IUCrData

ISSN 2414-3146

Received 19 January 2017
Accepted 20 January 2017

Edited by J. Simpson, University of Otago, New Zealand

Keywords: crystal structure; bis-chalcone; *E* configuration.

CCDC reference: 1528716

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

(*E*)-1-(Benzo[*d*][1,3]dioxol-5-yl)-3-(2,3-dichlorophenyl)prop-2-en-1-one

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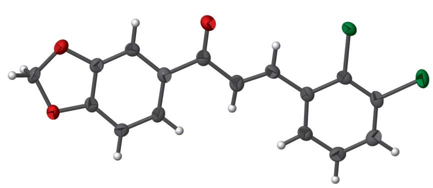
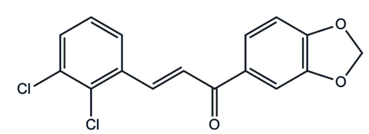
In the title compound, $C_{16}H_{10}Cl_2O_3$, the olefinic double bond adopts an *E* conformation [C—C═C—C torsion angle = −172.9 (3)°]. The dihedral angle between the benzodioxole and dichlorobenzene rings is 5.57 (9)°. In the crystal, molecules are linked by weak C—H···O and C—H···Cl hydrogen bonds, forming chains propagating along the *c*-axis direction.

3D view



Chemical scheme



Structure description

Chalcones constitute the central cores for the construction of a wide range of bioactive compounds (Naveen *et al.*, 2016). As part of our ongoing work on such molecules (Kumara *et al.*, 2017), we report the synthesis and crystal structure of the title compound here.

The structure of the molecule is shown in Fig. 1. The molecule is nearly planar, with a dihedral angle of 5.57 (9)° between the benzodioxole and dichloro benzene rings that are bridged by the olefinic double bond. This value is comparable with the value of 6.99 (6)° reported earlier between the aromatic rings in the related chalcone derivative (*E*)-1-(1,3-benzodioxol-5-yl)-3-(2,4,5-trimethoxy-phenyl)prop-2-en-1-one. (Sunitha *et al.*, 2017). The *trans* conformation about the C7═C8 double bond in the central enone group is confirmed by the C4—C7═C8—C9 torsion angle, −172.9 (3)°. The carbonyl group at C7 lies almost in the plane of the olefinic double bond and benzodioxole ring as indicated by the C3—C4—C7—O3 and O3—C7—C8—C9 torsion angles 6.2 (3)° and 7.6 (4)° respectively.

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
C9—H9$\cdots$Cl1	0.93	2.58	3.033 (3)	110
C9—H9$\cdots$O3	0.93	2.48	2.809 (4)	101
C14—H14$\cdots$O1^{i}	0.93	2.59	3.194 (3)	123

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

In the crystal, the molecules are linked *via* weak C—H$\cdots$O and C—H$\cdots$Cl hydrogen bonds, forming chains propagating along the *c*-axis direction (Table 1 and Fig. 2).

Synthesis and crystallization

A mixture of 2,3-dichlorobenzaldehyde (5 mmol), 1-(benzo[*d*][1,3]dioxol-5-yl)ethanone (5 mmol) and sodium hydroxide (5 mmol) in methanol (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 into ice-cold water and kept in a refrigerator for 18 h. The solid that formed was filtered, and washed with cold hydrochloric acid (5%). Pure white crystals were obtained by slow evaporation from a methanol solution (yield 88%, m.p. 401–402 K).

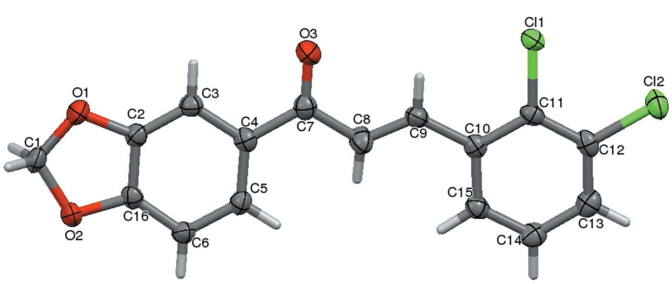


Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids for the non-H atoms are drawn at the 50% probability level.

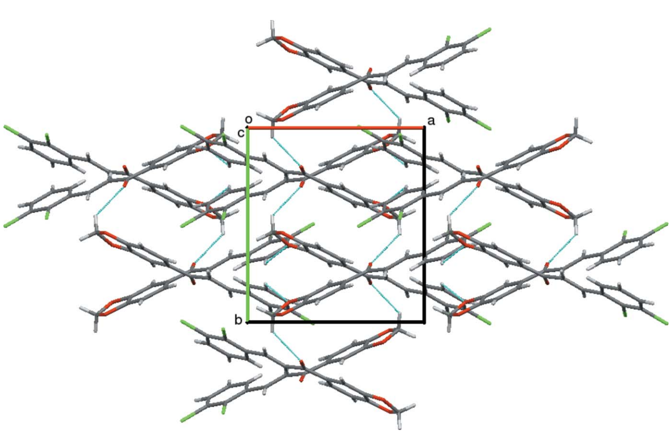


Figure 2
Packing of the molecules, viewed along the *c* axis, with hydrogen bonds shown as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{10}Cl_2O_3$
M_r	321.14
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	10.1854 (7), 11.0820 (8), 12.2077 (9)
β (°)	99.068 (3)
V (Å^3)	1360.72 (17)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	4.36
Crystal size (mm)	0.28 × 0.26 × 0.24
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.375, 0.421
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8712, 2232, 2086
R_{int}	0.048
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.586
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.158, 1.03
No. of reflections	2232
No. of parameters	190
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, −0.38

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

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**, x170103 [https://doi.org/10.1107/S2414314617001031]

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Crystal data

$C_{16}H_{10}Cl_2O_3$
M_r = 321.14
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 10.1854 (7) Å
b = 11.0820 (8) Å
c = 12.2077 (9) Å
β = 99.068 (3)°
V = 1360.72 (17) Å^3
Z = 4
$F(000)$ = 656
D_x = 1.568 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 2086 reflections
θ = 4.4–64.5°
μ = 4.36 mm^{-1}
T = 296 K
Rectangle, white
0.28 × 0.26 × 0.24 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} = 0.375, T_{max} = 0.421
8712 measured reflections
2232 independent reflections
2086 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 64.5°, θ_{min} = 4.4°
$h = -11 \rightarrow 11$
$k = -10 \rightarrow 12$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.158
S = 1.03
2232 reflections
190 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
$w = 1/[\sigma^2(F_o^2) + (0.1218P)^2 + 0.554P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.13750 (6)	0.03536 (6)	0.24874 (5)	0.0316 (3)
Cl2	−0.37087 (6)	−0.00563 (6)	0.38206 (6)	0.0377 (3)
O1	0.76711 (18)	0.39765 (18)	0.31535 (15)	0.0353 (6)
O2	0.80348 (18)	0.45912 (17)	0.49834 (15)	0.0322 (6)
O3	0.3035 (2)	0.20086 (19)	0.24062 (16)	0.0393 (7)
C1	0.8544 (3)	0.4624 (2)	0.3945 (2)	0.0304 (8)
C2	0.6590 (2)	0.3683 (2)	0.3649 (2)	0.0260 (7)
C3	0.5445 (2)	0.3131 (2)	0.3184 (2)	0.0272 (8)
C4	0.4480 (2)	0.2919 (2)	0.3881 (2)	0.0261 (7)
C5	0.4743 (3)	0.3250 (2)	0.4999 (2)	0.0273 (7)
C6	0.5927 (2)	0.3814 (2)	0.5459 (2)	0.0282 (7)
C7	0.3208 (3)	0.2367 (2)	0.3373 (2)	0.0292 (8)
C8	0.2132 (3)	0.2262 (3)	0.4053 (2)	0.0353 (8)
C9	0.1024 (3)	0.1677 (2)	0.3710 (2)	0.0287 (8)
C10	−0.0084 (2)	0.1487 (2)	0.4320 (2)	0.0252 (7)
C11	−0.1248 (2)	0.0878 (2)	0.3834 (2)	0.0255 (7)
C12	−0.2286 (2)	0.0698 (2)	0.4426 (2)	0.0273 (8)
C13	−0.2211 (3)	0.1102 (2)	0.5504 (2)	0.0311 (8)
C14	−0.1074 (3)	0.1695 (2)	0.5994 (2)	0.0311 (8)
C15	−0.0023 (3)	0.1880 (2)	0.5422 (2)	0.0284 (8)
C16	0.6823 (2)	0.4034 (2)	0.4754 (2)	0.0256 (7)
H1	0.86110	0.54520	0.37040	0.0360*
H3	0.94230	0.42640	0.40360	0.0360*
H5	0.41090	0.30880	0.54500	0.0330*
H6	0.61000	0.40300	0.62040	0.0340*
H8	0.22450	0.26250	0.47490	0.0420*
H9	0.09340	0.13470	0.30020	0.0340*
H10	0.53010	0.29020	0.24420	0.0330*
H13	−0.29130	0.09770	0.58940	0.0370*
H14	−0.10160	0.19730	0.67180	0.0370*
H15	0.07360	0.22720	0.57720	0.0340*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0261 (4)	0.0429 (5)	0.0243 (4)	−0.0032 (2)	−0.0007 (3)	−0.0032 (2)
Cl2	0.0241 (4)	0.0427 (5)	0.0458 (5)	−0.0060 (3)	0.0038 (3)	−0.0033 (3)
O1	0.0295 (10)	0.0491 (11)	0.0295 (10)	−0.0069 (8)	0.0113 (8)	−0.0014 (8)
O2	0.0233 (10)	0.0450 (11)	0.0277 (10)	−0.0076 (8)	0.0026 (8)	0.0003 (8)

O3	0.0365 (11)	0.0494 (12)	0.0314 (11)	−0.0144 (9)	0.0039 (8)	−0.0068 (9)
C1	0.0240 (13)	0.0345 (14)	0.0344 (15)	−0.0024 (11)	0.0103 (11)	0.0075 (11)
C2	0.0224 (12)	0.0284 (13)	0.0274 (12)	0.0036 (10)	0.0045 (10)	0.0040 (10)
C3	0.0279 (13)	0.0290 (13)	0.0246 (13)	0.0002 (10)	0.0042 (10)	−0.0016 (10)
C4	0.0240 (13)	0.0234 (12)	0.0303 (13)	0.0023 (10)	0.0027 (10)	0.0005 (10)
C5	0.0231 (12)	0.0290 (13)	0.0307 (13)	0.0012 (10)	0.0069 (10)	0.0051 (10)
C6	0.0273 (13)	0.0342 (13)	0.0229 (12)	0.0005 (11)	0.0037 (10)	0.0023 (10)
C7	0.0282 (14)	0.0260 (12)	0.0334 (14)	−0.0007 (10)	0.0048 (11)	0.0015 (10)
C8	0.0293 (14)	0.0390 (15)	0.0387 (15)	−0.0037 (12)	0.0092 (12)	−0.0097 (12)
C9	0.0308 (14)	0.0293 (13)	0.0245 (12)	−0.0035 (11)	−0.0004 (11)	0.0002 (10)
C10	0.0258 (13)	0.0201 (12)	0.0286 (13)	0.0010 (10)	0.0013 (10)	0.0026 (10)
C11	0.0268 (13)	0.0233 (12)	0.0245 (12)	0.0034 (10)	−0.0015 (10)	0.0008 (10)
C12	0.0218 (13)	0.0216 (12)	0.0369 (14)	0.0021 (10)	−0.0005 (11)	0.0008 (10)
C13	0.0310 (14)	0.0275 (13)	0.0365 (14)	0.0056 (11)	0.0103 (11)	0.0025 (11)
C14	0.0392 (15)	0.0249 (13)	0.0302 (13)	−0.0001 (11)	0.0086 (11)	−0.0031 (10)
C15	0.0320 (14)	0.0237 (13)	0.0280 (13)	−0.0030 (10)	−0.0001 (10)	−0.0003 (10)
C16	0.0217 (12)	0.0251 (12)	0.0286 (13)	0.0015 (10)	−0.0002 (10)	0.0043 (10)

Geometric parameters (Å, º)

Cl1—C11	1.729 (2)	C10—C11	1.411 (3)
Cl2—C12	1.734 (2)	C10—C15	1.406 (3)
O1—C1	1.403 (3)	C11—C12	1.386 (3)
O1—C2	1.376 (3)	C12—C13	1.381 (3)
O2—C1	1.444 (3)	C13—C14	1.383 (4)
O2—C16	1.369 (3)	C14—C15	1.383 (4)
O3—C7	1.231 (3)	C1—H1	0.9700
C2—C3	1.359 (3)	C1—H3	0.9700
C2—C16	1.388 (3)	C3—H10	0.9300
C3—C4	1.417 (3)	C5—H5	0.9300
C4—C5	1.398 (3)	C6—H6	0.9300
C4—C7	1.478 (4)	C8—H8	0.9300
C5—C6	1.395 (4)	C9—H9	0.9300
C6—C16	1.371 (3)	C13—H13	0.9300
C7—C8	1.480 (4)	C14—H14	0.9300
C8—C9	1.312 (4)	C15—H15	0.9300
C9—C10	1.462 (4)		
C1—O1—C2	106.40 (19)	C13—C14—C15	121.0 (2)
C1—O2—C16	105.59 (19)	C10—C15—C14	121.1 (2)
O1—C1—O2	108.4 (2)	O2—C16—C2	109.62 (19)
O1—C2—C3	128.0 (2)	O2—C16—C6	128.3 (2)
O1—C2—C16	109.56 (19)	C2—C16—C6	122.1 (2)
C3—C2—C16	122.4 (2)	O1—C1—H1	110.00
C2—C3—C4	117.0 (2)	O1—C1—H3	110.00
C3—C4—C5	119.9 (2)	O2—C1—H1	110.00
C3—C4—C7	117.5 (2)	O2—C1—H3	110.00
C5—C4—C7	122.6 (2)	H1—C1—H3	108.00

C4—C5—C6	122.1 (2)	C2—C3—H10	122.00
C5—C6—C16	116.5 (2)	C4—C3—H10	121.00
O3—C7—C4	121.3 (3)	C4—C5—H5	119.00
O3—C7—C8	120.4 (3)	C6—C5—H5	119.00
C4—C7—C8	118.4 (2)	C5—C6—H6	122.00
C7—C8—C9	122.4 (2)	C16—C6—H6	122.00
C8—C9—C10	127.1 (2)	C7—C8—H8	119.00
C9—C10—C11	121.3 (2)	C9—C8—H8	119.00
C9—C10—C15	121.4 (2)	C8—C9—H9	116.00
C11—C10—C15	117.3 (2)	C10—C9—H9	116.00
Cl1—C11—C10	119.50 (17)	C12—C13—H13	121.00
Cl1—C11—C12	119.90 (17)	C14—C13—H13	121.00
C10—C11—C12	120.6 (2)	C13—C14—H14	120.00
Cl2—C12—C11	120.14 (18)	C15—C14—H14	120.00
Cl2—C12—C13	118.65 (19)	C10—C15—H15	120.00
C11—C12—C13	121.2 (2)	C14—C15—H15	119.00
C12—C13—C14	118.9 (2)		

C2—O1—C1—O2	6.8 (2)	C5—C6—C16—O2	178.6 (2)
C1—O1—C2—C3	174.9 (2)	C5—C6—C16—C2	−2.0 (3)
C1—O1—C2—C16	−5.4 (3)	O3—C7—C8—C9	7.6 (4)
C16—O2—C1—O1	−5.7 (2)	C4—C7—C8—C9	−172.9 (3)
C1—O2—C16—C2	2.4 (2)	C7—C8—C9—C10	178.1 (2)
C1—O2—C16—C6	−178.1 (2)	C8—C9—C10—C11	177.4 (3)
O1—C2—C3—C4	179.5 (2)	C8—C9—C10—C15	−4.0 (4)
C16—C2—C3—C4	−0.2 (3)	C9—C10—C11—Cl1	−0.4 (3)
O1—C2—C16—O2	1.8 (3)	C9—C10—C11—C12	179.6 (2)
O1—C2—C16—C6	−177.7 (2)	C15—C10—C11—Cl1	−179.11 (17)
C3—C2—C16—O2	−178.5 (2)	C15—C10—C11—C12	0.9 (3)
C3—C2—C16—C6	2.0 (4)	C9—C10—C15—C14	−179.9 (2)
C2—C3—C4—C5	−1.5 (3)	C11—C10—C15—C14	−1.2 (3)
C2—C3—C4—C7	177.4 (2)	Cl1—C11—C12—Cl2	0.2 (3)
C3—C4—C5—C6	1.4 (3)	Cl1—C11—C12—C13	179.73 (18)
C7—C4—C5—C6	−177.4 (2)	C10—C11—C12—Cl2	−179.78 (17)
C3—C4—C7—O3	6.2 (3)	C10—C11—C12—C13	−0.3 (3)
C3—C4—C7—C8	−173.3 (2)	Cl2—C12—C13—C14	179.45 (18)
C5—C4—C7—O3	−175.0 (2)	C11—C12—C13—C14	−0.1 (3)
C5—C4—C7—C8	5.5 (3)	C12—C13—C14—C15	−0.3 (4)
C4—C5—C6—C16	0.3 (3)	C13—C14—C15—C10	0.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···Cl1	0.93	2.58	3.033 (3)	110
C9—H9···O3	0.93	2.48	2.809 (4)	101
C14—H14···O1^{i}	0.93	2.59	3.194 (3)	123

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