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

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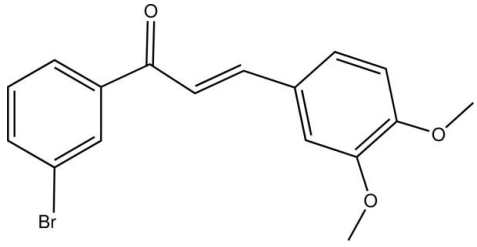
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.074; data-to-parameter ratio = 17.9.

The molecular structure of the title compound, $C_{17}H_{15}BrO_3$, consists of a bromophenyl and a 3,4-dimethoxyphenyl group linked through a prop-2-en-1-one spacer. The C=C double bond displays an *E* conformation, while the carbonyl group shows an *S-cis* conformation relative to the double bond.

Related literature

For the Suzuki reaction, see: Miyaura & Suzuki (1995); Bringmann *et al.* (2005). For bichalcone derivatives, see: Shetonde *et al.* (2010). For related structures, see: Escobar *et al.* (2008); Valdebenito *et al.* (2010); Chu *et al.* (2004); Radha Krishna *et al.* (2005); Wu *et al.* (2005).



Experimental

Crystal data

$C_{17}H_{15}BrO_3$
M_r = 347.20
Monoclinic, $P2_1/c$
a = 12.7946 (5) Å
b = 3.9373 (1) Å
c = 29.8209 (10) Å
β = 109.219 (3)°
V = 1418.54 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.91 mm^{-1}
T = 120 K
0.2 × 0.12 × 0.08 mm

Data collection

Agilent Xcalibur Sapphire3 Gemini ultra diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.802, T_{max} = 1.000
12861 measured reflections
3429 independent reflections
2895 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.074
S = 1.10
3429 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2515).

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supplementary materials

Acta Cryst. (2012). E**68**, o887 [doi:10.1107/S1600536812006836]

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

Carlos A. Escobar, Alexander Trujillo, Judith A. K. Howard and Mauricio Fuentealba

Comment

From the synthetic point of view, bromochalcones are the choice precursors to accomplish the C—C bond formation, through the Suzuki reaction, one of the most popular and powerful methods for coupling aryl–aryl moieties (Miyaura & Suzuki, 1995; Bringmann *et al.*, 2005), to produce symmetric or asymmetric biphenyls, this being the entry to bichalcones (Shetonde *et al.*, 2010).

The molecular structure of the title compound displays two phenyl rings connected through the organic prop-2-en-1-one spacer. As shown in Fig. 1, one phenyl ring is substituted at positions 3 and 4 with methoxy groups, while the other is substituted at position 3′ with one Br atom.

The dihedral angle between the two aromatic rings joined by the conjugated spacer is 26.59 (9)°. On the other hand, the spacer formed by C10—C9—C8—C7—O1—C1 can be considered as a plane with a RMSD of 0.029 Å. This feature is also observed in other chalcones (Escobar *et al.* 2008; Valdebenito *et al.*, 2010; Chu *et al.* 2004; Radha Krishna *et al.* 2005; Wu *et al.* 2005).

Finally, both inter- and intramolecular hydrogen bonds are not observed in the crystalline packing of title compound.

Experimental

A mixture of 3-bromoacetophenone (0.5 g, 2,5 mmol) and 3,4-dimethoxibenzaldehyde (0.41 g, 2,5 mmol), were dissolved in Methanol (50 ml), and were treated with KOH (2 g, dissolved in 20 ml methanol). After 20 min 30 ml of water were added, and the title compound precipitated as a yellow solid. Then, it was filtered and recrystallized in ethanol to yield 1.27 g (73%) of a yellow solid. m.p.117–120°C. **IR** (KBr): ν = 1657 (CO), cm^{-1}. **^{1}H-NMR** (400 MHz, $CDCl_3$): d = 3.94 (3*H*, s, OCH_3), 3.96 (3*H*, s, OCH_3), 6.90 (1*H*, d, *J* = 8.3 Hz, H5), 7.16 (1*H*, m, H2), 7.24 (1*H*, d, *J* = 7.2 Hz., H6), 7.32 (1*H*, d, *J* = 15.6 Hz., Ha), 7.38 (1*H*, t, *J* = 7.8 Hz., H5), 7.70 (1*H*, d, *J* = 7.9 Hz., H4), 7.77 (1*H*, d, *J* = 15.6 Hz., Hb), 7.93 (1*H*, d, *J* = 7.7 Hz., H6), 8.13 (1*H*, m, H2). **^{13}C-NMR** (400 MHz, $CDCl_3$): d = 56.1, 110.2, 111.2, 119.4, 122.9, 123.5, 127.0, 127.6, 130.2, 131.4, 135.4, 140.4, 146.0, 149.3, 151.8, 189.1. **HRMS** calc. for $C_{17}H_{15}BrO_3$ 346.02046; Found 346.019994.

Refinement

The H atoms positions were calculated after each cycle of refinement using a riding model with C—H distances in the range 0.95—0.98 Å and U_{iso}(H) = 1.2–1.5U_{eq}(C).

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

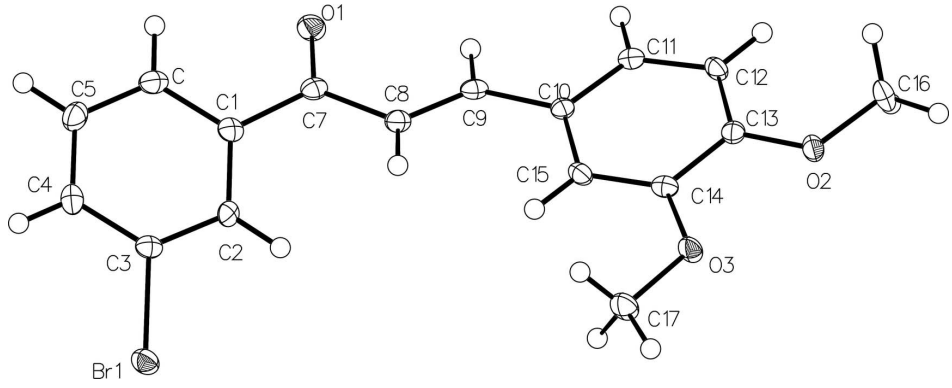

Figure 1

The molecular structure of title compound with full atom numbering scheme. Displacement ellipsoids are presented at 30% probability level and H atoms are shown as spheres.

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

Crystal data

$C_{17}H_{15}BrO_3$
$M_r = 347.20$
Monoclinic, $P2_1/c$
$a = 12.7946\ (5)$ Å
$b = 3.9373\ (1)$ Å
$c = 29.8209\ (10)$ Å
$\beta = 109.219\ (3)°$
$V = 1418.54\ (8)$ Å^3
$Z = 4$
$F(000) = 704$
$D_x = 1.626$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 3587 reflections
$\theta = 2.6–29.0°$
$\mu = 2.91$ mm^{-1}
$T = 120$ K
Prism, colourless
0.2 × 0.12 × 0.08 mm

Data collection

Agilent Xcalibur Sapphire3 Gemini ultra diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.1511 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min} = 0.802$, $T_{max} = 1.000$
12861 measured reflections
3429 independent reflections
2895 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 29.0°$, $\theta_{min} = 2.6°$
$h = -17\rightarrow16$
$k = -5\rightarrow5$
$l = -40\rightarrow40$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.074$
$S = 1.10$
3429 reflections
192 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
$w = 1/[\sigma^2(F_o^2) + (0.019P)^2 + 1.5139P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.63$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

Special details

Experimental. Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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}
Br1	−0.11568 (2)	0.90741 (7)	0.528213 (9)	0.01853 (9)
O1	−0.25274 (14)	0.3062 (5)	0.31571 (6)	0.0198 (4)
O2	0.40922 (14)	0.3072 (5)	0.32327 (6)	0.0173 (4)
O3	0.37580 (14)	0.5457 (5)	0.39741 (6)	0.0190 (4)
C16	0.4322 (2)	0.1612 (7)	0.28340 (10)	0.0205 (6)
H16A	0.3873	0.2751	0.2542	0.031*
H16B	0.4141	−0.0814	0.2813	0.031*
H16C	0.5109	0.1902	0.2874	0.031*
C9	−0.0247 (2)	0.2800 (7)	0.32953 (9)	0.0143 (5)
H9	−0.0759	0.1507	0.3052	0.017*
C11	0.1119 (2)	0.1273 (6)	0.29162 (9)	0.0143 (5)
H11	0.0535	0.0184	0.2676	0.017*
C12	0.2171 (2)	0.1330 (6)	0.28749 (9)	0.0148 (5)
H12	0.2298	0.0357	0.2606	0.018*
C14	0.2834 (2)	0.4212 (7)	0.36346 (9)	0.0143 (5)
C1	−0.2297 (2)	0.6078 (7)	0.38681 (9)	0.0140 (5)
C15	0.1780 (2)	0.4234 (7)	0.36627 (9)	0.0144 (5)
H15	0.1649	0.5243	0.3928	0.017*
C5	−0.3827 (2)	0.8723 (7)	0.40364 (10)	0.0201 (6)
H5	−0.4579	0.9429	0.3932	0.024*
C10	0.0897 (2)	0.2760 (6)	0.32972 (9)	0.0130 (5)
C3	−0.2072 (2)	0.8273 (6)	0.46427 (9)	0.0142 (5)
C4	−0.3163 (2)	0.9344 (7)	0.45004 (10)	0.0178 (6)
H4	−0.3450	1.0479	0.4716	0.021*
C8	−0.0668 (2)	0.4429 (7)	0.35928 (9)	0.0149 (5)
H8	−0.0187	0.5619	0.3858	0.018*
C2	−0.1622 (2)	0.6663 (6)	0.43363 (9)	0.0135 (5)
H2	−0.0869	0.5968	0.4442	0.016*
C13	0.3031 (2)	0.2829 (6)	0.32325 (9)	0.0138 (5)
C7	−0.1871 (2)	0.4390 (7)	0.35127 (9)	0.0143 (5)
C17	0.3612 (2)	0.6874 (7)	0.43882 (9)	0.0212 (6)
H17A	0.3096	0.8790	0.4298	0.032*
H17B	0.4327	0.7667	0.4604	0.032*
H17C	0.3313	0.5140	0.4548	0.032*
C6	−0.3401 (2)	0.7083 (7)	0.37247 (10)	0.0175 (6)

H6	−0.3866	0.6638	0.3409	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02037 (14)	0.02143 (14)	0.01387 (14)	−0.00093 (12)	0.00574 (10)	−0.00270 (12)
O1	0.0164 (9)	0.0267 (11)	0.0153 (10)	−0.0041 (8)	0.0037 (8)	−0.0030 (8)
O2	0.0138 (9)	0.0241 (10)	0.0154 (10)	0.0002 (8)	0.0068 (7)	−0.0033 (8)
O3	0.0141 (9)	0.0302 (11)	0.0126 (10)	−0.0035 (8)	0.0042 (7)	−0.0081 (9)
C16	0.0222 (14)	0.0244 (15)	0.0198 (15)	0.0009 (12)	0.0138 (12)	−0.0032 (12)
C9	0.0151 (13)	0.0143 (12)	0.0115 (13)	−0.0014 (11)	0.0017 (10)	0.0032 (11)
C11	0.0151 (12)	0.0144 (13)	0.0115 (13)	0.0002 (10)	0.0018 (10)	0.0005 (11)
C12	0.0198 (13)	0.0146 (13)	0.0115 (13)	0.0031 (11)	0.0073 (10)	−0.0011 (11)
C14	0.0173 (12)	0.0132 (12)	0.0113 (13)	−0.0004 (11)	0.0030 (10)	−0.0002 (11)
C1	0.0150 (12)	0.0128 (12)	0.0148 (13)	−0.0034 (11)	0.0058 (10)	0.0026 (11)
C15	0.0190 (13)	0.0143 (12)	0.0111 (13)	0.0019 (11)	0.0065 (10)	−0.0001 (11)
C5	0.0137 (13)	0.0235 (15)	0.0237 (15)	0.0011 (11)	0.0071 (11)	0.0050 (13)
C10	0.0150 (12)	0.0115 (12)	0.0126 (13)	0.0025 (10)	0.0048 (10)	0.0031 (10)
C3	0.0156 (12)	0.0133 (13)	0.0131 (14)	−0.0031 (10)	0.0039 (10)	0.0019 (10)
C4	0.0168 (13)	0.0176 (13)	0.0217 (15)	0.0010 (11)	0.0102 (11)	−0.0001 (12)
C8	0.0146 (12)	0.0164 (13)	0.0128 (13)	−0.0009 (11)	0.0033 (10)	0.0017 (11)
C2	0.0104 (12)	0.0140 (13)	0.0174 (14)	−0.0024 (10)	0.0064 (10)	0.0030 (11)
C13	0.0141 (12)	0.0135 (12)	0.0141 (13)	0.0016 (10)	0.0052 (10)	0.0031 (11)
C7	0.0155 (12)	0.0124 (12)	0.0141 (13)	−0.0005 (11)	0.0036 (10)	0.0036 (11)
C17	0.0201 (14)	0.0285 (16)	0.0147 (14)	−0.0036 (12)	0.0054 (11)	−0.0065 (12)
C6	0.0159 (13)	0.0203 (14)	0.0141 (14)	−0.0026 (11)	0.0019 (10)	0.0025 (11)

Geometric parameters (Å, º)

Br1—C3	1.907 (3)	C1—C2	1.398 (3)
O1—C7	1.232 (3)	C1—C7	1.498 (4)
O2—C16	1.436 (3)	C1—C6	1.391 (4)
O2—C13	1.361 (3)	C15—H15	0.9500
O3—C14	1.369 (3)	C15—C10	1.411 (3)
O3—C17	1.422 (3)	C5—H5	0.9500
C16—H16A	0.9800	C5—C4	1.387 (4)
C16—H16B	0.9800	C5—C6	1.383 (4)
C16—H16C	0.9800	C3—C4	1.384 (4)
C9—H9	0.9500	C3—C2	1.384 (4)
C9—C10	1.462 (3)	C4—H4	0.9500
C9—C8	1.343 (4)	C8—H8	0.9500
C11—H11	0.9500	C8—C7	1.477 (3)
C11—C12	1.391 (3)	C2—H2	0.9500
C11—C10	1.388 (3)	C17—H17A	0.9800
C12—H12	0.9500	C17—H17B	0.9800
C12—C13	1.387 (3)	C17—H17C	0.9800
C14—C15	1.379 (3)	C6—H6	0.9500
C14—C13	1.413 (4)		
C13—O2—C16	116.6 (2)	C11—C10—C15	118.5 (2)

C14—O3—C17	117.0 (2)	C15—C10—C9	123.0 (2)
O2—C16—H16A	109.5	C4—C3—Br1	118.8 (2)
O2—C16—H16B	109.5	C2—C3—Br1	118.96 (19)
O2—C16—H16C	109.5	C2—C3—C4	122.3 (2)
H16A—C16—H16B	109.5	C5—C4—H4	120.7
H16A—C16—H16C	109.5	C3—C4—C5	118.5 (2)
H16B—C16—H16C	109.5	C3—C4—H4	120.7
C10—C9—H9	115.8	C9—C8—H8	119.6
C8—C9—H9	115.8	C9—C8—C7	120.8 (2)
C8—C9—C10	128.5 (2)	C7—C8—H8	119.6
C12—C11—H11	119.0	C1—C2—H2	120.7
C10—C11—H11	119.0	C3—C2—C1	118.7 (2)
C10—C11—C12	122.0 (2)	C3—C2—H2	120.7
C11—C12—H12	120.5	O2—C13—C12	124.7 (2)
C13—C12—C11	119.0 (2)	O2—C13—C14	115.4 (2)
C13—C12—H12	120.5	C12—C13—C14	119.9 (2)
O3—C14—C15	125.2 (2)	O1—C7—C1	119.5 (2)
O3—C14—C13	114.5 (2)	O1—C7—C8	121.5 (2)
C15—C14—C13	120.3 (2)	C8—C7—C1	118.9 (2)
C2—C1—C7	122.0 (2)	O3—C17—H17A	109.5
C6—C1—C2	119.4 (2)	O3—C17—H17B	109.5
C6—C1—C7	118.6 (2)	O3—C17—H17C	109.5
C14—C15—H15	119.9	H17A—C17—H17B	109.5
C14—C15—C10	120.1 (2)	H17A—C17—H17C	109.5
C10—C15—H15	119.9	H17B—C17—H17C	109.5
C4—C5—H5	119.8	C1—C6—H6	119.6
C6—C5—H5	119.8	C5—C6—C1	120.7 (2)
C6—C5—C4	120.3 (2)	C5—C6—H6	119.6
C11—C10—C9	118.4 (2)		

Br1—C3—C4—C5	−179.3 (2)	C10—C11—C12—C13	2.0 (4)
Br1—C3—C2—C1	179.90 (18)	C4—C5—C6—C1	−1.0 (4)
O3—C14—C15—C10	−177.6 (2)	C4—C3—C2—C1	−0.5 (4)
O3—C14—C13—O2	−2.7 (3)	C8—C9—C10—C11	−171.3 (3)
O3—C14—C13—C12	176.7 (2)	C8—C9—C10—C15	7.1 (4)
C16—O2—C13—C12	−0.9 (4)	C2—C1—C7—O1	159.4 (2)
C16—O2—C13—C14	178.5 (2)	C2—C1—C7—C8	−22.0 (4)
C9—C8—C7—O1	−4.7 (4)	C2—C1—C6—C5	1.6 (4)
C9—C8—C7—C1	176.7 (2)	C2—C3—C4—C5	1.1 (4)
C11—C12—C13—O2	−179.5 (2)	C13—C14—C15—C10	2.3 (4)
C11—C12—C13—C14	1.1 (4)	C7—C1—C2—C3	179.2 (2)
C12—C11—C10—C9	175.6 (2)	C7—C1—C6—C5	−178.5 (2)
C12—C11—C10—C15	−2.9 (4)	C17—O3—C14—C15	0.2 (4)
C14—C15—C10—C9	−177.7 (2)	C17—O3—C14—C13	−179.8 (2)
C14—C15—C10—C11	0.7 (4)	C6—C1—C2—C3	−0.8 (4)
C15—C14—C13—O2	177.3 (2)	C6—C1—C7—O1	−20.6 (4)
C15—C14—C13—C12	−3.3 (4)	C6—C1—C7—C8	158.1 (2)
C10—C9—C8—C7	175.1 (2)	C6—C5—C4—C3	−0.3 (4)