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2,5-Bis(4-chlorobenzylidene)cyclopentanone

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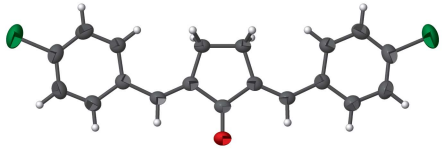
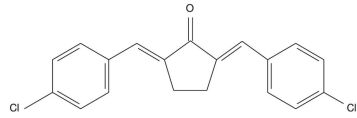
Structural data: full structural data are available from iucrdata.iucr.org

The title bis-chalcone compound, $C_{19}H_{14}Cl_2O$, crystallizes with one half-molecule in the asymmetric unit. The molecule has crystallographic mirror symmetry with the C=O bond on the mirror plane. The molecule adopts an *E* configuration about the central olefinic bonds. In the crystal, molecules are linked *via* weak C—H···O hydrogen bonds, forming supramolecular chains propagating along the [100] direction.

3D view



Chemical scheme



Structure description

The development of highly efficient non-linear optical crystals is extremely important for laser spectroscopy and laser processing. Bis(arylmethylidene) cycloalkanones have been reported to exhibit promising non-linear optical properties (Yu *et al.*, 2000). In addition, these compounds are widely used as precursors for the synthesis of biologically active heterocycles (Guilford *et al.*, 1999). In view of the importance of bis-chalcones, we report herein on the synthesis and crystal structure of title compound.

The molecular structure of the title compound is shown in Fig. 1. The molecule has crystallographic mirror symmetry and adopts an *E* configuration about the central olefinic bonds, exhibiting a butterfly-shaped geometry. In the crystal, the molecules are linked *via* weak C—H···O hydrogen bonds (Table 1), forming supramolecular chains propagating along the [100] direction.

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H5···O1^{i}	0.97	2.54	3.270 (3)	132

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

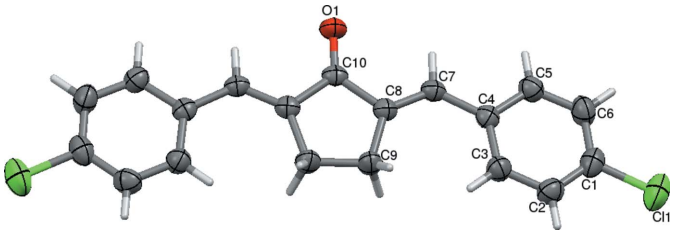


Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. Unlabelled atoms are generated by the symmetry operation $x, \frac{3}{2}-y, z$.

Synthesis and crystallization

A mixture of cyclopentanone (0.84 g, 0.01 mol) and 4-chlorobenzaldehyde (2.80 g, 0.02 mol) in 30 ml ethanolic sodium hydroxide (0.1 mol) was stirred at 278–283 K for 3 h. The precipitate formed was collected by filtration and purified by recrystallization from ethanol solution. Single crystals were grown from DMF in 86% yield by slow evaporation.

Refinement

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

Acknowledgements

The authors thank Alva's Education Foundation, Moodbidri, for proving research facilities.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{14}Cl_2O$
M_r	329.20
Crystal system, space group	Orthorhombic, *Pnma*
Temperature (K)	296
a, b, c (Å)	6.1029 (4), 35.7084 (18), 7.2217 (6)
V (Å^3)	1573.79 (18)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.41
Crystal size (mm)	0.29 × 0.27 × 0.25
Data collection	
Diffractometer	Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2011)
T_{min}, T_{max}	0.888, 0.902
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6328, 1583, 1063
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.619
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.111, 1.04
No. of reflections	1583
No. of parameters	103
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.20

Computer programs: *APEX2* and *SAINT* (Bruker, 2011), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

References

Bruker (2011). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Guilford, W. J., Shaw, K. J., Dallas, J. L., Koovakkat, S., Lee, W., Liang, A., Light, D. R., McCarrick, M. A., Whitlow, M., Ye, B. & Morrissey, M. M. (1999). *J. Med. Chem.* **42**, 5415–5425.
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.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yu, R. C., Yakimansky, A. V., Kothe, H., Voigt-Martin, I. G., Schollmeyer, D., Jansen, J., Zandbergen, H. & Tenkovtsev, A. V. (2000). *Acta Cryst.* A**56**, 436–450.

full crystallographic data

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2,5-Bis(4-chlorobenzylidene)cyclopentanone

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(2*E*,5*E*)-2,5-Bis(4-chlorobenzylidene)cyclopentanone

Crystal data

$C_{19}H_{14}Cl_2O$
M_r = 329.20
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
a = 6.1029 (4) Å
b = 35.7084 (18) Å
c = 7.2217 (6) Å
V = 1573.79 (18) Å^3
Z = 4
F(000) = 680
D_x = 1.389 Mg m^{-3}
Melting point: 432 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1063 reflections
θ = 2.9–26.1°
μ = 0.41 mm^{-1}
T = 296 K
Rectangle, colorless
0.29 × 0.27 × 0.25 mm

Data collection

Bruker APEXII diffractometer
Radiation source: Enraf Nonius FR590
Graphite monochromator
Detector resolution: 18.4 pixels mm^{-1}
CCD rotation images, thick slices scans
Absorption correction: multi-scan (SADABS; Bruker, 2011)
T_{min} = 0.888, T_{max} = 0.902
6328 measured reflections
1583 independent reflections
1063 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 26.1°, θ_{min} = 2.9°
h = −7→5
k = −43→42
l = −4→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.111
S = 1.04
1583 reflections
103 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
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.4484P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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	0.78079 (13)	0.52896 (2)	1.04515 (13)	0.0841 (4)
O1	0.1615 (3)	0.75000	0.8758 (3)	0.0438 (7)
C1	0.6686 (4)	0.57327 (6)	1.0207 (3)	0.0482 (8)
C2	0.7870 (4)	0.60368 (6)	1.0815 (3)	0.0475 (8)
C3	0.7013 (3)	0.63916 (6)	1.0602 (3)	0.0425 (8)
C4	0.4964 (3)	0.64510 (5)	0.9785 (3)	0.0347 (7)
C5	0.3827 (4)	0.61340 (5)	0.9195 (3)	0.0422 (8)
C6	0.4653 (4)	0.57775 (6)	0.9402 (3)	0.0509 (9)
C7	0.3980 (3)	0.68207 (5)	0.9492 (3)	0.0341 (7)
C8	0.4771 (3)	0.71622 (5)	0.9844 (3)	0.0324 (6)
C9	0.6917 (3)	0.72845 (5)	1.0656 (3)	0.0411 (8)
C10	0.3465 (4)	0.75000	0.9390 (4)	0.0318 (9)
H1	0.78180	0.65970	1.10110	0.0510*
H2	0.38550	0.55710	0.90050	0.0610*
H3	0.24620	0.61640	0.86410	0.0510*
H4	0.25820	0.68170	0.89810	0.0410*
H5	0.81240	0.71900	0.99180	0.0490*
H7	0.92350	0.60030	1.13630	0.0570*
H8	0.70690	0.71900	1.19090	0.0490*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0893 (6)	0.0438 (4)	0.1191 (8)	0.0186 (3)	−0.0081 (5)	0.0048 (4)
O1	0.0301 (10)	0.0461 (12)	0.0551 (16)	0.0000	−0.0091 (10)	0.0000
C1	0.0577 (14)	0.0395 (12)	0.0474 (16)	0.0083 (11)	0.0044 (13)	0.0044 (10)
C2	0.0426 (12)	0.0511 (14)	0.0489 (17)	0.0045 (11)	−0.0060 (11)	0.0071 (11)
C3	0.0422 (12)	0.0399 (12)	0.0454 (16)	−0.0008 (10)	−0.0064 (11)	0.0028 (10)
C4	0.0356 (10)	0.0376 (11)	0.0308 (13)	−0.0018 (9)	0.0028 (10)	0.0018 (9)
C5	0.0413 (11)	0.0429 (13)	0.0425 (16)	−0.0042 (9)	−0.0035 (11)	−0.0005 (10)
C6	0.0593 (15)	0.0366 (12)	0.0567 (18)	−0.0069 (11)	−0.0052 (13)	−0.0023 (11)
C7	0.0293 (10)	0.0406 (12)	0.0323 (14)	−0.0015 (9)	−0.0019 (9)	0.0012 (9)
C8	0.0313 (10)	0.0388 (11)	0.0272 (12)	0.0001 (9)	0.0010 (9)	0.0027 (9)
C9	0.0364 (11)	0.0400 (12)	0.0469 (16)	−0.0004 (9)	−0.0127 (11)	0.0026 (10)
C10	0.0272 (14)	0.0429 (16)	0.0254 (18)	0.0000	0.0018 (13)	0.0000

Geometric parameters (Å, º)

Cl1—C1	1.733 (2)	C8—C9	1.500 (3)
O1—C10	1.218 (3)	C8—C10	1.483 (2)

C1—C2	1.376 (3)	C9—C9^{i}	1.539 (3)
C1—C6	1.380 (3)	C2—H7	0.9300
C2—C3	1.379 (3)	C3—H1	0.9300
C3—C4	1.399 (3)	C5—H3	0.9300
C4—C5	1.394 (3)	C6—H2	0.9300
C4—C7	1.466 (3)	C7—H4	0.9300
C5—C6	1.377 (3)	C9—H5	0.9700
C7—C8	1.336 (3)	C9—H8	0.9700
Cl1—C1—C2	118.72 (18)	C8—C10—C8^{i}	108.91 (19)
Cl1—C1—C6	120.27 (17)	C1—C2—H7	120.00
C2—C1—C6	121.0 (2)	C3—C2—H7	120.00
C1—C2—C3	119.4 (2)	C2—C3—H1	119.00
C2—C3—C4	121.69 (19)	C4—C3—H1	119.00
C3—C4—C5	116.79 (18)	C4—C5—H3	119.00
C3—C4—C7	124.32 (17)	C6—C5—H3	119.00
C5—C4—C7	118.89 (18)	C1—C6—H2	121.00
C4—C5—C6	122.3 (2)	C5—C6—H2	121.00
C1—C6—C5	118.8 (2)	C4—C7—H4	115.00
C4—C7—C8	130.29 (18)	C8—C7—H4	115.00
C7—C8—C9	130.97 (17)	C8—C9—H5	110.00
C7—C8—C10	120.42 (18)	C8—C9—H8	110.00
C9—C8—C10	108.60 (15)	H5—C9—H8	109.00
C8—C9—C9^{i}	106.93 (15)	C9^{i}—C9—H5	110.00
O1—C10—C8	125.55 (11)	C9^{i}—C9—H8	110.00
O1—C10—C8^{i}	125.55 (11)		
Cl1—C1—C2—C3	178.96 (17)	C4—C5—C6—C1	−0.6 (3)
C6—C1—C2—C3	−0.5 (3)	C4—C7—C8—C9	−0.1 (4)
Cl1—C1—C6—C5	−178.73 (18)	C4—C7—C8—C10	178.9 (2)
C2—C1—C6—C5	0.7 (3)	C7—C8—C9—C9^{i}	178.0 (2)
C1—C2—C3—C4	0.1 (3)	C10—C8—C9—C9^{i}	−1.1 (2)
C2—C3—C4—C5	0.0 (3)	C7—C8—C10—O1	3.0 (4)
C2—C3—C4—C7	−179.0 (2)	C7—C8—C10—C8^{i}	−177.4 (2)
C3—C4—C5—C6	0.2 (3)	C9—C8—C10—O1	−177.8 (3)
C7—C4—C5—C6	179.3 (2)	C9—C8—C10—C8^{i}	1.8 (3)
C3—C4—C7—C8	2.5 (4)	C8—C9—C9^{i}—C8^{i}	0.0 (2)
C5—C4—C7—C8	−176.4 (2)		

Symmetry code: (i) x, $-y+3/2$, z.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H5···O1^{ii}	0.97	2.54	3.270 (3)	132

Symmetry code: (ii) $x+1$, y, z.