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ISSN 2414-3146

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

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Received 23 December 2016
Accepted 28 December 2016

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

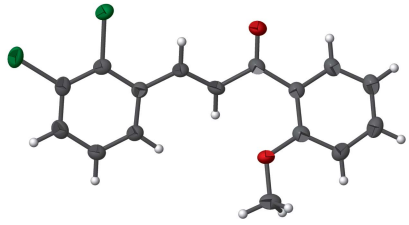
Keywords: crystal structure; bis-chalcone; weak C—H···O hydrogen bonds.

CCDC reference: 1524698

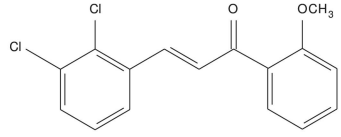
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_{12}Cl_2O_2$, the olefinic double bond adopts an *E* configuration. The molecule is non-planar, as shown by the dihedral angle of 15.40 (19)° between the 2,3-dichlorophenyl ring and the 2-methoxyphenyl ring. In the crystal, molecules are linked *via* weak C—H···O hydrogen bonds, forming zigzag chains propagating along the *c* axis.

3D view



Chemical scheme



Structure description

Chalcones form the central core for the construction of a variety of bioactive compounds. The usual method for the synthesis of chalcones involves the condensation of aromatic aldehyde and aromatic ketone in the presence of aqueous alkaline bases (Naveen *et al.*, 2016*a*). Chalcones and their derivatives demonstrate a wide range of biological activities such as anti-diabetic, anti-neoplastic, anti-hypertensive, anti-inflammatory, anti-malarial, anti-oxidant, anti-fungal, *etc*. (Mahapatra *et al.*, 2015). The α,β-unsaturated carbonyl system of chalcones makes them useful as building blocks in organic synthesis. They have been efficiently employed as precursors in the synthesis of biologically potent benzothiazepines (Naveen *et al.*, 2016*b*). In view of the diverse applications of chalcones and as a part of our ongoing work on such molecules (Tejkiran *et al.*, 2016), we report here the synthesis and crystal structure of the title compound.

The title molecule (Fig. 1) is non-planar, with a dihedral angle of 15.40 (19)° between the dichlorophenyl (C1–C6) and methoxyphenyl (C10–C15) rings that are bridged by the olefinic double bond C7═C8. This is comparable to the value of 19.13 (15)° reported for

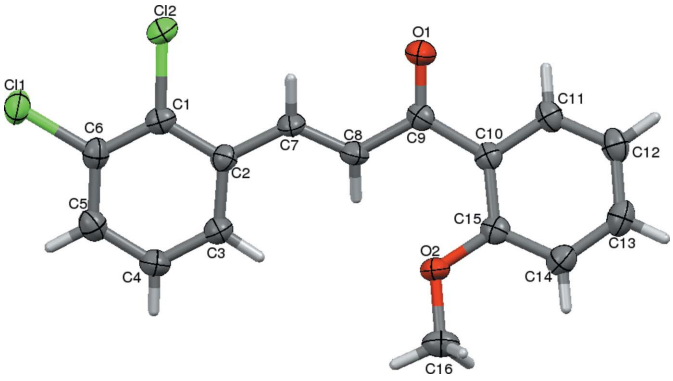


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

the related chalcone derivative (*E*)-3-(2,3-dichlorophenyl)-1-(4-fluorophenyl)prop-2-en-1-one (Naveen *et al.*, 2016*a*). The *trans* configuration of the C7═C8 double bond in the central enone group is confirmed by the C7═C8—C9—C10 torsion angle value of 162.3 (4)°. The carbonyl group at C9 lies nearly in the plane of the benzene ring (C10–C15), as indicated by the torsion angle values of −10.4 (5)° and −19.3 (6)° for O1—C9—C10—C11 and C7—C8—C9—O1, respectively. The methoxy group at C15 also lies in the plane of the benzene ring (C10–C15), as indicated by the torsion angle value of 0.7 (5)° for C16—O2—C15—C14.

In the crystal, molecules are linked *via* weak C—H···O hydrogen bonds, forming zigzag chains propagating along the *c* axis (Table 1 and Fig. 2).

Synthesis and crystallization

A mixture of 2,4,5-trimethoxybenzaldehyde (5 mmol), 1-(benzo[*d*][1,3]dioxol-5-yl)ethanone (5 mmol) and sodium hydroxide (5 mmol) in 95% 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 overnight. The solid that formed was filtered off, and washed with cold hydrochloric acid (5%). Yellow block-like crystals were obtained by slow evaporation of a solution in methanol (yield 89%, m.p. 399–401 K).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O1^{i}	0.93	2.40	3.315 (5)	167
C16—H16*A*···O1^{ii}	0.96	2.51	3.443 (5)	164

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{12}Cl_2O_2$
M_r	307.16
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	296
a, *b*, *c* (Å)	24.7531 (9), 3.9036 (2), 14.3360 (6)
V (Å^3)	1385.23 (10)
Z	4
Radiation type	Cu *Kα*
μ (mm^{-1})	4.20
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}	0.386, 0.420
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7897, 2175, 1893
R_{int}	0.067
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.586
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.049, 0.127, 1.03
No. of reflections	2175
No. of parameters	182
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.33, −0.37
Absolute structure	971 (86%) Friedel pairs; Flack (1983)
Absolute structure parameter	0.03 (2)

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

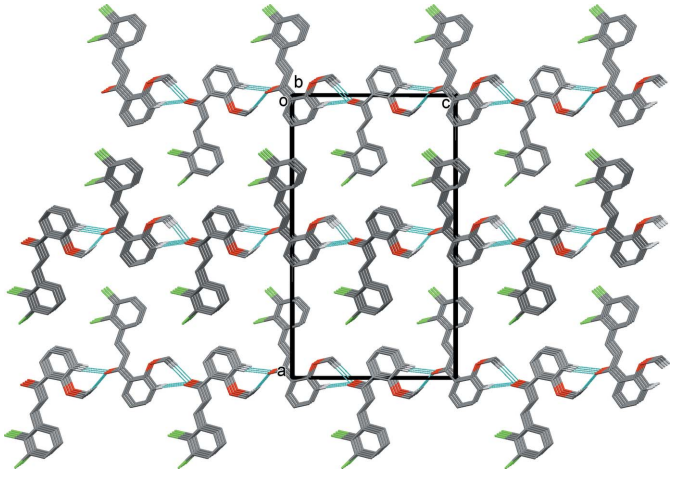


Figure 2
A view along the *b* axis of the crystal packing of the title compound. The dashed lines represent hydrogen bonds (see Table 1) and, for clarity, only H atoms H14 and H16*A* have been included.

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.

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full crystallographic data

IUCrData (2017). **2**, x162056 [https://doi.org/10.1107/S2414314616020563]

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

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

Crystal data

$C_{16}H_{12}Cl_2O_2$
M_r = 307.16
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
a = 24.7531 (9) Å
b = 3.9036 (2) Å
c = 14.3360 (6) Å
V = 1385.23 (10) Å^3
Z = 4
$F(000)$ = 632
D_x = 1.473 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 1893 reflections
θ = 3.6–64.6°
μ = 4.20 mm^{-1}
T = 296 K
Block, yellow
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} = 0.386, T_{max} = 0.420
7897 measured reflections
2175 independent reflections
1893 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 64.6°, θ_{min} = 3.6°
h = −28→27
k = −4→4
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.127
S = 1.03
2175 reflections
182 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0694P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Absolute structure: 971 (86%) Friedel pairs; Flack (1983)
Absolute structure parameter: 0.03 (2)

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	0.80662 (4)	1.1660 (3)	0.33612 (8)	0.0407 (3)
Cl2	0.69437 (4)	0.8842 (3)	0.26987 (7)	0.0381 (3)
O1	0.51800 (11)	0.4104 (7)	0.3625 (2)	0.0379 (10)
O2	0.55245 (10)	0.1930 (7)	0.6377 (2)	0.0327 (8)
C1	0.70987 (16)	0.8885 (10)	0.3882 (3)	0.0287 (12)
C2	0.67209 (15)	0.7666 (10)	0.4528 (3)	0.0285 (11)
C3	0.68707 (15)	0.7817 (13)	0.5468 (3)	0.0328 (11)
C4	0.73578 (17)	0.9109 (11)	0.5754 (3)	0.0363 (14)
C5	0.77279 (16)	1.0284 (11)	0.5106 (3)	0.0357 (14)
C6	0.75962 (15)	1.0168 (11)	0.4167 (3)	0.0318 (11)
C7	0.62046 (16)	0.6269 (10)	0.4238 (3)	0.0307 (12)
C8	0.57938 (16)	0.5491 (11)	0.4795 (3)	0.0315 (11)
C9	0.52915 (15)	0.3920 (10)	0.4451 (3)	0.0277 (11)
C10	0.49098 (15)	0.2178 (9)	0.5104 (3)	0.0266 (11)
C11	0.44040 (15)	0.1365 (10)	0.4748 (3)	0.0313 (11)
C12	0.40200 (15)	−0.0369 (10)	0.5257 (3)	0.0352 (13)
C13	0.41414 (17)	−0.1308 (10)	0.6165 (3)	0.0383 (14)
C14	0.46347 (17)	−0.0531 (11)	0.6552 (3)	0.0360 (12)
C15	0.50218 (16)	0.1218 (9)	0.6032 (3)	0.0291 (11)
C16	0.56561 (19)	0.0802 (11)	0.7298 (3)	0.0373 (14)
H3	0.66300	0.70090	0.59150	0.0390*
H4	0.74400	0.91970	0.63870	0.0440*
H5	0.80600	1.11400	0.52980	0.0430*
H7	0.61570	0.58820	0.36030	0.0370*
H8	0.58250	0.59580	0.54290	0.0380*
H11	0.43210	0.20190	0.41410	0.0370*
H12	0.36860	−0.09000	0.49960	0.0420*
H13	0.38860	−0.24780	0.65180	0.0460*
H14	0.47100	−0.11760	0.71620	0.0430*
H16A	0.54100	0.18170	0.77370	0.0560*
H16B	0.60190	0.14790	0.74470	0.0560*
H16C	0.56270	−0.16480	0.73300	0.0560*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0297 (5)	0.0456 (6)	0.0467 (7)	−0.0055 (4)	0.0074 (4)	0.0066 (5)
Cl2	0.0386 (5)	0.0509 (6)	0.0248 (5)	−0.0053 (4)	0.0038 (4)	0.0028 (4)
O1	0.0391 (16)	0.0498 (18)	0.0248 (16)	−0.0084 (13)	−0.0048 (12)	0.0012 (12)

O2	0.0357 (15)	0.0411 (15)	0.0212 (14)	−0.0048 (13)	−0.0032 (11)	0.0045 (11)
C1	0.034 (2)	0.025 (2)	0.027 (2)	0.0053 (16)	0.0015 (17)	0.0002 (15)
C2	0.0266 (19)	0.032 (2)	0.027 (2)	0.0017 (17)	0.0019 (16)	−0.0003 (16)
C3	0.0324 (19)	0.039 (2)	0.027 (2)	−0.002 (2)	0.0010 (17)	0.0017 (16)
C4	0.035 (2)	0.046 (3)	0.028 (2)	−0.0023 (19)	−0.0037 (17)	0.0019 (19)
C5	0.030 (2)	0.035 (2)	0.042 (3)	0.0002 (17)	−0.0047 (18)	0.0006 (19)
C6	0.0274 (17)	0.028 (2)	0.040 (2)	0.0005 (17)	0.0033 (16)	0.0023 (17)
C7	0.032 (2)	0.040 (2)	0.020 (2)	−0.0046 (18)	0.0007 (15)	−0.0016 (15)
C8	0.0315 (19)	0.040 (2)	0.023 (2)	−0.0008 (19)	−0.0050 (15)	−0.0015 (16)
C9	0.0267 (18)	0.0285 (19)	0.028 (2)	0.0040 (15)	−0.0015 (16)	−0.0041 (14)
C10	0.0270 (18)	0.0244 (18)	0.0284 (19)	0.0026 (15)	0.0015 (15)	−0.0040 (16)
C11	0.0270 (19)	0.029 (2)	0.038 (2)	0.0024 (16)	−0.0014 (16)	−0.0050 (15)
C12	0.0256 (18)	0.034 (2)	0.046 (3)	0.0032 (17)	0.0018 (18)	−0.005 (2)
C13	0.035 (2)	0.036 (2)	0.044 (3)	−0.0021 (18)	0.0114 (18)	−0.0003 (18)
C14	0.037 (2)	0.037 (2)	0.034 (2)	0.001 (2)	0.0063 (17)	0.0037 (17)
C15	0.031 (2)	0.0274 (19)	0.029 (2)	0.0040 (16)	0.0007 (16)	−0.0043 (14)
C16	0.049 (3)	0.034 (2)	0.029 (2)	0.0068 (19)	−0.0054 (18)	0.0011 (16)

Geometric parameters (Å, º)

Cl1—C6	1.740 (4)	C11—C12	1.376 (6)
Cl2—C1	1.739 (4)	C12—C13	1.385 (6)
O1—C9	1.218 (5)	C13—C14	1.375 (6)
O2—C15	1.368 (5)	C14—C15	1.393 (6)
O2—C16	1.429 (5)	C3—H3	0.9300
C1—C2	1.400 (6)	C4—H4	0.9300
C1—C6	1.391 (6)	C5—H5	0.9300
C2—C3	1.399 (6)	C7—H7	0.9300
C2—C7	1.450 (5)	C8—H8	0.9300
C3—C4	1.370 (6)	C11—H11	0.9300
C4—C5	1.383 (6)	C12—H12	0.9300
C5—C6	1.386 (6)	C13—H13	0.9300
C7—C8	1.328 (6)	C14—H14	0.9300
C8—C9	1.472 (6)	C16—H16A	0.9600
C9—C10	1.494 (6)	C16—H16B	0.9600
C10—C11	1.389 (5)	C16—H16C	0.9600
C10—C15	1.410 (6)		
C15—O2—C16	118.6 (3)	O2—C15—C14	122.1 (4)
Cl2—C1—C2	119.7 (3)	C10—C15—C14	120.0 (4)
Cl2—C1—C6	119.0 (3)	C2—C3—H3	119.00
C2—C1—C6	121.3 (4)	C4—C3—H3	119.00
C1—C2—C3	116.5 (4)	C3—C4—H4	120.00
C1—C2—C7	121.8 (4)	C5—C4—H4	120.00
C3—C2—C7	121.7 (4)	C4—C5—H5	120.00
C2—C3—C4	122.5 (4)	C6—C5—H5	121.00
C3—C4—C5	120.3 (4)	C2—C7—H7	117.00
C4—C5—C6	119.1 (4)	C8—C7—H7	117.00

Cl1—C6—C1	121.2 (3)	C7—C8—H8	119.00
Cl1—C6—C5	118.5 (3)	C9—C8—H8	119.00
C1—C6—C5	120.4 (4)	C10—C11—H11	119.00
C2—C7—C8	126.0 (4)	C12—C11—H11	119.00
C7—C8—C9	122.7 (4)	C11—C12—H12	121.00
O1—C9—C8	119.5 (4)	C13—C12—H12	121.00
O1—C9—C10	119.5 (3)	C12—C13—H13	120.00
C8—C9—C10	120.9 (4)	C14—C13—H13	120.00
C9—C10—C11	116.4 (4)	C13—C14—H14	120.00
C9—C10—C15	126.0 (3)	C15—C14—H14	120.00
C11—C10—C15	117.6 (4)	O2—C16—H16A	109.00
C10—C11—C12	122.7 (4)	O2—C16—H16B	110.00
C11—C12—C13	118.6 (4)	O2—C16—H16C	110.00
C12—C13—C14	120.9 (4)	H16A—C16—H16B	109.00
C13—C14—C15	120.2 (4)	H16A—C16—H16C	109.00
O2—C15—C10	117.8 (3)	H16B—C16—H16C	109.00

C16—O2—C15—C10	−176.5 (3)	C2—C7—C8—C9	−176.9 (4)
C16—O2—C15—C14	0.7 (5)	C7—C8—C9—O1	−19.3 (6)
Cl2—C1—C2—C3	−179.3 (3)	C7—C8—C9—C10	162.3 (4)
Cl2—C1—C2—C7	1.7 (5)	O1—C9—C10—C11	−10.4 (5)
C6—C1—C2—C3	0.1 (6)	O1—C9—C10—C15	167.8 (4)
C6—C1—C2—C7	−178.9 (4)	C8—C9—C10—C11	168.1 (4)
Cl2—C1—C6—Cl1	−1.0 (5)	C8—C9—C10—C15	−13.7 (6)
Cl2—C1—C6—C5	179.0 (3)	C9—C10—C11—C12	177.2 (4)
C2—C1—C6—Cl1	179.7 (3)	C15—C10—C11—C12	−1.2 (6)
C2—C1—C6—C5	−0.4 (6)	C9—C10—C15—O2	0.0 (6)
C1—C2—C3—C4	0.6 (7)	C9—C10—C15—C14	−177.2 (4)
C7—C2—C3—C4	179.6 (4)	C11—C10—C15—O2	178.3 (3)
C1—C2—C7—C8	−170.2 (4)	C11—C10—C15—C14	1.0 (5)
C3—C2—C7—C8	10.8 (7)	C10—C11—C12—C13	0.7 (6)
C2—C3—C4—C5	−1.0 (7)	C11—C12—C13—C14	0.0 (6)
C3—C4—C5—C6	0.7 (7)	C12—C13—C14—C15	−0.1 (6)
C4—C5—C6—Cl1	180.0 (3)	C13—C14—C15—O2	−177.5 (4)
C4—C5—C6—C1	0.0 (6)	C13—C14—C15—C10	−0.4 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O1^{i}	0.93	2.40	3.315 (5)	167
C16—H16*A*···O1^{ii}	0.96	2.51	3.443 (5)	164

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