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

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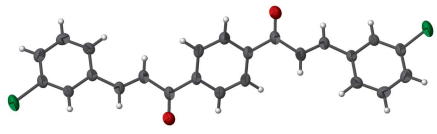
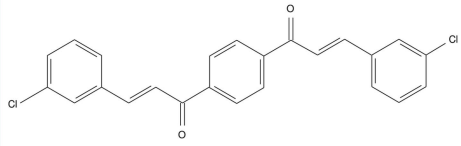
Structural data: full structural data are available from iucrdata.iucr.org

The title bis-chalcone compound, $C_{24}H_{16}Cl_2O_2$, crystallizes with one half-molecule in the asymmetric unit. The molecule has crystallographic inversion symmetry and lies about an inversion centre at the centroid of the central benzene ring. The olefinic double bonds adopt *E* configurations. The *s-trans* conformation of the central C—C bond of the enone group is confirmed by a C—C—C═C torsion angle of −162.88 (17)°.

3D view



Chemical scheme



Structure description

The title compound is a bis-chalcone and a diketone. Numerous studies have shown that bis-chalcones possess multiple pharmacological properties (Nowakowska, 2007). Crystalline chalcone derivatives are also of interest due to their second and third harmonic generation properties (Chidan *et al.*, 2015). The optical properties of the molecules are also associated with their molecular geometry (Kumar *et al.* 2013) and, as a part of our ongoing work on such molecules (Naveen *et al.* 2017), we report here the crystal structure of the title compound.

The title compound crystallizes with one half-molecule in the asymmetric unit and its structure is shown in Fig. 1. The molecule has crystallographic inversion symmetry and lies about an inversion centre at the centroid of the central benzene ring. The olefinic double bond adopts an *E* configuration. The *s-trans* conformation of the central C—C bond of the enone group is confirmed by the C10—C9—C8═C7 torsion angle of

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{16}Cl_2O_2$
M_r	407.27
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	294
a, b, c (Å)	22.6336 (13), 7.0895 (4), 5.9515 (3)
β (°)	95.485 (2)
V (Å^3)	950.61 (9)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.36
Crystal size (mm)	0.44 × 0.26 × 0.14
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Multi-scan (*NUMABS*; Rigaku, 1999)
T_{min}, T_{max}	0.859, 0.951
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	27758, 3656, 2542
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.772
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.166, 1.04
No. of reflections	3656
No. of parameters	127
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.21

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

−162.88 (17)°. This value is less than that reported for the related compound 2,5-bis(4-chlorobenzylidene)cyclopentanone (Samshuddin *et al.*, 2016).

Synthesis and crystallization

1,4-Diacetylbenzene (1.62 g, 0.01 mol) was mixed with 3-chlorobenzaldehyde (2.80 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 finally recrystallized from methanol (95%) to give the pure bis-chalcone. Single crystals suitable for X-ray diffraction studies were grown by slow evaporation of an acetone-methanol (1:1) solution (m.p. 413–415 K).

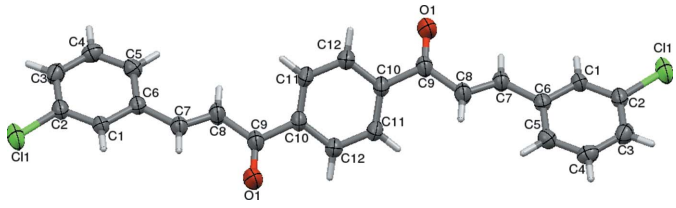


Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. Labelled atoms are related to unlabelled atoms by the symmetry operation $-x + 1, -y, -z + 1$.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

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References

Chidan Kumar, C. S., Balachandran, V., Fun, H. K., Chandraju, S. & Quah, C. K. (2015). *J. Mol. Struct.* **1100**, 299–310.
Kumar, C. S. C., Loh, W. S., Ooi, C. W., Quah, C. K. & Fun, H. K. (2013). *Molecules*, **18**, 11996–12011.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Naveen, S., Dileep Kumar, A., Lokeshwari, M., Ajay Kumar, K., Lokanath, N. K. & Warad, I. (2017). *IUCrData*, **2**, x170126.
Nowakowska, Z. (2007). *Eur. J. Med. Chem.* **42**, 125–137.
Rigaku. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
Rigaku. (2011). *CrystalClear SM Expert*. Rigaku Corporation, Tokyo, Japan.
Samshuddin, S., Naveen, S., Ananya, L. N., Vishwanatha, P., Lokanath, N. K. & Abdoh, M. (2016). *IUCrData*, **1**, x161865.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

full crystallographic data

IUCrData (2017). **2**, x170212 [https://doi.org/10.1107/S2414314617002127]

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Crystal data

$C_{24}H_{16}Cl_2O_2$
M_r = 407.27
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 22.6336 (13) Å
b = 7.0895 (4) Å
c = 5.9515 (3) Å
β = 95.485 (2)°
V = 950.61 (9) Å^3
Z = 2
$F(000)$ = 420
D_x = 1.423 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2542 reflections
θ = 2.7–33.3°
μ = 0.36 mm^{-1}
T = 294 K
Rectangle, white
0.44 × 0.26 × 0.14 mm

Data collection

Rigaku Saturn724+ diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 18.4 pixels mm^{-1}
profile data from ω–scans
Absorption correction: multi-scan (NUMABS; Rigaku, 1999)
T_{min} = 0.859, T_{max} = 0.951
27758 measured reflections
3656 independent reflections
2542 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 33.3°, θ_{min} = 2.7°
h = −34→34
k = −10→10
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.166
S = 1.04
3656 reflections
127 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
$w = 1/[\sigma^2(F_o^2) + (0.0803P)^2 + 0.2606P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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	0.04854 (2)	0.08183 (9)	0.31155 (9)	0.0694 (2)
O1	0.36614 (6)	0.0056 (3)	0.7913 (2)	0.0676 (6)
C1	0.16738 (6)	0.0708 (2)	0.3479 (2)	0.0351 (4)
C2	0.11601 (6)	0.0359 (2)	0.2094 (3)	0.0404 (4)
C3	0.11768 (7)	−0.0401 (2)	−0.0040 (3)	0.0447 (5)
C4	0.17253 (8)	−0.0790 (2)	−0.0789 (3)	0.0428 (5)
C5	0.22454 (7)	−0.0476 (2)	0.0578 (3)	0.0399 (4)
C6	0.22246 (6)	0.0251 (2)	0.2750 (2)	0.0343 (4)
C7	0.27480 (6)	0.0426 (2)	0.4386 (3)	0.0390 (4)
C8	0.32993 (6)	−0.0063 (3)	0.4068 (3)	0.0446 (5)
C9	0.37737 (6)	−0.0004 (3)	0.5956 (3)	0.0427 (4)
C10	0.44036 (6)	−0.0027 (2)	0.5403 (2)	0.0368 (4)
C11	0.45674 (6)	0.0564 (2)	0.3326 (3)	0.0399 (4)
C12	0.51588 (6)	0.0598 (2)	0.2924 (3)	0.0402 (4)
H1A	0.16530	0.12460	0.48940	0.0420*
H3A	0.08280	−0.06450	−0.09520	0.0540*
H4A	0.17440	−0.12700	−0.22350	0.0510*
H5A	0.26100	−0.07490	0.00500	0.0480*
H7A	0.26880	0.09300	0.57880	0.0470*
H8A	0.33880	−0.04460	0.26450	0.0540*
H11A	0.42780	0.09370	0.22000	0.0480*
H12A	0.52660	0.10040	0.15330	0.0480*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0279 (2)	0.1097 (5)	0.0716 (3)	0.0012 (2)	0.0101 (2)	−0.0044 (3)
O1	0.0364 (6)	0.1204 (14)	0.0466 (7)	0.0026 (7)	0.0066 (5)	0.0003 (8)
C1	0.0278 (6)	0.0424 (7)	0.0354 (6)	0.0011 (5)	0.0044 (5)	−0.0001 (5)
C2	0.0273 (6)	0.0482 (8)	0.0456 (8)	−0.0009 (5)	0.0038 (5)	0.0042 (6)
C3	0.0387 (8)	0.0496 (9)	0.0440 (8)	−0.0044 (6)	−0.0055 (6)	0.0008 (6)
C4	0.0496 (9)	0.0444 (8)	0.0340 (7)	0.0013 (6)	0.0014 (6)	−0.0025 (6)
C5	0.0364 (7)	0.0446 (8)	0.0396 (7)	0.0032 (6)	0.0082 (5)	−0.0006 (6)
C6	0.0280 (6)	0.0378 (7)	0.0371 (6)	−0.0001 (5)	0.0035 (5)	0.0012 (5)
C7	0.0289 (6)	0.0471 (8)	0.0407 (7)	0.0004 (5)	0.0023 (5)	−0.0023 (6)
C8	0.0275 (6)	0.0599 (10)	0.0463 (8)	0.0002 (6)	0.0025 (5)	−0.0070 (7)
C9	0.0271 (6)	0.0542 (9)	0.0467 (8)	−0.0004 (6)	0.0029 (5)	−0.0019 (7)
C10	0.0249 (6)	0.0423 (7)	0.0425 (7)	0.0000 (5)	0.0001 (5)	−0.0021 (6)
C11	0.0276 (6)	0.0482 (8)	0.0426 (7)	0.0025 (5)	−0.0035 (5)	0.0046 (6)

C12	0.0312 (6)	0.0505 (8)	0.0385 (7)	−0.0006 (6)	0.0010 (5)	0.0028 (6)

Geometric parameters (Å, º)

Cl1—C2	1.7277 (15)	C10—C11	1.389 (2)
O1—C9	1.216 (2)	C10—C12^{i}	1.396 (2)
C1—C2	1.382 (2)	C11—C12	1.382 (2)
C1—C6	1.3967 (19)	C1—H1A	0.9300
C2—C3	1.384 (2)	C3—H3A	0.9300
C3—C4	1.386 (2)	C4—H4A	0.9300
C4—C5	1.384 (2)	C5—H5A	0.9300
C5—C6	1.397 (2)	C7—H7A	0.9300
C6—C7	1.465 (2)	C8—H8A	0.9300
C7—C8	1.326 (2)	C11—H11A	0.9300
C8—C9	1.479 (2)	C12—H12A	0.9300
C9—C10	1.4936 (19)		
C2—C1—C6	119.89 (12)	C10—C11—C12	120.31 (15)
Cl1—C2—C1	118.55 (12)	C10^{i}—C12—C11	120.29 (15)
Cl1—C2—C3	119.94 (12)	C2—C1—H1A	120.00
C1—C2—C3	121.47 (13)	C6—C1—H1A	120.00
C2—C3—C4	118.46 (15)	C2—C3—H3A	121.00
C3—C4—C5	121.11 (16)	C4—C3—H3A	121.00
C4—C5—C6	120.11 (15)	C3—C4—H4A	119.00
C1—C6—C5	118.89 (12)	C5—C4—H4A	119.00
C1—C6—C7	117.58 (12)	C4—C5—H5A	120.00
C5—C6—C7	123.35 (13)	C6—C5—H5A	120.00
C6—C7—C8	126.53 (16)	C6—C7—H7A	117.00
C7—C8—C9	120.60 (16)	C8—C7—H7A	117.00
O1—C9—C8	121.71 (14)	C7—C8—H8A	120.00
O1—C9—C10	120.17 (14)	C9—C8—H8A	120.00
C8—C9—C10	118.12 (14)	C10—C11—H11A	120.00
C9—C10—C11	122.31 (13)	C12—C11—H11A	120.00
C9—C10—C12^{i}	118.24 (13)	C11—C12—H12A	120.00
C11—C10—C12^{i}	119.40 (13)	C10^{i}—C12—H12A	120.00
C6—C1—C2—Cl1	176.37 (11)	C6—C7—C8—C9	−173.15 (16)
C6—C1—C2—C3	−1.5 (2)	C7—C8—C9—O1	17.4 (3)
C2—C1—C6—C5	2.9 (2)	C7—C8—C9—C10	−162.88 (17)
C2—C1—C6—C7	−172.42 (13)	O1—C9—C10—C11	−156.71 (19)
Cl1—C2—C3—C4	−178.63 (12)	O1—C9—C10—C12^{i}	20.8 (3)
C1—C2—C3—C4	−0.8 (2)	C8—C9—C10—C11	23.5 (3)
C2—C3—C4—C5	1.7 (2)	C8—C9—C10—C12^{i}	−158.96 (16)
C3—C4—C5—C6	−0.2 (2)	C9—C10—C11—C12	177.09 (15)
C4—C5—C6—C1	−2.1 (2)	C12^{i}—C10—C11—C12	−0.4 (2)
C4—C5—C6—C7	172.97 (14)	C9—C10—C12^{i}—C11^{i}	−177.19 (15)

C1—C6—C7—C8	174.96 (17)	C11—C10—C12^{i}—C11^{i}	0.4 (2)
C5—C6—C7—C8	−0.1 (2)	C10—C11—C12—C10^{i}	0.4 (2)

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