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(*E*)-1-(1,3-Benzodioxol-5-yl)-3-(3-bromophenyl)prop-2-en-1-one

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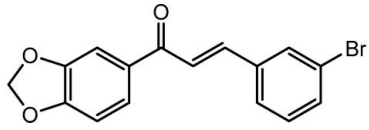
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.026; wR factor = 0.073; data-to-parameter ratio = 13.0.

In the title compound, $C_{16}H_{11}BrO_3$, the molecules adopt an E configuration with respect to the C=C double bond of the propenone unit. The 13 non-H atoms of the benzodioxole and propenone units are approximately coplanar (r.m.s. deviation = 0.027 Å) and the bromobenzene ring plane forms a dihedral angle of 10.8 (1)° to this plane. The structure is layered, with the molecules forming a herring-bone arrangement within each layer.

Related literature

For the use of chalcones as starting materials in the preparation of various molecules including fused heterocyclic compounds, see: Insuasty *et al.* (1997). For related structures, see: Butcher *et al.* (2007*a*,*b*,*c*); Low *et al.* (2002).



Experimental

Crystal data

$C_{16}H_{11}BrO_3$
M_r = 331.16
Monoclinic, $P2_1/c$
a = 14.237 (3) Å
b = 8.1811 (17) Å
c = 11.717 (2) Å
β = 100.658 (3)°
V = 1341.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.07 mm^{-1}
T = 273 (2) K
0.12 × 0.10 × 0.06 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.710, T_{max} = 0.837
6752 measured reflections
2362 independent reflections
1869 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.073
S = 1.04
2362 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2315).

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supporting information

Acta Cryst. (2008). E**64**, o2387 [doi:10.1107/S1600536808037446]

(*E*)-1-(1,3-Benzodioxol-5-yl)-3-(3-bromophenyl)prop-2-en-1-one

Hongqi Li, T. V. Sreevidya, B. Narayana, B. K. Sarojini and H. S. Yathirajan

S1. Comment

Chalcones have been widely used as starting materials in preparation of various molecules including fused heterocyclic compounds (Insuasty *et al.*, 1997). Chalcones are also finding application as organic nonlinear optical (NLO) materials because of their SHG conversion efficiency. The crystal structures of some benzodioxol- and bromo-substituted chalcones have been studied (Butcher *et al.*, 2007*a*,*b*,*c*). In continuation of this theme, and also owing to the general importance of these flavanoid analogues, we report herein the synthesis and crystal structure of a new chalcone, namely (2*E*)-1-(1,3-benzodioxol-5-yl)- 3-(3-bromophenyl)prop-2-en-1-one.

S2. Experimental

To a mixture of 1-(1,3-benzodioxol-5-yl)ethanone (1.64 g, 0.01 mol) and 3-bromobenzaldehyde (1.86 g, 0.01 mol) in 25 ml of ethanol, 50% KOH(aq) was added. The mixture was stirred for one hour at room temperature then the precipitate was collected by filtration and purified by recrystallization from ethanol. Single crystals were grown from toluene by slow evaporation. Yield: 82 %, m.p. 393–395 K. Elemental analysis calculated: C 58.03, H 3.35%; found: C 58.12, H 3.21%.

S3. Refinement

H atoms were placed at calculated positions and refined using a riding model, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C).

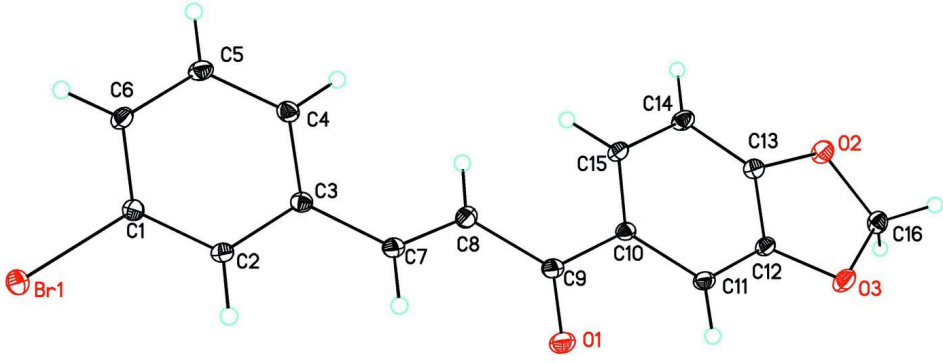

Figure 1

Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

(*E*)-1-(1,3-Benzodioxol-5-yl)-3-(3-bromophenyl)prop-2-en-1-one

Crystal data

$C_{16}H_{11}BrO_3$
$M_r = 331.16$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.237$ (3) Å
$b = 8.1811$ (17) Å
$c = 11.717$ (2) Å
$\beta = 100.658$ (3)°
$V = 1341.1$ (5) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.640$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3007 reflections
$\theta = 2.9$–$28.0°$
$\mu = 3.07$ mm^{-1}
$T = 273$ K
Block, colorless
0.12 × 0.10 × 0.06 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.710$, $T_{max} = 0.837$
6752 measured reflections
2362 independent reflections
1869 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.9°$
$h = -16 \rightarrow 16$
$k = -9 \rightarrow 9$
$l = -13 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.073$
$S = 1.04$
2362 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.3973P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0063 (7)

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}
Br1	0.489230 (17)	1.25202 (3)	0.29468 (2)	0.05206 (13)
O1	0.93644 (14)	0.7277 (2)	0.27018 (16)	0.0622 (6)
O2	1.29937 (12)	0.4896 (2)	0.61302 (13)	0.0541 (5)

O3	1.26700 (12)	0.4663 (3)	0.41361 (14)	0.0599 (5)
C1	0.58905 (15)	1.1426 (3)	0.39752 (18)	0.0360 (5)
C2	0.65910 (15)	1.0623 (3)	0.35360 (18)	0.0368 (5)
H2	0.6575	1.0608	0.2739	0.044*
C3	0.73304 (16)	0.9827 (3)	0.42861 (18)	0.0370 (5)
C4	0.73303 (17)	0.9887 (3)	0.54787 (19)	0.0433 (6)
H4	0.7819	0.9381	0.5994	0.052*
C5	0.66100 (17)	1.0691 (3)	0.58983 (19)	0.0455 (6)
H5	0.6615	1.0709	0.6693	0.055*
C6	0.58831 (16)	1.1468 (3)	0.51479 (19)	0.0427 (6)
H6	0.5399	1.2009	0.5430	0.051*
C7	0.80648 (16)	0.8953 (3)	0.3795 (2)	0.0424 (6)
H7	0.7946	0.8819	0.2993	0.051*
C8	0.88707 (17)	0.8340 (3)	0.4361 (2)	0.0464 (6)
H8	0.9018	0.8455	0.5164	0.056*
C9	0.95504 (19)	0.7472 (3)	0.3760 (2)	0.0431 (6)
C10	1.04589 (16)	0.6841 (3)	0.44539 (18)	0.0355 (5)
C11	1.11052 (16)	0.6051 (3)	0.38609 (19)	0.0427 (6)
H11	1.0972	0.5928	0.3058	0.051*
C12	1.19286 (16)	0.5475 (3)	0.45046 (18)	0.0386 (5)
C13	1.21294 (16)	0.5615 (3)	0.56996 (19)	0.0394 (5)
C14	1.15146 (17)	0.6343 (3)	0.6304 (2)	0.0493 (6)
H14	1.1650	0.6418	0.7109	0.059*
C15	1.06741 (18)	0.6969 (3)	0.5658 (2)	0.0418 (6)
H15	1.0242	0.7489	0.6042	0.050*
C16	1.33689 (17)	0.4360 (3)	0.5143 (2)	0.0473 (6)
H16A	1.3952	0.4951	0.5098	0.057*
H16B	1.3516	0.3203	0.5208	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03784 (17)	0.0701 (2)	0.04670 (18)	0.00990 (12)	0.00367 (11)	0.00788 (12)
O1	0.0524 (12)	0.0986 (16)	0.0368 (11)	0.0217 (10)	0.0111 (8)	0.0093 (9)
O2	0.0400 (10)	0.0774 (12)	0.0419 (9)	0.0128 (9)	−0.0005 (7)	−0.0046 (9)
O3	0.0465 (10)	0.0928 (14)	0.0404 (9)	0.0245 (10)	0.0086 (8)	−0.0085 (9)
C1	0.0279 (11)	0.0424 (13)	0.0372 (12)	−0.0039 (9)	0.0045 (9)	0.0031 (9)
C2	0.0360 (12)	0.0434 (13)	0.0317 (11)	−0.0044 (10)	0.0080 (9)	0.0011 (9)
C3	0.0351 (12)	0.0400 (12)	0.0361 (12)	−0.0047 (10)	0.0069 (9)	0.0021 (10)
C4	0.0381 (13)	0.0520 (14)	0.0383 (12)	0.0012 (11)	0.0034 (10)	0.0063 (11)
C5	0.0462 (14)	0.0609 (16)	0.0301 (12)	−0.0019 (12)	0.0090 (10)	−0.0019 (11)
C6	0.0352 (13)	0.0548 (15)	0.0403 (13)	0.0008 (11)	0.0128 (10)	−0.0035 (11)
C7	0.0419 (14)	0.0489 (14)	0.0382 (13)	0.0028 (11)	0.0127 (10)	0.0068 (10)
C8	0.0439 (14)	0.0554 (15)	0.0406 (13)	0.0076 (12)	0.0101 (11)	−0.0007 (11)
C9	0.0440 (14)	0.0498 (14)	0.0375 (14)	0.0025 (11)	0.0129 (10)	0.0058 (10)
C10	0.0352 (12)	0.0384 (11)	0.0347 (12)	−0.0018 (10)	0.0113 (9)	0.0034 (10)
C11	0.0437 (14)	0.0549 (15)	0.0300 (12)	0.0041 (11)	0.0081 (10)	0.0013 (10)
C12	0.0352 (12)	0.0470 (13)	0.0356 (12)	0.0019 (10)	0.0115 (10)	−0.0016 (10)

C13	0.0329 (12)	0.0474 (13)	0.0367 (12)	−0.0029 (10)	0.0031 (9)	−0.0013 (10)
C14	0.0473 (15)	0.0695 (17)	0.0300 (12)	0.0052 (13)	0.0042 (11)	−0.0067 (11)
C15	0.0412 (14)	0.0499 (13)	0.0369 (13)	0.0013 (11)	0.0142 (10)	−0.0034 (10)
C16	0.0366 (13)	0.0539 (15)	0.0504 (14)	0.0058 (11)	0.0056 (11)	−0.0028 (11)

Geometric parameters (Å, º)

Br1—C1	1.908 (2)	C7—C8	1.314 (3)
O1—C9	1.230 (3)	C7—H7	0.930
O2—C13	1.373 (3)	C8—C9	1.479 (3)
O2—C16	1.429 (3)	C8—H8	0.930
O3—C12	1.382 (3)	C9—C10	1.487 (3)
O3—C16	1.418 (3)	C10—C15	1.391 (3)
C1—C2	1.371 (3)	C10—C11	1.408 (3)
C1—C6	1.376 (3)	C11—C12	1.356 (3)
C2—C3	1.401 (3)	C11—H11	0.930
C2—H2	0.930	C12—C13	1.381 (3)
C3—C4	1.398 (3)	C13—C14	1.360 (3)
C3—C7	1.469 (3)	C14—C15	1.390 (3)
C4—C5	1.383 (3)	C14—H14	0.930
C4—H4	0.930	C15—H15	0.930
C5—C6	1.383 (3)	C16—H16A	0.970
C5—H5	0.930	C16—H16B	0.970
C6—H6	0.930		
C13—O2—C16	106.13 (17)	O1—C9—C10	120.6 (2)
C12—O3—C16	106.43 (17)	C8—C9—C10	119.1 (2)
C2—C1—C6	121.8 (2)	C15—C10—C11	119.6 (2)
C2—C1—Br1	119.75 (16)	C15—C10—C9	122.3 (2)
C6—C1—Br1	118.45 (17)	C11—C10—C9	118.13 (19)
C1—C2—C3	120.1 (2)	C12—C11—C10	117.5 (2)
C1—C2—H2	119.9	C12—C11—H11	121.3
C3—C2—H2	119.9	C10—C11—H11	121.3
C4—C3—C2	118.1 (2)	C11—C12—C13	122.1 (2)
C4—C3—C7	122.7 (2)	C11—C12—O3	128.7 (2)
C2—C3—C7	119.15 (19)	C13—C12—O3	109.26 (19)
C5—C4—C3	120.6 (2)	C14—C13—O2	128.0 (2)
C5—C4—H4	119.7	C14—C13—C12	122.1 (2)
C3—C4—H4	119.7	O2—C13—C12	109.89 (19)
C6—C5—C4	120.7 (2)	C13—C14—C15	116.7 (2)
C6—C5—H5	119.7	C13—C14—H14	121.6
C4—C5—H5	119.7	C15—C14—H14	121.6
C1—C6—C5	118.7 (2)	C14—C15—C10	122.0 (2)
C1—C6—H6	120.7	C14—C15—H15	119.0
C5—C6—H6	120.7	C10—C15—H15	119.0
C8—C7—C3	127.3 (2)	O3—C16—O2	108.07 (18)
C8—C7—H7	116.3	O3—C16—H16A	110.1
C3—C7—H7	116.3	O2—C16—H16A	110.1

C7—C8—C9	122.0 (2)	O3—C16—H16B	110.1
C7—C8—H8	119.0	O2—C16—H16B	110.1
C9—C8—H8	119.0	H16A—C16—H16B	108.4
O1—C9—C8	120.3 (2)		

C6—C1—C2—C3	−0.5 (3)	C15—C10—C11—C12	−1.4 (3)
Br1—C1—C2—C3	179.54 (16)	C9—C10—C11—C12	−179.7 (2)
C1—C2—C3—C4	−0.3 (3)	C10—C11—C12—C13	1.4 (4)
C1—C2—C3—C7	179.0 (2)	C10—C11—C12—O3	179.9 (2)
C2—C3—C4—C5	1.0 (4)	C16—O3—C12—C11	178.7 (2)
C7—C3—C4—C5	−178.4 (2)	C16—O3—C12—C13	−2.6 (3)
C3—C4—C5—C6	−0.8 (4)	C16—O2—C13—C14	−178.3 (3)
C2—C1—C6—C5	0.7 (3)	C16—O2—C13—C12	3.2 (3)
Br1—C1—C6—C5	−179.37 (17)	C11—C12—C13—C14	−0.2 (4)
C4—C5—C6—C1	0.0 (4)	O3—C12—C13—C14	−179.0 (2)
C4—C3—C7—C8	−10.9 (4)	C11—C12—C13—O2	178.4 (2)
C2—C3—C7—C8	169.8 (2)	O3—C12—C13—O2	−0.4 (3)
C3—C7—C8—C9	179.6 (2)	O2—C13—C14—C15	−179.3 (2)
C7—C8—C9—O1	−1.4 (4)	C12—C13—C14—C15	−1.0 (4)
C7—C8—C9—C10	178.7 (2)	C13—C14—C15—C10	1.0 (4)
O1—C9—C10—C15	−176.9 (2)	C11—C10—C15—C14	0.2 (4)
C8—C9—C10—C15	3.1 (3)	C9—C10—C15—C14	178.5 (2)
O1—C9—C10—C11	1.4 (4)	C12—O3—C16—O2	4.5 (3)
C8—C9—C10—C11	−178.6 (2)	C13—O2—C16—O3	−4.7 (3)