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2-Bromo-1-chlorophenyl-3-(4-methoxyphenyl)prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.024
wR factor = 0.060
Data-to-parameter ratio = 17.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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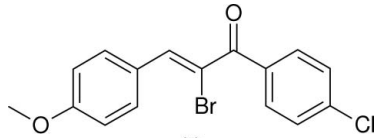
The geometrical parameters for the title compound, $C_{16}H_{12}BrClO_2$, are normal. The observed bond lengths and angles imply that there is little electronic conjugation between the two benzene ring systems. An intramolecular C—H···Br interaction may help to establish the molecular conformation. The crystal packing results in a centrosymmetric structure.

Comment

Many chalcone ($C_{15}H_{12}O$) derivatives crystallize as non-centrosymmetric structures and display significant non-linear optical (NLO) properties (Uchida *et al.*, 1998). The title compound, (I), (Fig. 1), was prepared as part of our ongoing studies in this area (Harrison *et al.*, 2005). However, (I) crystallizes in a centrosymmetric space group, thus it has a zero NLO response (Watson *et al.*, 1993).



(I)

The geometrical parameters for (I) are normal (Allen *et al.*, 1987) and consistent with those of other chalcone derivatives (Moorthi *et al.*, 2005; Patil *et al.*, 2006). The molecule of (I) is distinctly twisted about the C4—C7 and C7—C8 bonds (Table 1). This twisting, and the C4—C7 and C7—C8 bond lengths of greater than 1.48 Å, imply that there is limited electronic conjugation between the two aromatic ring systems. The dihedral angle between the benzene ring mean planes (C1–C6 and C10–C15) is 53.35 (6)°. C7 and O2 deviate from the former mean plane by 0.176 (3) and 0.895 (3) Å, respectively. By contrast, the terminal methyl atom C16 is almost coplanar with the C10–C15 ring [deviation = 0.045 (4) Å].

A *PLATON* (Spek, 2003) analysis of (I) indicated a possible intramolecular C—H···Br interaction (Table 2) that might help to maintain near coplanarity between the C8/C9/Br1 fragment and the C10-benzene ring. The predicted (Bondi, 1964) van der Waals contact distance for H and Br is 3.05 Å. There are no π···π stacking interactions in the crystal structure of (I).

Experimental

2,3-Dibromo-1-chlorophenyl-3-(4-methoxyphenyl)-2-propan-1-one (4.32 g, 0.01 mol) was mixed with triethylamine (5 ml, 0.05 mol) in toluene (100 ml). The reaction was stirred for 24 hrs. and the precipitated triethylamine hydrobromide was removed by filtration.

The solvent was removed under reduced pressure and the resulting solid mass obtained on cooling was collected by filtration. The crude product was recrystallized from ethanol to yield blocks of (I) in 60% yield. M.p.: 403 K. Analysis for $C_{16}H_{12}BrClO_2$: calc. C 54.65, H 3.44%, found: C 54.53, H 3.64%.

Crystal data

$C_{16}H_{12}BrClO_2$
M_r = 351.62
Monoclinic, $P2_1/c$
a = 13.9793 (3) Å
b = 8.8780 (1) Å
c = 11.4870 (3) Å
β = 96.7094 (10)°
V = 1415.87 (5) Å^3
Z = 4
D_x = 1.650 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3426 reflections
θ = 2.9–27.5°
μ = 3.09 mm^{-1}
T = 120 (2) K
Block, colourless
0.55 × 0.37 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan *SADABS* (Bruker, 2003)
T_{min} = 0.266, T_{max} = 0.573
19150 measured reflections
3249 independent reflections
2906 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.5°
$h = -18 \rightarrow 18$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.060
S = 1.04
3249 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2 + 1.1891P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0135 (6)

Table 1
Selected geometric parameters (Å, °).

C4—C7	1.494 (2)	C8—C9	1.346 (2)
C7—C8	1.488 (2)	C9—C10	1.460 (2)
C3—C4—C7—O2	33.7 (2)	C8—C9—C10—C15	−2.9 (3)
O2—C7—C8—Br1	19.6 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15···Br1	0.95	2.62	3.3339 (18)	132

H atoms were positioned geometrically (C—H = 0.95–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(methyl carrier). The methyl group was rotated to fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor 1997); data reduction: *HKL SCALEPACK* and *DENZO* (Otwinowski & Minor 1997), *SCALEPACK* and *SORTAV* (Blessing 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

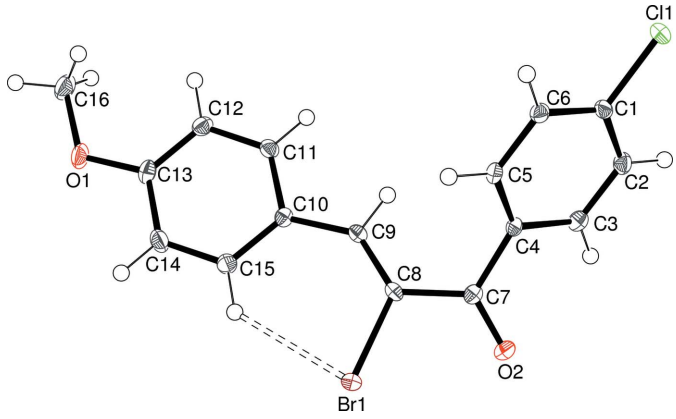


Figure 1
View of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The possible C—H···Br interaction is indicated by a dashed line.

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supporting information

Acta Cryst. (2006). E**62**, o1578–o1579 [https://doi.org/10.1107/S1600536806010464]

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$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
$h = -18 \rightarrow 18$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.060$
$S = 1.04$
3249 reflections
183 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: none
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0257P)^2 + 1.1891P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.58$ e Å^{-3}
Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0135 (6)

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.06540 (12)	0.5985 (2)	0.21192 (16)	0.0146 (3)
C2	0.05661 (13)	0.6274 (2)	0.32927 (17)	0.0168 (4)
H2	0.0102	0.6973	0.3505	0.020*
C3	0.11671 (12)	0.5522 (2)	0.41443 (16)	0.0155 (4)
H3	0.1113	0.5701	0.4949	0.019*
C4	0.18529 (12)	0.45017 (19)	0.38280 (15)	0.0121 (3)
C5	0.19221 (12)	0.4226 (2)	0.26447 (15)	0.0133 (3)
H5	0.2387	0.3531	0.2428	0.016*
C6	0.13172 (12)	0.4960 (2)	0.17835 (15)	0.0142 (3)
H6	0.1357	0.4764	0.0978	0.017*
C7	0.23978 (12)	0.35782 (19)	0.47705 (15)	0.0126 (3)
C8	0.34100 (12)	0.31417 (19)	0.46523 (15)	0.0125 (3)
C9	0.39851 (12)	0.39414 (19)	0.40209 (15)	0.0124 (3)
H9	0.3665	0.4779	0.3636	0.015*
C10	0.49874 (12)	0.38168 (19)	0.37913 (15)	0.0124 (3)
C11	0.52953 (12)	0.4872 (2)	0.30057 (15)	0.0147 (3)
H11	0.4856	0.5621	0.2685	0.018*
C12	0.62195 (13)	0.4858 (2)	0.26810 (15)	0.0163 (4)
H12	0.6408	0.5586	0.2146	0.020*
C13	0.68678 (13)	0.3769 (2)	0.31473 (16)	0.0164 (4)
C14	0.65847 (13)	0.2725 (2)	0.39495 (18)	0.0196 (4)
H14	0.7032	0.1993	0.4280	0.024*
C15	0.56618 (13)	0.2744 (2)	0.42692 (16)	0.0167 (4)
H15	0.5482	0.2026	0.4817	0.020*
C16	0.81160 (14)	0.4659 (2)	0.20706 (19)	0.0240 (4)
H16A	0.8783	0.4420	0.1956	0.036*
H16B	0.8083	0.5691	0.2367	0.036*
H16C	0.7706	0.4571	0.1321	0.036*
O1	0.77892 (10)	0.36308 (15)	0.28995 (13)	0.0228 (3)
O2	0.20035 (9)	0.31697 (15)	0.56104 (11)	0.0184 (3)
Cl1	−0.00923 (3)	0.69297 (5)	0.10428 (4)	0.02141 (11)
Br1	0.382362 (13)	0.14483 (2)	0.558415 (16)	0.01882 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0103 (8)	0.0151 (8)	0.0181 (9)	−0.0012 (7)	−0.0002 (6)	0.0050 (7)
C2	0.0142 (8)	0.0156 (9)	0.0214 (9)	0.0031 (7)	0.0053 (7)	0.0006 (7)
C3	0.0159 (9)	0.0162 (9)	0.0150 (8)	−0.0007 (7)	0.0044 (7)	−0.0014 (7)
C4	0.0087 (8)	0.0131 (8)	0.0145 (8)	−0.0024 (6)	0.0015 (6)	0.0007 (6)

C5	0.0099 (8)	0.0142 (8)	0.0161 (8)	0.0003 (6)	0.0032 (6)	−0.0009 (6)
C6	0.0122 (8)	0.0174 (9)	0.0130 (8)	−0.0016 (7)	0.0012 (6)	0.0005 (7)
C7	0.0122 (8)	0.0127 (8)	0.0129 (8)	−0.0026 (6)	0.0014 (6)	−0.0019 (6)
C8	0.0125 (8)	0.0119 (8)	0.0125 (8)	0.0021 (6)	−0.0012 (6)	0.0003 (6)
C9	0.0126 (8)	0.0114 (8)	0.0127 (8)	0.0002 (6)	−0.0011 (6)	0.0005 (6)
C10	0.0106 (8)	0.0135 (8)	0.0129 (8)	−0.0023 (6)	0.0002 (6)	−0.0015 (6)
C11	0.0129 (8)	0.0159 (9)	0.0147 (8)	0.0003 (7)	−0.0008 (7)	0.0019 (7)
C12	0.0152 (8)	0.0186 (9)	0.0153 (8)	−0.0025 (7)	0.0029 (7)	0.0016 (7)
C13	0.0117 (8)	0.0171 (9)	0.0210 (9)	−0.0015 (7)	0.0045 (7)	−0.0033 (7)
C14	0.0151 (9)	0.0150 (9)	0.0290 (10)	0.0038 (7)	0.0034 (7)	0.0039 (7)
C15	0.0144 (9)	0.0146 (9)	0.0214 (9)	−0.0008 (7)	0.0032 (7)	0.0034 (7)
C16	0.0180 (9)	0.0257 (10)	0.0304 (11)	−0.0012 (8)	0.0114 (8)	0.0021 (8)
O1	0.0129 (6)	0.0220 (7)	0.0355 (8)	0.0016 (5)	0.0106 (6)	0.0050 (6)
O2	0.0164 (6)	0.0233 (7)	0.0161 (6)	−0.0004 (5)	0.0053 (5)	0.0043 (5)
Cl1	0.0155 (2)	0.0261 (2)	0.0222 (2)	0.00535 (18)	0.00078 (17)	0.00994 (18)
Br1	0.01765 (11)	0.01849 (11)	0.02112 (11)	0.00434 (7)	0.00564 (7)	0.00930 (7)

Geometric parameters (Å, º)

C1—C6	1.385 (2)	C9—H9	0.9500
C1—C2	1.392 (3)	C10—C11	1.403 (2)
C1—Cl1	1.7375 (18)	C10—C15	1.406 (2)
C2—C3	1.384 (3)	C11—C12	1.386 (2)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.398 (2)	C12—C13	1.390 (3)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.395 (2)	C13—O1	1.357 (2)
C4—C7	1.494 (2)	C13—C14	1.396 (3)
C5—C6	1.387 (2)	C14—C15	1.382 (3)
C5—H5	0.9500	C14—H14	0.9500
C6—H6	0.9500	C15—H15	0.9500
C7—O2	1.221 (2)	C16—O1	1.432 (2)
C7—C8	1.488 (2)	C16—H16A	0.9800
C8—C9	1.346 (2)	C16—H16B	0.9800
C8—Br1	1.8963 (17)	C16—H16C	0.9800
C9—C10	1.460 (2)		

C6—C1—C2	121.89 (17)	C10—C9—H9	112.6
C6—C1—Cl1	118.98 (14)	C11—C10—C15	117.48 (15)
C2—C1—Cl1	119.13 (14)	C11—C10—C9	116.02 (15)
C3—C2—C1	118.74 (16)	C15—C10—C9	126.49 (16)
C3—C2—H2	120.6	C12—C11—C10	122.22 (17)
C1—C2—H2	120.6	C12—C11—H11	118.9
C2—C3—C4	120.46 (16)	C10—C11—H11	118.9
C2—C3—H3	119.8	C11—C12—C13	119.26 (16)
C4—C3—H3	119.8	C11—C12—H12	120.4
C5—C4—C3	119.64 (16)	C13—C12—H12	120.4
C5—C4—C7	121.48 (15)	O1—C13—C12	125.05 (16)

C3—C4—C7	118.33 (15)	O1—C13—C14	115.33 (16)
C6—C5—C4	120.44 (16)	C12—C13—C14	119.61 (16)
C6—C5—H5	119.8	C15—C14—C13	120.84 (17)
C4—C5—H5	119.8	C15—C14—H14	119.6
C1—C6—C5	118.83 (16)	C13—C14—H14	119.6
C1—C6—H6	120.6	C14—C15—C10	120.58 (16)
C5—C6—H6	120.6	C14—C15—H15	119.7
O2—C7—C8	121.10 (16)	C10—C15—H15	119.7
O2—C7—C4	119.78 (15)	O1—C16—H16A	109.5
C8—C7—C4	119.11 (14)	O1—C16—H16B	109.5
C9—C8—C7	123.07 (16)	H16A—C16—H16B	109.5
C9—C8—Br1	124.08 (13)	O1—C16—H16C	109.5
C7—C8—Br1	112.66 (12)	H16A—C16—H16C	109.5
C8—C9—C10	134.71 (16)	H16B—C16—H16C	109.5
C8—C9—H9	112.6	C13—O1—C16	117.81 (15)

C6—C1—C2—C3	0.6 (3)	C4—C7—C8—Br1	−159.02 (12)
Cl1—C1—C2—C3	−179.65 (14)	C7—C8—C9—C10	177.38 (18)
C1—C2—C3—C4	0.4 (3)	Br1—C8—C9—C10	2.7 (3)
C2—C3—C4—C5	−0.8 (3)	C8—C9—C10—C11	176.46 (19)
C2—C3—C4—C7	−172.42 (16)	C8—C9—C10—C15	−2.9 (3)
C3—C4—C5—C6	0.2 (3)	C15—C10—C11—C12	1.4 (3)
C7—C4—C5—C6	171.47 (16)	C9—C10—C11—C12	−178.08 (16)
C2—C1—C6—C5	−1.3 (3)	C10—C11—C12—C13	−0.1 (3)
Cl1—C1—C6—C5	178.99 (13)	C11—C12—C13—O1	179.83 (17)
C4—C5—C6—C1	0.9 (3)	C11—C12—C13—C14	−1.2 (3)
C5—C4—C7—O2	−137.69 (17)	O1—C13—C14—C15	−179.67 (17)
C3—C4—C7—O2	33.7 (2)	C12—C13—C14—C15	1.2 (3)
C5—C4—C7—C8	40.9 (2)	C13—C14—C15—C10	0.0 (3)
C3—C4—C7—C8	−147.67 (16)	C11—C10—C15—C14	−1.3 (3)
O2—C7—C8—C9	−155.64 (17)	C9—C10—C15—C14	178.08 (18)
C4—C7—C8—C9	25.8 (2)	C12—C13—O1—C16	−1.7 (3)
O2—C7—C8—Br1	19.6 (2)	C14—C13—O1—C16	179.23 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···Br1	0.95	2.62	3.3339 (18)	132