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4-(5-Bromo-2-hydroxyphenyl)but-3-ene-2-one

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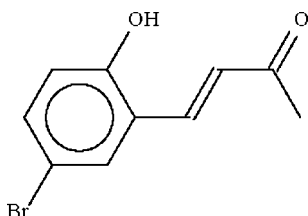
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Key indicators: single-crystal X-ray study; $T = 123$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.031; wR factor = 0.074; data-to-parameter ratio = 16.6.

The molecule of the title compound, $\text{C}_{10}\text{H}_9\text{BrO}_2$, a doubly conjugated unsaturated ketone, is almost planar (r.m.s. deviation of the non-H atoms = 0.039 Å). In the crystal structure, two molecules are linked across a centre of inversion to form a hydrogen-bonded dimer by way of two $\text{O}-\text{H}\cdots\text{O}$ links.

Related literature

The reactivity of doubly conjugated unsaturated ketones has been known for a long time; see: Buck & Heilbron (1922); Marvel *et al.* (1953). Their utility is discussed by Trost & Fleming (1991).



Experimental

Crystal data

$\text{C}_{10}\text{H}_9\text{BrO}_2$
 $M_r = 241.08$

Triclinic, $P\bar{1}$
 $a = 5.8619$ (2) Å

$b = 7.7495$ (2) Å
 $c = 10.9601$ (3) Å
 $\alpha = 106.432$ (2)°
 $\beta = 104.548$ (2)°
 $\gamma = 94.468$ (2)°
 $V = 456.25$ (2) Å³

$Z = 2$
Mo $K\alpha$ radiation
 $\mu = 4.47$ mm⁻¹
 $T = 123$ K
 $0.40 \times 0.10 \times 0.02$ mm

Data collection

Bruker SMART APEX
diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.268$, $T_{\max} = 0.916$

3659 measured reflections
2040 independent reflections
1797 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.074$
 $S = 1.04$
2040 reflections
123 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.66$ e Å⁻³
 $\Delta\rho_{\min} = -0.58$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.83 (1)	1.87 (1)	2.689 (3)	168 (4)

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *S SAINT* (Bruker, 2008); data reduction: *S SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2898).

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supplementary materials

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4-(5-Bromo-2-hydroxyphenyl)but-3-ene-2-one

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Comment

(type here to add)

Experimental

To a stirred solution of 5-bromo-2-hydroxy-benzaldehyde (1.01 g, 5 mmol) in acetone (50 ml) a solution of sodium hydroxide (1.25 g, 6 mmol) in water (20 ml) was added. The reaction was stirred for another 6 h before being neutralized with strong hydrochloric acid to a pH of 6. The organic phase was washed by aqueous solution of sodium bisulfate (40%). After removing the solvent, the light green powder recrystallized from dichloromethane to give colorless plates (yield 85%, m.p. 421 K).

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to 1.2 to 1.5 $U(\text{C})$.

The hydroxy H-atom was located in a difference Fourier map, and was refined with a distance restraint of O–H 0.84±0.01 Å.

Figures

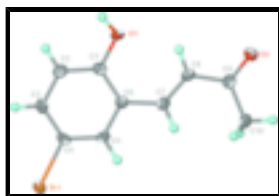


Fig. 1. Anisotropic displacement ellipsoid plot (Barbour, 2001) of $\text{C}_{10}\text{H}_9\text{BrNO}_2$; probability levels are set at 70% and H-atoms are drawn as spheres of arbitrary radius.

4-(5-Bromo-2-hydroxyphenyl)but-3-ene-2-one

Crystal data

$\text{C}_{10}\text{H}_9\text{BrO}_2$
 $M_r = 241.08$
Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.8619(2)$ Å
 $b = 7.7495(2)$ Å
 $c = 10.9601(3)$ Å

$Z = 2$

$F_{000} = 240$

$D_x = 1.755$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å

Cell parameters from 1830 reflections

$\theta = 2.8\text{--}28.1^\circ$

$\mu = 4.47$ mm⁻¹

supplementary materials

$\alpha = 106.432 (2)^\circ$
 $\beta = 104.548 (2)^\circ$
 $\gamma = 94.468 (2)^\circ$
 $V = 456.25 (2) \text{ \AA}^3$

$T = 123 \text{ K}$
Plate, colorless
 $0.40 \times 0.10 \times 0.02 \text{ mm}$

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
 $T = 123 \text{ K}$
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.268, T_{\max} = 0.916$
3659 measured reflections

2040 independent reflections
1797 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\max} = 27.5^\circ$
 $\theta_{\min} = 2.0^\circ$
 $h = -7 \rightarrow 7$
 $k = -10 \rightarrow 9$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.074$
 $S = 1.04$
2040 reflections
123 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 0.4716P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.66 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.58 \text{ e \AA}^{-3}$
Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.69822 (5)	0.19383 (4)	-0.18504 (3)	0.02165 (11)
O1	0.3724 (4)	0.3898 (3)	0.3090 (2)	0.0231 (5)
H1	0.256 (4)	0.319 (4)	0.307 (4)	0.033 (11)*
O2	0.9820 (4)	0.8201 (3)	0.6596 (2)	0.0226 (5)
C1	0.4405 (5)	0.3456 (4)	0.1967 (3)	0.0173 (6)
C2	0.2974 (5)	0.2178 (4)	0.0796 (3)	0.0188 (6)
H2	0.1468	0.1611	0.0782	0.023*
C3	0.3699 (5)	0.1715 (4)	-0.0348 (3)	0.0192 (6)
H3	0.2722	0.0828	-0.1138	0.023*
C4	0.5894 (5)	0.2583 (4)	-0.0309 (3)	0.0173 (6)
C5	0.7326 (5)	0.3867 (4)	0.0822 (3)	0.0168 (6)

H5	0.8812	0.4441	0.0815	0.020*
C6	0.6617 (5)	0.4341 (4)	0.1992 (3)	0.0166 (6)
C7	0.8291 (5)	0.5694 (4)	0.3152 (3)	0.0178 (6)
H7	0.9703	0.6173	0.3004	0.021*
C8	0.8136 (5)	0.6359 (4)	0.4390 (3)	0.0180 (6)
H8	0.6731	0.5958	0.4582	0.022*
C10	1.2237 (6)	0.8416 (4)	0.5192 (3)	0.0248 (7)
H10A	1.3292	0.9287	0.6014	0.037*
H10B	1.1786	0.9032	0.4521	0.037*
H10C	1.3071	0.7412	0.4868	0.037*
C9	1.0023 (5)	0.7671 (4)	0.5460 (3)	0.0177 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02299 (17)	0.02469 (17)	0.01502 (15)	−0.00029 (11)	0.00787 (11)	0.00169 (11)
O1	0.0206 (11)	0.0277 (12)	0.0199 (11)	−0.0035 (9)	0.0101 (9)	0.0037 (9)
O2	0.0263 (12)	0.0215 (11)	0.0178 (10)	−0.0001 (9)	0.0082 (9)	0.0025 (9)
C1	0.0201 (15)	0.0182 (14)	0.0161 (14)	0.0048 (11)	0.0067 (12)	0.0073 (11)
C2	0.0198 (15)	0.0189 (14)	0.0195 (14)	0.0013 (12)	0.0071 (12)	0.0080 (12)
C3	0.0196 (15)	0.0188 (14)	0.0160 (14)	0.0021 (12)	0.0023 (12)	0.0035 (11)
C4	0.0213 (15)	0.0173 (14)	0.0165 (13)	0.0068 (12)	0.0099 (12)	0.0054 (11)
C5	0.0147 (14)	0.0193 (14)	0.0189 (14)	0.0032 (11)	0.0066 (11)	0.0079 (11)
C6	0.0179 (14)	0.0157 (14)	0.0158 (13)	0.0031 (11)	0.0047 (11)	0.0047 (11)
C7	0.0157 (14)	0.0193 (14)	0.0189 (14)	0.0026 (11)	0.0043 (12)	0.0074 (12)
C8	0.0153 (14)	0.0181 (14)	0.0205 (14)	0.0011 (11)	0.0057 (12)	0.0056 (12)
C10	0.0215 (16)	0.0251 (16)	0.0223 (15)	−0.0054 (13)	0.0070 (13)	0.0005 (13)
C9	0.0205 (15)	0.0150 (14)	0.0179 (14)	0.0025 (11)	0.0059 (12)	0.0056 (11)

Geometric parameters (\AA , $^\circ$)

Br1—C4	1.903 (3)	C5—C6	1.406 (4)
O1—C1	1.350 (3)	C5—H5	0.9500
O1—H1	0.834 (10)	C6—C7	1.461 (4)
O2—C9	1.235 (3)	C7—C8	1.338 (4)
C1—C2	1.394 (4)	C7—H7	0.9500
C1—C6	1.409 (4)	C8—C9	1.460 (4)
C2—C3	1.385 (4)	C8—H8	0.9500
C2—H2	0.9500	C10—C9	1.508 (4)
C3—C4	1.390 (4)	C10—H10A	0.9800
C3—H3	0.9500	C10—H10B	0.9800
C4—C5	1.371 (4)	C10—H10C	0.9800
C1—O1—H1	114 (3)	C5—C6—C7	116.5 (3)
O1—C1—C2	121.9 (3)	C1—C6—C7	125.4 (3)
O1—C1—C6	118.3 (3)	C8—C7—C6	129.8 (3)
C2—C1—C6	119.8 (3)	C8—C7—H7	115.1
C3—C2—C1	121.5 (3)	C6—C7—H7	115.1
C3—C2—H2	119.2	C7—C8—C9	123.1 (3)

supplementary materials

C1—C2—H2	119.2	C7—C8—H8	118.5
C2—C3—C4	118.2 (3)	C9—C8—H8	118.5
C2—C3—H3	120.9	C9—C10—H10A	109.5
C4—C3—H3	120.9	C9—C10—H10B	109.5
C5—C4—C3	121.6 (3)	H10A—C10—H10B	109.5
C5—C4—Br1	119.0 (2)	C9—C10—H10C	109.5
C3—C4—Br1	119.3 (2)	H10A—C10—H10C	109.5
C4—C5—C6	120.7 (3)	H10B—C10—H10C	109.5
C4—C5—H5	119.6	O2—C9—C8	120.5 (3)
C6—C5—H5	119.6	O2—C9—C10	119.0 (3)
C5—C6—C1	118.1 (3)	C8—C9—C10	120.5 (3)
O1—C1—C2—C3	-179.2 (2)	O1—C1—C6—C5	179.6 (2)
C6—C1—C2—C3	1.6 (4)	C2—C1—C6—C5	-1.2 (4)
C1—C2—C3—C4	-1.0 (4)	O1—C1—C6—C7	1.2 (4)
C2—C3—C4—C5	0.0 (4)	C2—C1—C6—C7	-179.5 (3)
C2—C3—C4—Br1	178.5 (2)	C5—C6—C7—C8	-177.8 (3)
C3—C4—C5—C6	0.4 (4)	C1—C6—C7—C8	0.6 (5)
Br1—C4—C5—C6	-178.10 (19)	C6—C7—C8—C9	177.6 (3)
C4—C5—C6—C1	0.2 (4)	C7—C8—C9—O2	-177.8 (3)
C4—C5—C6—C7	178.7 (2)	C7—C8—C9—C10	2.9 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O2 ⁱ	0.83 (1)	1.87 (1)	2.689 (3)	168 (4)

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

Fig. 1

