13)	0.0550 (14)	0.0430 (17)	−0.0003 (10)	0.0066 (11)	0.0061 (11)
C2	0.0520 (16)	0.088 (2)	0.0457 (19)	0.0077 (14)	0.0069 (14)	0.0009 (15)
C3	0.067 (2)	0.107 (3)	0.049 (2)	0.0118 (18)	0.0146 (16)	−0.0114 (18)

C4	0.0566 (18)	0.080 (2)	0.067 (2)	0.0125 (15)	0.0187 (16)	−0.0123 (17)
C5	0.0472 (15)	0.0613 (15)	0.058 (2)	0.0083 (12)	0.0085 (13)	−0.0003 (14)
C6	0.0390 (13)	0.0522 (13)	0.0474 (17)	−0.0013 (11)	0.0076 (11)	0.0049 (12)
C7	0.0438 (14)	0.0530 (14)	0.0447 (17)	0.0030 (10)	0.0103 (11)	0.0074 (12)
C8	0.0431 (14)	0.0542 (15)	0.0484 (17)	0.0028 (11)	0.0080 (11)	0.0015 (12)
C9	0.0435 (13)	0.0475 (13)	0.0440 (17)	0.0008 (10)	0.0085 (11)	0.0049 (11)
C10	0.0406 (13)	0.0456 (12)	0.0431 (16)	0.0005 (10)	0.0106 (11)	0.0066 (11)
C11	0.0546 (16)	0.0687 (17)	0.0415 (18)	0.0126 (13)	0.0135 (13)	0.0034 (13)
C12	0.0542 (16)	0.076 (2)	0.061 (2)	0.0201 (14)	0.0206 (15)	0.0078 (15)
C13	0.0384 (14)	0.0694 (17)	0.062 (2)	0.0054 (12)	0.0086 (13)	0.0101 (15)
C14	0.0502 (15)	0.077 (2)	0.058 (2)	0.0015 (14)	0.0010 (14)	−0.0128 (15)
C15	0.0449 (15)	0.0621 (16)	0.059 (2)	0.0049 (12)	0.0100 (13)	−0.0106 (14)

Geometric parameters (Å, º)

Cl1—C5	1.737 (3)	C10—C15	1.387 (4)
Cl2—C6	1.721 (3)	C11—C12	1.382 (5)
F1—C13	1.345 (4)	C12—C13	1.366 (5)
O1—C9	1.219 (4)	C13—C14	1.371 (4)
C1—C2	1.381 (4)	C14—C15	1.379 (4)
C1—C6	1.411 (4)	C2—H2	0.9300
C1—C7	1.460 (4)	C3—H3	0.9300
C2—C3	1.392 (5)	C4—H4	0.9300
C3—C4	1.369 (5)	C7—H7	0.9300
C4—C5	1.372 (5)	C8—H8	0.9300
C5—C6	1.388 (4)	C11—H11	0.9300
C7—C8	1.325 (4)	C12—H12	0.9300
C8—C9	1.481 (4)	C14—H14	0.9300
C9—C10	1.495 (4)	C15—H15	0.9300
C10—C11	1.388 (4)		
C2—C1—C6	117.5 (3)	F1—C13—C14	119.4 (3)
C2—C1—C7	122.4 (3)	C12—C13—C14	122.2 (3)
C6—C1—C7	120.1 (3)	C13—C14—C15	118.7 (3)
C1—C2—C3	121.4 (3)	C10—C15—C14	121.0 (3)
C2—C3—C4	120.5 (4)	C1—C2—H2	119.00
C3—C4—C5	119.3 (3)	C3—C2—H2	119.00
Cl1—C5—C4	118.9 (2)	C2—C3—H3	120.00
Cl1—C5—C6	119.9 (2)	C4—C3—H3	120.00
C4—C5—C6	121.1 (3)	C3—C4—H4	120.00
Cl2—C6—C1	120.4 (2)	C5—C4—H4	120.00
Cl2—C6—C5	119.4 (2)	C1—C7—H7	117.00
C1—C6—C5	120.2 (3)	C8—C7—H7	117.00
C1—C7—C8	126.2 (3)	C7—C8—H8	120.00
C7—C8—C9	120.9 (3)	C9—C8—H8	120.00
O1—C9—C8	120.9 (3)	C10—C11—H11	120.00
O1—C9—C10	119.6 (3)	C12—C11—H11	120.00
C8—C9—C10	119.5 (3)	C11—C12—H12	121.00

C9—C10—C11	118.0 (3)	C13—C12—H12	121.00
C9—C10—C15	123.4 (2)	C13—C14—H14	121.00
C11—C10—C15	118.6 (3)	C15—C14—H14	121.00
C10—C11—C12	120.8 (3)	C10—C15—H15	120.00
C11—C12—C13	118.8 (3)	C14—C15—H15	120.00
F1—C13—C12	118.4 (3)		

C6—C1—C2—C3	0.0 (5)	C7—C8—C9—O1	3.0 (4)
C7—C1—C2—C3	−179.2 (3)	C7—C8—C9—C10	−177.3 (2)
C2—C1—C6—Cl2	179.9 (2)	O1—C9—C10—C11	−7.4 (4)
C2—C1—C6—C5	−0.1 (4)	O1—C9—C10—C15	174.9 (3)
C7—C1—C6—Cl2	−0.8 (3)	C8—C9—C10—C11	173.0 (3)
C7—C1—C6—C5	179.2 (3)	C8—C9—C10—C15	−4.8 (4)
C2—C1—C7—C8	−18.5 (4)	C9—C10—C11—C12	−179.4 (3)
C6—C1—C7—C8	162.3 (3)	C15—C10—C11—C12	−1.6 (5)
C1—C2—C3—C4	0.1 (6)	C9—C10—C15—C14	179.8 (3)
C2—C3—C4—C5	−0.1 (5)	C11—C10—C15—C14	2.1 (4)
C3—C4—C5—Cl1	178.0 (3)	C10—C11—C12—C13	0.1 (5)
C3—C4—C5—C6	0.0 (5)	C11—C12—C13—F1	179.9 (3)
Cl1—C5—C6—Cl2	2.1 (3)	C11—C12—C13—C14	1.0 (5)
Cl1—C5—C6—C1	−177.9 (2)	F1—C13—C14—C15	−179.4 (3)
C4—C5—C6—Cl2	−179.9 (3)	C12—C13—C14—C15	−0.6 (5)
C4—C5—C6—C1	0.1 (4)	C13—C14—C15—C10	−1.0 (5)
C1—C7—C8—C9	−179.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1^{i}	0.93	2.54	3.465 (4)	177

Symmetry code: (i) *x*, −*y*+1/2, *z*+1/2.