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(2*E*)-3-(3-Bromophenyl)-1-(4-chlorophenyl)prop-2-en-1-one: a non-merohedral twin

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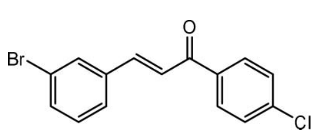
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.040; wR factor = 0.120; data-to-parameter ratio = 15.9.

In the title compound, $C_{15}H_{10}BrClO$, the molecule adopts an E configuration with respect to the C=C double bond and the dihedral angle between the aromatic ring planes is 3.98 (16)°. In the crystal, inversion dimers linked by pairs of C—H⋯O bonds are seen and weak π–π stacking [centroid–centroid separation = 3.8776 (19) Å] may further consolidate the structure. The crystal studied was a non-merohedral twin with a ratio of the twin components of 0.9093 (13):0.0907 (13). The twin operation is a twofold rotation around c*.

Related literature

For related structures and background to bromo- and chloro-substituted chalcones, see: Yathirajan *et al.* (2006), Sarojini *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{10}BrClO$
M_r = 321.59
Monoclinic, $P2_1/n$
a = 14.7421 (10) Å
b = 6.1024 (4) Å
c = 15.1874 (10) Å
β = 103.905 (2)°
V = 1326.25 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.28 mm^{-1}
T = 296 K
0.59 × 0.58 × 0.41 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.248, T_{max} = 0.346 (expected range = 0.186–0.260)
2609 measured reflections
2609 independent reflections
2273 reflections with $I > 2\sigma(I)$

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.120
S = 1.05
2609 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C1—H1⋯O1^{i}	0.93	2.53	3.328 (4)	144

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

BN thanks Mangalore University for providing research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2127).

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supplementary materials

Acta Cryst. (2009). E**65**, o1915 [doi:10.1107/S1600536809027615]

(2*E*)-3-(3-Bromophenyl)-1-(4-chlorophenyl)prop-2-en-1-one: a non-merohedral twin

H. Li, K. P. Kamath, B. Narayana, H. S. Yathirajan and W. T. A. Harrison

Comment

As part of our ongoing studies of bromo- and chloro-substituted chalcones (Yathirajan, *et al.*, 2006; Sarojini, *et al.*, 2007), we now present the synthesis and structure of the title compound (Fig. 1).

The molecule adopts an E configuration with respect to the C=C double bond of the propenone unit. The two benzene rings are approximately coplanar, with a dihedral angle of 3.98 (16)° between their mean planes. The bromine atom is displaced from the C1–C6 mean plane by -0.051 (1)Å and the chlorine atom is displaced from C10–C15 by 0.028 (1) Å. In the crystal, inversion dimers linked by pairs of weak C—H···O interactions (Table 1, Fig. 2) occur.

Experimental

50% KOH was added to a solution of 4-chloroacetophenone (1.54 g, 0.01 mol) and 3-bromobenzaldehyde (1.86 g, 0.01 mol) in 25 ml of ethanol at 273 K. The mixture was stirred for an hour at room temperature and then poured onto crushed ice. A yellow precipitate was collected by filtration and purified by recrystallization from ethanol. Yellow blocks of the title compound were grown from a mixture of acetone and toluene (1:1) by slow evaporation. Yield of the compound was 80%. Analysis: found (calculated): C%, 55.94 (56.02); H%, 3.10 (3.13).

Refinement

The H atoms were placed at calculated positions (C—H = 0.93 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C). The investigated crystal was a non-merohedral twin with a ratio of the twin components of 0.9093 (13):0.0907 (13) corresponding to a 2-fold rotation about (0 0 1), as determined with the TwinRotMat option of *PLATON* (Spek, 2009). The final refinement was carried out agains a detwinned data set.

Figures

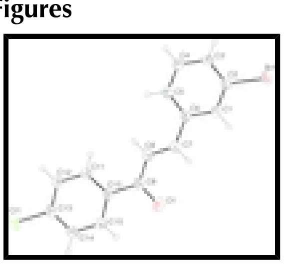

Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level and H atoms shown as spheres of arbitrary radius.

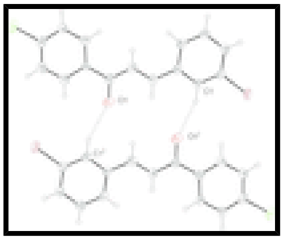

Fig. 2. An inversion dimer in the crystal of the title compound linked by two C—H···O bonds (double dashed lines). Symmetry code: (i) 1–x, –y, 1–z.

(2*E*)-3-(3-Bromophenyl)-1-(4-chlorophenyl)prop-2-en-1-one

Crystal data

$C_{15}H_{10}BrClO$	$F_{000} = 640$
$M_r = 321.59$	$D_x = 1.611$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 7751 reflections
$a = 14.7421$ (10) Å	$\theta = 2.2$–28.0°
$b = 6.1024$ (4) Å	$\mu = 3.28$ mm^{-1}
$c = 15.1874$ (10) Å	$T = 296$ K
$\beta = 103.905$ (2)°	Block, yellow
$V = 1326.25$ (15) Å^3	0.59 × 0.58 × 0.41 mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	2609 independent reflections
Radiation source: fine-focus sealed tube	2273 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.0000$
$T = 293$ K	$\theta_{max} = 26.0$°
ω scans	$\theta_{min} = 1.7$°
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -18 \rightarrow 18$
$T_{min} = 0.248$, $T_{max} = 0.346$	$k = -7 \rightarrow 7$
2609 measured reflections	$l = -7 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.120$	$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 0.8199P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2609 reflections	$\Delta\rho_{max} = 0.88$ e Å^{-3}
164 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}

Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.4051 (2)	0.2640 (5)	0.2830 (2)	0.0443 (7)
H1	0.4322	0.1261	0.2945	0.053*
C2	0.3780 (2)	0.3405 (5)	0.1953 (2)	0.0441 (7)
C3	0.3410 (2)	0.5470 (6)	0.1759 (2)	0.0509 (8)
H3	0.3226	0.5956	0.1163	0.061*
C4	0.3316 (2)	0.6805 (5)	0.2464 (2)	0.0504 (8)
H4	0.3079	0.8214	0.2342	0.060*
C5	0.3567 (2)	0.6075 (5)	0.3344 (2)	0.0477 (7)
H5	0.3506	0.7000	0.3813	0.057*
C6	0.3918 (2)	0.3935 (6)	0.3544 (2)	0.0472 (7)
C7	0.4073 (2)	0.2956 (6)	0.4461 (2)	0.0515 (8)
H7	0.4370	0.1601	0.4544	0.062*
C8	0.3834 (3)	0.3799 (6)	0.5171 (3)	0.0582 (9)
H8	0.3525	0.5139	0.5112	0.070*
C9	0.4039 (2)	0.2704 (6)	0.6041 (2)	0.0503 (8)
C10	0.3899 (2)	0.3888 (5)	0.6857 (2)	0.0422 (7)
C11	0.3495 (2)	0.5970 (6)	0.6819 (2)	0.0481 (7)
H11	0.3302	0.6680	0.6264	0.058*
C12	0.3382 (3)	0.6981 (6)	0.7600 (2)	0.0518 (8)
H12	0.3104	0.8355	0.7571	0.062*
C13	0.3684 (2)	0.5938 (6)	0.8414 (2)	0.0523 (8)
C14	0.4088 (3)	0.3879 (7)	0.8473 (2)	0.0580 (9)
H14	0.4292	0.3190	0.9032	0.070*
C15	0.4182 (2)	0.2872 (6)	0.7689 (2)	0.0525 (8)
H15	0.4442	0.1478	0.7721	0.063*
Br1	0.39001 (3)	0.15363 (7)	0.09849 (2)	0.06520 (19)
Cl1	0.35623 (9)	0.7258 (2)	0.93947 (7)	0.0815 (4)
O1	0.4300 (2)	0.0808 (5)	0.61022 (19)	0.0731 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0517 (16)	0.0409 (16)	0.0405 (16)	0.0034 (13)	0.0114 (13)	−0.0024 (13)
C2	0.0526 (17)	0.0432 (16)	0.0401 (16)	−0.0059 (13)	0.0181 (13)	−0.0060 (13)
C3	0.0623 (19)	0.0523 (19)	0.0414 (16)	0.0001 (16)	0.0187 (14)	0.0089 (15)
C4	0.0551 (18)	0.0391 (16)	0.060 (2)	0.0048 (14)	0.0191 (15)	0.0078 (14)
C5	0.0545 (18)	0.0426 (17)	0.0468 (17)	0.0035 (13)	0.0139 (14)	−0.0091 (14)
C6	0.0535 (17)	0.0461 (17)	0.0403 (16)	0.0080 (14)	0.0078 (13)	−0.0028 (13)
C7	0.0591 (19)	0.0495 (18)	0.0469 (18)	0.0030 (15)	0.0145 (15)	−0.0012 (15)
C8	0.071 (2)	0.059 (2)	0.0481 (19)	0.0158 (17)	0.0221 (17)	0.0028 (15)
C9	0.0515 (17)	0.0513 (19)	0.0463 (18)	0.0074 (15)	0.0081 (14)	−0.0043 (14)
C10	0.0420 (15)	0.0409 (16)	0.0451 (16)	−0.0005 (12)	0.0129 (12)	−0.0015 (13)
C11	0.0614 (18)	0.0457 (17)	0.0396 (16)	0.0064 (14)	0.0173 (14)	0.0036 (13)
C12	0.066 (2)	0.0447 (17)	0.0488 (18)	0.0072 (15)	0.0212 (15)	−0.0015 (14)
C13	0.0562 (18)	0.064 (2)	0.0406 (17)	−0.0010 (16)	0.0194 (14)	−0.0062 (15)
C14	0.068 (2)	0.067 (2)	0.0410 (18)	0.0094 (18)	0.0172 (16)	0.0081 (16)
C15	0.0568 (18)	0.0491 (18)	0.0523 (19)	0.0081 (15)	0.0147 (15)	0.0062 (15)
Br1	0.1023 (3)	0.0578 (3)	0.0422 (2)	−0.00177 (19)	0.0304 (2)	−0.00801 (15)
Cl1	0.1037 (8)	0.0997 (9)	0.0472 (5)	0.0144 (7)	0.0302 (5)	−0.0135 (5)
O1	0.102 (2)	0.0526 (15)	0.0671 (17)	0.0213 (15)	0.0250 (15)	−0.0046 (13)

Geometric parameters (Å, °)

C1—C2	1.377 (5)	C8—H8	0.9300
C1—C6	1.393 (4)	C9—O1	1.216 (5)
C1—H1	0.9300	C9—C10	1.491 (4)
C2—C3	1.377 (5)	C10—C15	1.379 (5)
C2—Br1	1.902 (3)	C10—C11	1.398 (4)
C3—C4	1.379 (5)	C11—C12	1.382 (5)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.373 (5)	C12—C13	1.366 (5)
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.411 (5)	C13—C14	1.384 (5)
C5—H5	0.9300	C13—Cl1	1.740 (3)
C6—C7	1.482 (5)	C14—C15	1.375 (5)
C7—C8	1.317 (5)	C14—H14	0.9300
C7—H7	0.9300	C15—H15	0.9300
C8—C9	1.446 (5)		
C2—C1—C6	119.9 (3)	C9—C8—H8	119.0
C2—C1—H1	120.1	O1—C9—C8	120.1 (3)
C6—C1—H1	120.1	O1—C9—C10	120.2 (3)
C3—C2—C1	121.6 (3)	C8—C9—C10	119.7 (3)
C3—C2—Br1	119.3 (2)	C15—C10—C11	118.6 (3)
C1—C2—Br1	119.0 (2)	C15—C10—C9	118.2 (3)
C2—C3—C4	118.9 (3)	C11—C10—C9	123.2 (3)
C2—C3—H3	120.5	C12—C11—C10	120.5 (3)

C4—C3—H3	120.5	C12—C11—H11	119.7
C5—C4—C3	120.7 (3)	C10—C11—H11	119.7
C5—C4—H4	119.6	C13—C12—C11	119.2 (3)
C3—C4—H4	119.6	C13—C12—H12	120.4
C4—C5—C6	120.6 (3)	C11—C12—H12	120.4
C4—C5—H5	119.7	C12—C13—C14	121.5 (3)
C6—C5—H5	119.7	C12—C13—Cl1	118.7 (3)
C1—C6—C5	118.1 (3)	C14—C13—Cl1	119.7 (3)
C1—C6—C7	118.9 (3)	C15—C14—C13	118.7 (3)
C5—C6—C7	122.7 (3)	C15—C14—H14	120.6
C8—C7—C6	126.9 (3)	C13—C14—H14	120.6
C8—C7—H7	116.5	C14—C15—C10	121.4 (3)
C6—C7—H7	116.5	C14—C15—H15	119.3
C7—C8—C9	122.0 (3)	C10—C15—H15	119.3
C7—C8—H8	119.0		

C6—C1—C2—C3	2.5 (5)	O1—C9—C10—C15	7.9 (5)
C6—C1—C2—Br1	−176.2 (2)	C8—C9—C10—C15	−174.1 (3)
C1—C2—C3—C4	0.5 (5)	O1—C9—C10—C11	−172.1 (3)
Br1—C2—C3—C4	179.3 (3)	C8—C9—C10—C11	5.9 (5)
C2—C3—C4—C5	−1.4 (5)	C15—C10—C11—C12	−0.2 (5)
C3—C4—C5—C6	−0.7 (5)	C9—C10—C11—C12	179.8 (3)
C2—C1—C6—C5	−4.5 (5)	C10—C11—C12—C13	1.1 (5)
C2—C1—C6—C7	170.4 (3)	C11—C12—C13—C14	−1.0 (6)
C4—C5—C6—C1	3.6 (5)	C11—C12—C13—Cl1	178.5 (3)
C4—C5—C6—C7	−171.1 (3)	C12—C13—C14—C15	−0.2 (6)
C1—C6—C7—C8	−167.8 (4)	Cl1—C13—C14—C15	−179.6 (3)
C5—C6—C7—C8	6.9 (6)	C13—C14—C15—C10	1.2 (5)
C6—C7—C8—C9	−178.8 (3)	C11—C10—C15—C14	−1.0 (5)
C7—C8—C9—O1	−12.1 (6)	C9—C10—C15—C14	179.0 (3)
C7—C8—C9—C10	169.8 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1^{i}	0.93	2.53	3.328 (4)	144

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

Fig. 1

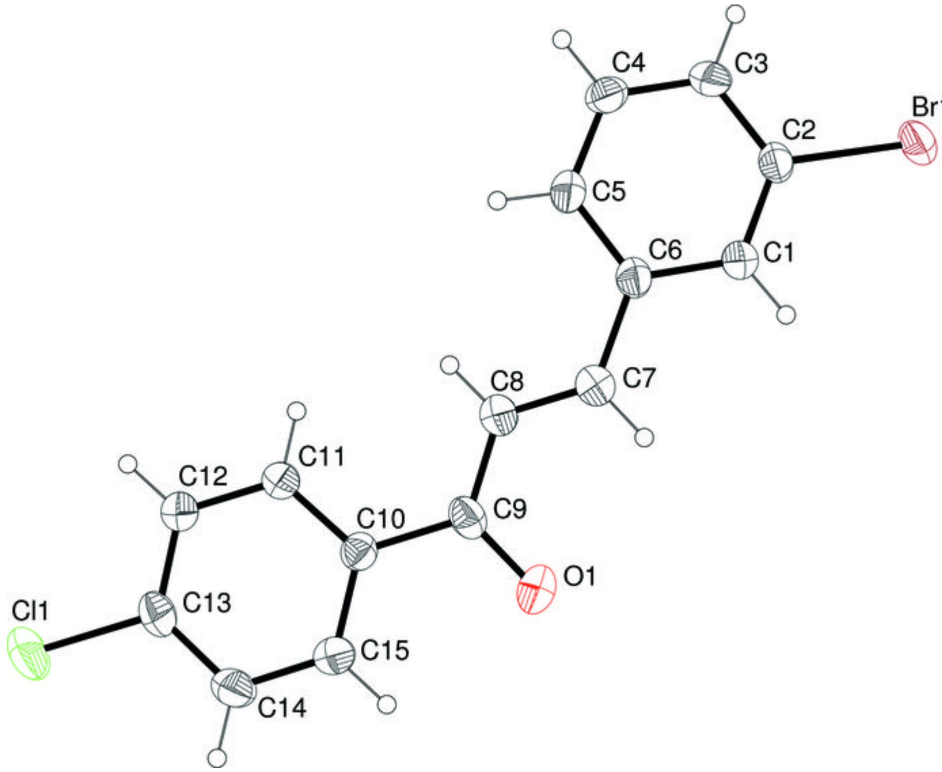

Fig. 2