organic compounds

(1*E*,4*E*)-1,5-Bis(2-chlorophenyl)penta-1,4-dien-3-one

M. Nizam Mohideen,[a] S. Thenmozhi,[b] A. Subbiah Pandi,[b]* Ramalingam Murugan[c] and S. Sriman Narayanan[c]

[a]Department of Physics, The New College, Chennai 600 014, India, [b]Department of Physics, Presidency College (Autonomous), Chennai 600 005, India, and [c]Department of Analytical Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

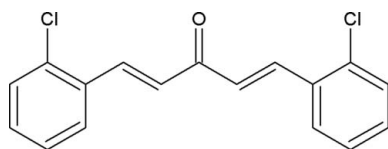Correspondence e-mail: a_spandian@yahoo.com

The title compound, $C_{17}H_{12}Cl_2O$, crystallizes in a centrosymmetric space group. The dihedral angle between the two benzene rings is 24.7 (1)°. The crystal packing is characterized by non-classical C—H···O and C—H···Cl inter- and intramolecular hydrogen bonds.

Related literature

For related literature, see: Allen *et al.* (1987); Botek *et al.* (2004); Butcher *et al.* (2006); Fichou *et al.* (1988); Go *et al.* (2005); Gould *et al.* (1995); Indira *et al.* (2002); Ravishankar *et al.* (2005); Uchida *et al.* (1998).

Experimental

Crystal data

$C_{17}H_{12}Cl_2O$	V = 2972.69 (14) Å³
M_r = 303.17	Z = 8
Orthorhombic, *Pbca*	Mo *K*α radiation
a = 14.2816 (4) Å	μ = 0.43 mm⁻¹
b = 8.3056 (2) Å	T = 293 (2) K
c = 25.0612 (7) Å	0.28 × 0.25 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	4611 independent reflections
Absorption correction: none	2450 reflections with $I > 2\sigma(I)$
22033 measured reflections	R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049	182 parameters
$wR(F^2)$ = 0.164	H-atom parameters constrained
S = 1.00	$\Delta\rho_{max}$ = 0.47 e Å⁻³
4611 reflections	$\Delta\rho_{min}$ = −0.53 e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O[i]	0.93	2.55	3.283 (3)	136
C16—H16···O[ii]	0.93	2.56	3.416 (3)	152
C7—H7···Cl1	0.93	2.62	3.040 (2)	108
C11—H11···Cl2	0.93	2.63	3.050 (2)	108

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: ʼ*SHELXL97* and *PLATON* (Spek, 2003).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Botek, E., Champagne, B., Turki, M. & André, J.-M. (2004). *J. Chem. Phys.* **120**, 2042–2048.

Bruker (2004). *APEX2* (Version 2.0-2) and *SAINT*. Bruker AXS Inc., Madison Wisconsin, USA.

Butcher, R. J., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Mithun, A. (2006). *Acta Cryst.* E**62**, o1629–o1630.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Fichou, D., Watanabe, T., Takeda, T., Miyata, S., Goto, Y. & Nakayama, M. (1988). *Jpn J. Appl. Phys.* **27**, 429–430.

Go, M. L., Wu, X. & Liu, X. L. (2005). *Curr. Med. Chem.* **12**, 483–499.

Gould, B. S., Panneerelvam, K., Zacharias, D. E. & Desiraju, G. R. (1995). *J. Chem. Soc. Perkin Trans. 2*, pp. 325–330.

Indira, J., Karat, P. P. & Sarojini, B. K. (2002). *J. Cryst. Growth*, **242**, 209–214.

Ravishankar, T., Chinnakali, K., Nanjundan, S., Selvam, P., Fun, H. K. & Yu, X.-L. (2005). *Acta Cryst.* E**61**, o405–o407.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Uchida, T., Kozawa, K., Sakai, T., Aoki, M., Yoguchi, H., Abduryim, A. & Watanabe, Y. (1998). *Mol. Cryst. Liq. Cryst.* **315**, 135–140.

supplementary materials

(1*E*,4*E*)-1,5-Bis(2-chlorophenyl)penta-1,4-dien-3-one

M. Nizam Mohideen, S. Thenmozhi, A. Subbiah Pandi, R. Murugan and **S. S. Narayanan**

Comment

Chalcones with the general formula Ar—CH=CH—CO—Ar are an important class of compounds, with the common structural entity being the central –CH=CH—C(=O)- group, in which the H atoms can be substituted. The –C=C– double bond can be photoreactive and can produce various products through solid-state photocycloaddition. Therefore, chalcones are widely used in organic solid-state photochemistry (Gould *et al.*, 1995). Reviews on the bioactivities of various chalcones have been reported (Go *et al.*, 2005). Recently, it has been noted that, among many organic compounds reported for their second harmonic generation, chalcone derivatives are known for their excellent blue light transmittance and good crystallizability (Fichou *et al.*, 1988). They provide a necessary configuration for NLO activity, with two planar rings connected through a conjugated double bond (Indira *et al.*, 2002). They are also inherently chiral owing to the fact that the two phenyl rings are mutually twisted with respect to the linking backbone (Butcher *et al.*, 2006). This helicity has also been shown to lead to NLO activity (Botek *et al.*, 2004). Bis-chalcones are also found to exhibit good NLO properties (Uchida *et al.*, 1998). In order to obtain detailed information on the molecular conformation of the chlorophenyl-substituted chalcone (I) in the solid state, its X-ray crystal structure was determined.

The bond lengths and angles in the title compound (Fig. 1) lie whithin normal ranges (Allen *et al.*, 1987). The enone group and the two benzene rings of the chalcone are individually planar, with a maximum deviation of −0.005 (3), −0.007 (2) 0.051 (2) Å for atoms C4, C17 and C9, from the planes C1—C6, C12—C17 and O/C7—C11, respectively. The dihedral angle between the least-squares planes of the two benzene rings is 24.7 (1)°. The least-squaes plane through the enone unit (C7—C11/O) makes dihedral angles of 15.4 (1) and 11.4 (1)° with the C1—C6 and C12—C17 benzene rings, respectively. The five-C-atom backbone shows alternating double and single bonds [C7—C8 = 1.310 (3) Å, C8—C9 = 1.463 (3) Å, C9—C10 = 1.470 (3) Å, C10—C11 = 1.322 (3) Å and C11—C12 = 1.457 (3) Å] and thus gives no indication of conjugation. The most commonly cited factors found in organic molecules possessing NLO properties are the presence of donor-acceptor substituents on a conjugated backbone. Since this molecule does not possess either of these factors the helicity of the molecule must be the origin of the observed NLO effects.

Atoms Cl1 and Cl2 deivate by 0.027 (4) and 0.022 (1) Å, respectively, from the plane of the attached benzene rings. The widening of the C11—C12—C13 angle to 121.7 (2)° and C10—C11—C12 to 126.4 (2)° can be ascribed to the short interatomic contact between atoms H13 and H10 (2.15 Å). In addition, the strain induced by the short H8—H5 (2.19 Å) contact results in a slight opening of the C5—C6—C7 angle to 121.7 (2)° and similar to those observed in other comparable structures (Ravishankar *et al.*, 2005).

The crystal packing is characterized by non-classical C—H···O and C—H···Cl inter and intramolecular hydrogen bonds (Table 2). Atoms C8 and C16 in the molecule at (*x*, *y*, *z*) act as hydrogen donors to O in the molecule at (−*x* + 1/2, *y* + 1/2, *z*) and to O with molecule at (−*x* + 1, *y* + 1/2, −*z* − 1/2), respectively. The chain is formed between symmetry related molecules through C8—H8···O and C16—H16···O hydrogen bonds.

Experimental

1 mol (5 ml) of acetone was taken in 50 ml me thanol and then 2 mol (25 ml) of chloro benzaldehyde was added. To this solution 50 ml of 5 mol (10 g in 50 ml water) sodium hydroxide solution was added at 273 K. This mixture was stirred over night and the product was filtered. Single crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of a solution in ethylacetate.

Refinement

All H atoms were fixed geometrically and allowed to ride on their parent C atoms, with C—H distances of 0.93 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.
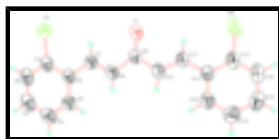
Figures

Fig. 1. The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level.

(1E,4E)-1,5-Bis(2-chlorophenyl)penta-1,4-dien-3-one

Crystal data

$C_{17}H_{12}Cl_2O$	$Z = 8$
$M_r = 303.17$	$F_{000} = 1248$
Orthorhombic, *Pbca*	$D_x = 1.355$ Mg m^{-3}
Hall symbol: -P 2ac 2ab	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 14.2816$ (4) Å	$\mu = 0.43$ mm^{-1}
$b = 8.3056$ (2) Å	$T = 293$ (2) K
$c = 25.0612$ (7) Å	Block, colourless
$V = 2972.69$ (14) Å^3	$0.28 \times 0.25 \times 0.22$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2450 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.038$
Monochromator: graphite	$\theta_{max} = 30.7°$
$T = 293(2)$ K	$\theta_{min} = 2.2°$
ω and π scans	$h = -18 \rightarrow 20$
Absorption correction: none	$k = -10 \rightarrow 11$
22033 measured reflections	$l = -35 \rightarrow 36$
4611 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 0.9731P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.164$	$(\Delta/\sigma)_{max} = 0.011$
$S = 1.00$	$\Delta\rho_{max} = 0.47$ e Å^{-3}
4611 reflections	$\Delta\rho_{min} = -0.53$ e Å^{-3}
182 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0030 (8)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) *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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Cl1	0.19158 (5)	0.33914 (8)	0.05290 (3)	0.0752 (2)
Cl2	0.53509 (5)	0.68564 (13)	−0.27210 (4)	0.1094 (4)
O	0.34751 (11)	0.5172 (2)	−0.11420 (6)	0.0648 (4)
C1	0.12252 (15)	0.5095 (3)	0.04574 (8)	0.0516 (5)
C2	0.05863 (18)	0.5433 (3)	0.08563 (10)	0.0654 (6)
H2	0.0541	0.4765	0.1153	0.078*
C3	0.00218 (19)	0.6754 (3)	0.08120 (11)	0.0707 (7)
H3	−0.0406	0.6991	0.1081	0.085*
C4	0.00836 (18)	0.7731 (3)	0.03728 (11)	0.0672 (6)
H4	−0.0308	0.8620	0.0341	0.081*
C5	0.07240 (16)	0.7397 (3)	−0.00203 (10)	0.0590 (5)
H5	0.0764	0.8078	−0.0314	0.071*
C6	0.13174 (14)	0.6063 (2)	0.00091 (8)	0.0462 (4)
C7	0.20176 (14)	0.5733 (2)	−0.04012 (8)	0.0484 (5)
H7	0.2463	0.4952	−0.0322	0.058*
C8	0.20827 (15)	0.6427 (3)	−0.08693 (9)	0.0521 (5)

H8	0.1618	0.7151	−0.0969	0.063*
C9	0.28492 (14)	0.6120 (2)	−0.12432 (8)	0.0470 (4)
C10	0.28378 (15)	0.7056 (3)	−0.17410 (8)	0.0515 (5)
H10	0.2327	0.7720	−0.1808	0.062*
C11	0.35188 (15)	0.6998 (2)	−0.20975 (8)	0.0496 (5)
H11	0.4015	0.6305	−0.2025	0.059*
C12	0.35691 (14)	0.7913 (2)	−0.25926 (8)	0.0475 (4)
C13	0.28148 (18)	0.8818 (3)	−0.27774 (9)	0.0624 (6)
H13	0.2261	0.8818	−0.2582	0.075*
C14	0.2859 (2)	0.9710 (3)	−0.32385 (10)	0.0711 (7)
H14	0.2341	1.0296	−0.3352	0.085*
C15	0.3674 (2)	0.9731 (3)	−0.35301 (10)	0.0709 (7)
H15	0.3711	1.0343	−0.3840	0.085*
C16	0.44293 (19)	0.8857 (3)	−0.33661 (10)	0.0703 (7)
H16	0.4980	0.8871	−0.3564	0.084*
C17	0.43749 (16)	0.7952 (3)	−0.29065 (9)	0.0578 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0917 (5)	0.0741 (4)	0.0596 (4)	0.0182 (3)	0.0139 (3)	0.0108 (3)
Cl2	0.0541 (4)	0.1692 (9)	0.1050 (7)	0.0231 (5)	0.0262 (4)	0.0501 (6)
O	0.0577 (10)	0.0728 (10)	0.0640 (10)	0.0112 (8)	0.0196 (8)	0.0131 (8)
C1	0.0510 (12)	0.0584 (11)	0.0453 (11)	−0.0054 (9)	0.0057 (9)	−0.0072 (8)
C2	0.0693 (15)	0.0781 (15)	0.0489 (12)	−0.0078 (12)	0.0187 (11)	−0.0046 (11)
C3	0.0610 (14)	0.0860 (17)	0.0652 (16)	−0.0031 (13)	0.0208 (13)	−0.0230 (13)
C4	0.0583 (14)	0.0683 (14)	0.0752 (16)	0.0071 (11)	0.0113 (13)	−0.0175 (12)
C5	0.0575 (13)	0.0581 (12)	0.0615 (13)	0.0019 (10)	0.0091 (11)	−0.0047 (10)
C6	0.0436 (10)	0.0513 (10)	0.0436 (10)	−0.0047 (8)	0.0053 (9)	−0.0078 (8)
C7	0.0475 (11)	0.0486 (10)	0.0492 (11)	−0.0009 (8)	0.0073 (9)	−0.0030 (8)
C8	0.0455 (11)	0.0614 (12)	0.0493 (11)	0.0038 (9)	0.0095 (9)	0.0042 (9)
C9	0.0428 (11)	0.0512 (10)	0.0470 (11)	−0.0066 (9)	0.0050 (9)	0.0001 (8)
C10	0.0440 (11)	0.0645 (12)	0.0458 (11)	−0.0012 (9)	0.0043 (9)	0.0018 (9)
C11	0.0451 (11)	0.0560 (11)	0.0475 (11)	−0.0044 (9)	0.0035 (9)	0.0018 (8)
C12	0.0494 (11)	0.0515 (10)	0.0416 (10)	−0.0082 (9)	0.0036 (9)	−0.0046 (8)
C13	0.0594 (14)	0.0776 (14)	0.0502 (12)	0.0061 (12)	0.0113 (11)	0.0018 (10)
C14	0.0816 (19)	0.0757 (15)	0.0561 (14)	0.0101 (13)	0.0011 (13)	0.0078 (11)
C15	0.093 (2)	0.0719 (15)	0.0474 (13)	−0.0124 (14)	0.0050 (14)	0.0074 (11)
C16	0.0673 (16)	0.0889 (17)	0.0546 (14)	−0.0196 (14)	0.0173 (12)	0.0047 (12)
C17	0.0487 (12)	0.0711 (13)	0.0538 (13)	−0.0100 (10)	0.0068 (10)	0.0015 (10)

Geometric parameters (Å, °)

Cl1—C1	1.734 (2)	C8—H8	0.9300
Cl2—C17	1.729 (3)	C9—C10	1.470 (3)
O—C9	1.218 (2)	C10—C11	1.322 (3)
C1—C2	1.382 (3)	C10—H10	0.9300
C1—C6	1.387 (3)	C11—C12	1.457 (3)
C1—Cl1	1.734 (2)	C11—H11	0.9300

C2—C3	1.366 (4)	C12—C13	1.393 (3)
C2—H2	0.9300	C12—C17	1.394 (3)
C3—C4	1.370 (4)	C13—C14	1.374 (3)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.373 (3)	C14—C15	1.375 (4)
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.397 (3)	C15—C16	1.363 (4)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.460 (3)	C16—C17	1.377 (3)
C7—C8	1.310 (3)	C16—H16	0.9300
C7—H7	0.9300	C17—Cl2	1.729 (3)
C8—C9	1.463 (3)		
C2—C1—Cl1	117.80 (18)	C8—C9—C10	116.29 (19)
C6—C1—Cl1	120.16 (16)	C11—C10—C9	123.1 (2)
C2—C1—Cl1	117.80 (18)	C11—C10—H10	118.4
C6—C1—Cl1	120.16 (16)	C9—C10—H10	118.4
C3—C2—C1	119.6 (2)	C10—C11—C12	126.4 (2)
C3—C2—H2	120.2	C10—C11—H11	116.8
C1—C2—H2	120.2	C12—C11—H11	116.8
C2—C3—C4	120.2 (2)	C13—C12—C17	116.0 (2)
C2—C3—H3	119.9	C13—C12—C11	121.77 (19)
C4—C3—H3	119.9	C17—C12—C11	122.2 (2)
C3—C4—C5	120.0 (2)	C14—C13—C12	122.3 (2)
C3—C4—H4	120.0	C14—C13—H13	118.8
C5—C4—H4	120.0	C12—C13—H13	118.8
C4—C5—C6	121.8 (2)	C13—C14—C15	119.5 (3)
C4—C5—H5	119.1	C13—C14—H14	120.2
C6—C5—H5	119.1	C15—C14—H14	120.2
C1—C6—C5	116.40 (19)	C16—C15—C14	120.2 (2)
C1—C6—C7	121.79 (19)	C16—C15—H15	119.9
C5—C6—C7	121.79 (19)	C14—C15—H15	119.9
C8—C7—C6	126.6 (2)	C15—C16—C17	119.9 (2)
C8—C7—H7	116.7	C15—C16—H16	120.1
C6—C7—H7	116.7	C17—C16—H16	120.1
C7—C8—C9	123.4 (2)	C16—C17—C12	122.1 (2)
C7—C8—H8	118.3	C16—C17—Cl2	117.81 (19)
C9—C8—H8	118.3	C12—C17—Cl2	120.11 (18)
O—C9—C8	121.90 (19)	C16—C17—Cl2	117.81 (19)
O—C9—C10	121.78 (19)	C12—C17—Cl2	120.11 (18)
C6—C1—C2—C3	0.2 (4)	O—C9—C10—C11	−3.4 (3)
Cl1—C1—C2—C3	−179.26 (19)	C8—C9—C10—C11	174.8 (2)
Cl1—C1—C2—C3	−179.26 (19)	C9—C10—C11—C12	−178.49 (19)
C1—C2—C3—C4	0.5 (4)	C10—C11—C12—C13	−8.8 (3)
C2—C3—C4—C5	−1.0 (4)	C10—C11—C12—C17	170.5 (2)
C3—C4—C5—C6	0.8 (4)	C17—C12—C13—C14	−0.6 (3)
C2—C1—C6—C5	−0.4 (3)	C11—C12—C13—C14	178.8 (2)
Cl1—C1—C6—C5	179.06 (16)	C12—C13—C14—C15	−0.4 (4)
Cl1—C1—C6—C5	179.06 (16)	C13—C14—C15—C16	0.8 (4)

C2—C1—C6—C7	177.7 (2)	C14—C15—C16—C17	−0.1 (4)
Cl1—C1—C6—C7	−2.9 (3)	C15—C16—C17—C12	−1.0 (4)
Cl1—C1—C6—C7	−2.9 (3)	C15—C16—C17—Cl2	179.5 (2)
C4—C5—C6—C1	−0.1 (3)	C15—C16—C17—Cl2	179.5 (2)
C4—C5—C6—C7	−178.2 (2)	C13—C12—C17—C16	1.3 (3)
C1—C6—C7—C8	170.1 (2)	C11—C12—C17—C16	−178.0 (2)
C5—C6—C7—C8	−11.9 (3)	C13—C12—C17—Cl2	−179.23 (18)
C6—C7—C8—C9	175.07 (19)	C11—C12—C17—Cl2	1.5 (3)
C7—C8—C9—O	0.9 (3)	C13—C12—C17—Cl2	−179.23 (18)
C7—C8—C9—C10	−177.3 (2)	C11—C12—C17—Cl2	1.5 (3)

Hydrogen-bond geometry (Å, °)

D—H$\cdots A$	D—H	H$\cdots A$	$D \cdots A$	D—H$\cdots A$
C8—H8$\cdots$O^i	0.93	2.55	3.283 (3)	136
C16—H16$\cdots$O^{ii}	0.93	2.56	3.416 (3)	152
C7—H7$\cdots$Cl1	0.93	2.62	3.040 (2)	108
C11—H11$\cdots$Cl2	0.93	2.63	3.050 (2)	108

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

Fig. 1