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(2*E*)-3-(5-Bromo-2-thienyl)-1-(4-hydroxyphenyl)prop-2-en-1-one

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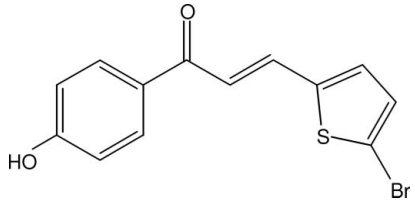
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.066; data-to-parameter ratio = 15.5.

Geometric parameters of the title compound, $C_{13}H_9BrO_2S$, a chalcone derivative, are in the usual ranges. The C=C double bond is *trans* configured. The molecule is essentially planar (r.m.s. deviation for all non-H atoms = 0.069 Å). The crystal packing is stabilized by an O—H···O hydrogen bond.

Related literature

For related literature, see: Arty *et al.* (2000); Balaji *et al.* (2003); Batt *et al.* (1993); De Leon *et al.* (2003); Dimmock *et al.* (1999); Harrison *et al.* (2006); Modzelewska *et al.* (2006); Ng *et al.* (2006); Opletalova & Sedivy (1999); Opletalova *et al.* (2003); Patil *et al.* (2007); Sogawa *et al.* (1994); Won *et al.* (2005); Yathirajan *et al.* (2006); Zhao *et al.* (2000).



Experimental

Crystal data

$C_{13}H_9BrO_2S$
M_r = 309.17
Orthorhombic, *Pbca*
a = 13.2344 (8) Å
b = 11.0471 (8) Å
c = 16.1466 (9) Å
V = 2360.7 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.64 mm^{-1}
T = 173 (2) K
0.31 × 0.26 × 0.21 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.398, T_{max} = 0.515
(expected range = 0.360–0.465)
18671 measured reflections
2461 independent reflections
2186 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.066
S = 1.07
2461 reflections
159 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O1^{i}	0.92 (5)	1.79 (5)	2.701 (3)	173 (5)

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2456).

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supplementary materials

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(2*E*)-3-(5-Bromo-2-thienyl)-1-(4-hydroxyphenyl)prop-2-en-1-one

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Comment

Chalcone derivatives are of interest because of their pharmacological properties (Batt *et al.*, 1993; Sogawa *et al.*, 1994; Arty *et al.*, 2000). They have a wide variety of pharmaceutical activities including anticancer (Modzelewska *et al.*, 2006), anti inflammatory (Won *et al.*, 2005) and antipyretic (De Leon *et al.*, 2003). Chalcones and their heterocyclic derivatives show numerous biological effects (Opletalova & Sedivy, 1999). The cytotoxic, anticancer, chemopreventative and mutagenic properties of a number of chalcones have been reviewed (Dimmock *et al.*, 1999). Chalcones and their analogues are used as potential therapeutic agents in diseases of the cardiovascular system (Opletalova *et al.*, 2003). Photo-cross-linkable polymers having the chalcone moiety act as negative photo resist materials used in a wide variety of applications (Balaji *et al.*, 2003). Chalcones are also used in designing effective second-order non-linear optical materials (Zhao *et al.*, 2000)·The crystal structures of 3-(5-bromo-2-thienyl)-1-(4-methoxyphenyl)-prop-2-en-1-one (Patil *et al.*, 2007), 1-(3-bromo-2-thienyl)-3-(4-methoxyphenyl)prop-2-en-1-one (Harrison *et al.*, 2006), 1-(4-chlorophenyl)-3-(2-thienyl)prop-2-en-1-one (Ng *et al.*, 2006), 1-(3-bromo-2-thienyl)-3-(4,5-dimethoxy-2-nitrophenyl)prop-2-en-1-one (Yathirajan *et al.*, 2006) have been reported. The structure determination of the title compound, (I), was undertaken as a part of our study on chalcones.

Geometric parameters of the title compound are in the usual ranges. The C—C double bond is *trans* configured. The molecule is essentially planar (r.m.s. deviation for all non-H atoms 0.069 Å). The crystal packing is stabilized by a O—H···O hydrogen bond.

Experimental

5-Bromo-2-thiophene carbaldehyde (1.91 g, 0.01 mol) and 4-hydroxyacetophenone (1.36 g, 0.01 mol) were stirred in ethanol (30 ml) at 298 K. 10 ml of a 10% aqueous NaOH solution was added slowly. The mixture was stirred for 2 h. The resulting precipitate was filtered off, washed with water and dried. The resulting crude product was recrystallized from 1,4-dioxane. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of the solution of the compound in 1,4-dioxane.

Refinement

All H atoms were found in a difference map, but those bonded to C were geometrically positioned and refined with fixed individual displacement parameters [U_{iso}(H) = 1.2 U_{eq}(C)] using a riding model with C—H = 0.95 Å. The hydroxyl H atom was freely refined.

Figures

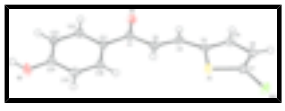

Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

(2E)-3-(5-Bromo-2-thienyl)-1-(4-hydroxyphenyl)prop-2-en-1-one

Crystal data

$C_{13}H_9BrO_2S$	F_{000} = 1232
M_r = 309.17	D_x = 1.740 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 17692 reflections
a = 13.2344 (8) Å	θ = 3.5–26.7º
b = 11.0471 (8) Å	μ = 3.64 mm^{-1}
c = 16.1466 (9) Å	*T* = 173 (2) K
V = 2360.7 (3) Å^3	Block, yellow
Z = 8	0.31 × 0.26 × 0.21 mm

Data collection

Stoe IPDSII two-circle diffractometer	2461 independent reflections
Radiation source: fine-focus sealed tube	2186 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.050
T = 173(2) K	θ_{max} = 26.6º
ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	*h* = −16→13
T_{min} = 0.398, T_{max} = 0.515	*k* = −13→13
18671 measured reflections	*l* = −20→20

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2 + 2.4425P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.066	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.07	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
2461 reflections	$\Delta\rho_{min}$ = −0.44 e Å^{-3}
159 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0129 (5)
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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	0.864761 (19)	0.01930 (2)	0.897108 (16)	0.02544 (11)
S1	0.79325 (4)	0.23561 (5)	0.78663 (4)	0.02105 (15)
O1	0.80436 (13)	0.61367 (15)	0.57092 (11)	0.0227 (4)
O2	0.32915 (13)	0.69338 (18)	0.52976 (12)	0.0272 (4)
H2O	0.318 (4)	0.761 (4)	0.499 (3)	0.077 (14)*
C1	0.73140 (17)	0.5591 (2)	0.60266 (14)	0.0170 (4)
C2	0.75165 (19)	0.4590 (2)	0.66109 (14)	0.0199 (5)
H2	0.6972	0.4235	0.6908	0.024*
C3	0.84604 (18)	0.41744 (19)	0.67284 (14)	0.0174 (5)
H3	0.8977	0.4551	0.6412	0.021*
C4	0.87774 (17)	0.3218 (2)	0.72835 (14)	0.0178 (5)
C5	0.97564 (18)	0.2833 (2)	0.74100 (14)	0.0191 (5)
H5	1.0323	0.3196	0.7147	0.023*
C6	0.98421 (19)	0.1849 (2)	0.79680 (14)	0.0202 (5)
H6	1.0463	0.1485	0.8126	0.024*
C7	0.89154 (19)	0.1490 (2)	0.82491 (14)	0.0195 (5)
C11	0.62570 (17)	0.5936 (2)	0.58404 (13)	0.0152 (4)
C12	0.60622 (18)	0.6967 (2)	0.53534 (14)	0.0178 (5)
H12	0.6612	0.7430	0.5146	0.021*
C13	0.50800 (17)	0.7317 (2)	0.51712 (13)	0.0169 (4)
H13	0.4962	0.8017	0.4844	0.020*
C14	0.42628 (17)	0.6641 (2)	0.54686 (13)	0.0176 (5)
C15	0.44386 (19)	0.5611 (2)	0.59508 (14)	0.0201 (5)
H15	0.3887	0.5147	0.6153	0.024*
C16	0.54232 (18)	0.5271 (2)	0.61317 (13)	0.0182 (5)
H16	0.5537	0.4571	0.6461	0.022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02815 (16)	0.02015 (15)	0.02802 (15)	0.00418 (10)	0.00377 (10)	0.00766 (10)
S1	0.0174 (3)	0.0206 (3)	0.0251 (3)	0.0030 (2)	0.0005 (2)	0.0052 (2)

O1	0.0151 (8)	0.0228 (9)	0.0303 (9)	−0.0014 (7)	0.0005 (7)	0.0082 (7)
O2	0.0135 (8)	0.0327 (10)	0.0355 (10)	0.0010 (7)	−0.0017 (7)	0.0147 (8)
C1	0.0174 (11)	0.0165 (10)	0.0171 (10)	0.0017 (8)	−0.0010 (9)	−0.0009 (9)
C2	0.0187 (11)	0.0192 (11)	0.0217 (11)	−0.0003 (9)	0.0005 (9)	0.0044 (9)
C3	0.0202 (12)	0.0135 (10)	0.0185 (11)	0.0005 (8)	−0.0018 (9)	−0.0012 (8)
C4	0.0171 (11)	0.0172 (11)	0.0191 (11)	0.0000 (8)	−0.0015 (9)	−0.0016 (8)
C5	0.0183 (11)	0.0178 (11)	0.0211 (11)	−0.0015 (9)	−0.0020 (9)	−0.0003 (9)
C6	0.0202 (11)	0.0204 (11)	0.0200 (11)	0.0044 (9)	−0.0034 (9)	−0.0002 (9)
C7	0.0243 (12)	0.0154 (10)	0.0189 (11)	0.0040 (9)	−0.0019 (9)	0.0005 (9)
C11	0.0156 (11)	0.0156 (10)	0.0144 (10)	0.0011 (8)	−0.0009 (8)	0.0003 (8)
C12	0.0167 (11)	0.0168 (10)	0.0200 (11)	−0.0018 (9)	0.0023 (9)	0.0021 (9)
C13	0.0168 (11)	0.0173 (10)	0.0167 (11)	0.0010 (8)	0.0003 (8)	0.0044 (8)
C14	0.0147 (11)	0.0209 (11)	0.0171 (10)	0.0009 (8)	−0.0003 (8)	0.0011 (9)
C15	0.0172 (11)	0.0212 (11)	0.0218 (11)	−0.0034 (9)	0.0019 (9)	0.0056 (9)
C16	0.0193 (11)	0.0169 (11)	0.0185 (11)	0.0003 (9)	−0.0007 (9)	0.0036 (9)

Geometric parameters (Å, °)

Br1—C7	1.881 (2)	C5—H5	0.9500
S1—C7	1.729 (2)	C6—C7	1.366 (4)
S1—C4	1.744 (2)	C6—H6	0.9500
O1—C1	1.249 (3)	C11—C16	1.407 (3)
O2—C14	1.354 (3)	C11—C12	1.408 (3)
O2—H2O	0.92 (5)	C12—C13	1.388 (3)
C1—C2	1.478 (3)	C12—H12	0.9500
C1—C11	1.481 (3)	C13—C14	1.399 (3)
C2—C3	1.344 (3)	C13—H13	0.9500
C2—H2	0.9500	C14—C15	1.398 (3)
C3—C4	1.448 (3)	C15—C16	1.387 (3)
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.379 (3)	C16—H16	0.9500
C5—C6	1.417 (3)		
C7—S1—C4	90.71 (11)	C6—C7—Br1	126.56 (18)
C14—O2—H2O	118 (3)	S1—C7—Br1	120.08 (14)
O1—C1—C2	118.9 (2)	C16—C11—C12	117.8 (2)
O1—C1—C11	121.5 (2)	C16—C11—C1	122.6 (2)
C2—C1—C11	119.6 (2)	C12—C11—C1	119.6 (2)
C3—C2—C1	120.9 (2)	C13—C12—C11	121.0 (2)
C3—C2—H2	119.5	C13—C12—H12	119.5
C1—C2—H2	119.5	C11—C12—H12	119.5
C2—C3—C4	127.3 (2)	C12—C13—C14	120.2 (2)
C2—C3—H3	116.4	C12—C13—H13	119.9
C4—C3—H3	116.4	C14—C13—H13	119.9
C5—C4—C3	126.1 (2)	O2—C14—C15	117.8 (2)
C5—C4—S1	110.76 (17)	O2—C14—C13	122.4 (2)
C3—C4—S1	123.13 (18)	C15—C14—C13	119.8 (2)
C4—C5—C6	113.9 (2)	C16—C15—C14	119.6 (2)
C4—C5—H5	123.0	C16—C15—H15	120.2
C6—C5—H5	123.0	C14—C15—H15	120.2

C7—C6—C5	111.2 (2)	C15—C16—C11	121.7 (2)
C7—C6—H6	124.4	C15—C16—H16	119.2
C5—C6—H6	124.4	C11—C16—H16	119.2
C6—C7—S1	113.35 (17)		
O1—C1—C2—C3	−7.7 (3)	O1—C1—C11—C16	174.3 (2)
C11—C1—C2—C3	173.2 (2)	C2—C1—C11—C16	−6.6 (3)
C1—C2—C3—C4	179.1 (2)	O1—C1—C11—C12	−5.7 (3)
C2—C3—C4—C5	−177.9 (2)	C2—C1—C11—C12	173.4 (2)
C2—C3—C4—S1	4.2 (3)	C16—C11—C12—C13	0.3 (3)
C7—S1—C4—C5	−0.92 (18)	C1—C11—C12—C13	−179.7 (2)
C7—S1—C4—C3	177.3 (2)	C11—C12—C13—C14	−0.3 (3)
C3—C4—C5—C6	−177.9 (2)	C12—C13—C14—O2	−179.0 (2)
S1—C4—C5—C6	0.3 (3)	C12—C13—C14—C15	0.0 (3)
C4—C5—C6—C7	0.8 (3)	O2—C14—C15—C16	179.2 (2)
C5—C6—C7—S1	−1.5 (3)	C13—C14—C15—C16	0.1 (3)
C5—C6—C7—Br1	177.93 (17)	C14—C15—C16—C11	−0.1 (4)
C4—S1—C7—C6	1.41 (19)	C12—C11—C16—C15	−0.1 (3)
C4—S1—C7—Br1	−178.06 (14)	C1—C11—C16—C15	179.9 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2O···O1^{i}	0.92 (5)	1.79 (5)	2.701 (3)	173 (5)

Symmetry codes: (i) *x*−1/2, −*y*+3/2, −*z*+1.

Fig. 1