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ISSN 2414-3146

(*E*)-1-(4-Bromophenyl)but-2-en-1-one

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Received 30 April 2025
Accepted 8 May 2025

Edited by M. Weil, Vienna University of Technology, Austria

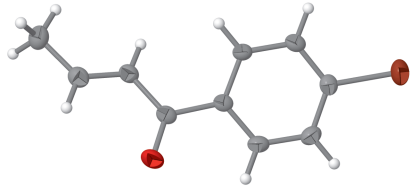
Keywords: crystal structure; unsaturated compound; carbonyl side chain; stacking interactions.

CCDC reference: 1495201

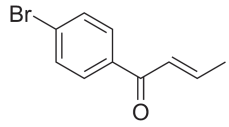
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{10}H_9BrO$, consists of a *para*-substituted bromophenyl core and an unsaturated carbonyl side chain. The angle between the plane through the carbon atoms of the aryl ring and the plane through the carbon atoms of the unsaturated side chain is 29.12 (16)°. The cohesion in the crystal is ensured by π–π stacking and C—H···O interactions.

3D view



Chemical scheme



Structure description

The structures of α,β-unsaturated carbonyl compounds are a common motif in a variety of natural products or bulk chemicals. These compounds are versatile synthetic intermediates for multiple organic transformation reactions, such as Michael addition, Diels–Alder reaction or Heck reaction (Ponec, 1997; Engel & Dudley, 2009; Desimoni *et al.*, 2018). The title compound, $C_{10}H_9BrO$, was received in low yield in high purity in a Friedel–Crafts acylation. It can be designated as a suitable building block in the ongoing efforts to synthesize feasible new ligands for Cu-based complexes (Sonneck *et al.*, 2015, 2016).

The molecular structure of the title compound consists of a *para*-substituted bromophenyl core and an unsaturated carbonyl side chain (Fig. 1). The angle between the plane defined by the aryl ring (C5–C10) and the plane through the carbon atoms of the unsaturated side chain (C1–C4) is 29.12 (16)°. Carbonyl oxygen atom O1 is 0.246 (4) Å out of the latter plane. In the crystal, weak π–π stacking interactions between adjacent molecules are observed, with a centroid(C5–C10)-to-centroid(C5–C10)' distance of 3.724 (1) Å [ring slippage = 1.31 Å; symmetry code: (') $1-x, 2-y, 2-z$]. Additionally, weak intermolecular C—H···O interactions are present in the crystal packing (Table 1, Fig. 2). All bond lengths and angles are in expected ranges and the C=O bond length is 1.2278 (17) Å.

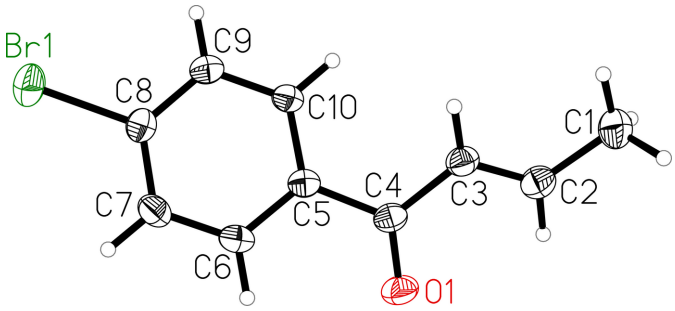


Figure 1
Molecular structure of the title compound with atom labeling and displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

The title compound, $C_{10}H_9BrO$, was obtained as colorless crystals in low yield from the Friedel–Crafts acylation of bromobenzene and crotonyl chloride in CS_2. $AlCl_3$ (61.2 g, 459.2 mmol, 1.20 eq.) was added to a stirred solution of bromobenzene (81.1 g, 516.6 mmol, 1.35 eq.) in 150 ml of CS_2 at room temperature. Crotonyl chloride (40.0 g, 382.7 mmol, 1.00 eq.) was added dropwise to the thoroughly stirred suspension and afterwards the solution was heated under reflux for 24 h. The resulting red solution was poured onto a mixture of ice and concentrated hydrochloric acid (500 g: 50 g) and extracted 3× with 150 ml portions of ethyl acetate. The volume of the combined organic phases was reduced to 150 ml and extracted twice with 100 ml portions of brine. The organic phase was dried with Na_2SO_4 and the solvent was removed completely under diminished pressure. The resulting raw product was distilled under reduced pressure to give an orange-colored distillate. After storing the distillate for several days at 243 K, colorless single crystals of the product were obtained in low yield (9.5 g, 11%).

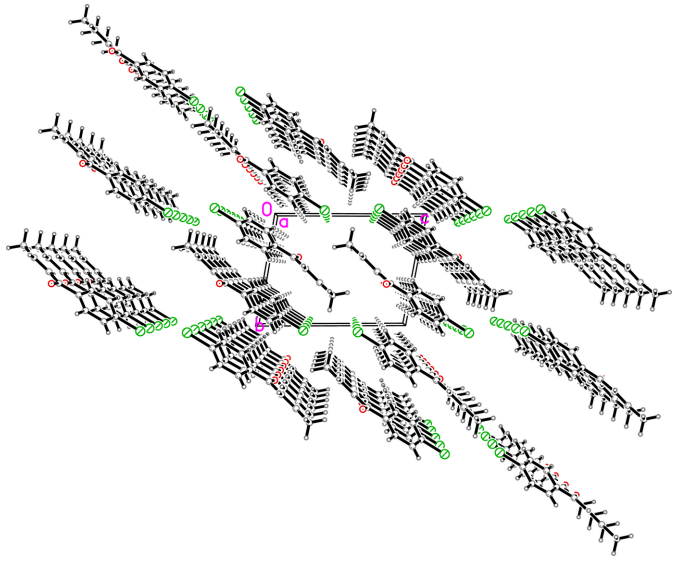


Figure 2
Packing diagram (ball-and-stick representation) for the title compound in a view along [100].

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.95	2.56	3.5094 (18)	177
C1—H1C···O1^{ii}	0.98	2.58	3.555 (2)	172

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_9BrO$
M_r	225.08
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	5.5734 (9), 8.1618 (13), 10.6194 (16)
α, β, γ (°)	98.577 (2), 96.441 (2), 102.546 (2)
V (Å^3)	461.00 (13)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	4.41
Crystal size (mm)	0.36 × 0.23 × 0.04
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.63, 0.85
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12194, 2232, 2077
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.661
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.019, 0.051, 1.05
No. of reflections	2232
No. of parameters	110
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.27

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

Analytical data for $C_{10}H_9BrO$: mp. 323 K, elemental analysis % (calculated): C 53.40 (53.36), H 3.95 (4.03); Br 35.42 (35.50). ^{1}H NMR (300 MHz, MeOD): δ (p.p.m.) = 8.75–8.67 (*m*, 2H, ArH); 8.56–8.48 (*m*, 2H, ArH); 8.01–7.81 (*m*, 2H); 2.87 (*d*, ^3J = 6.0 Hz, 3H, –Me); ^{13}C NMR (75 MHz, MeOD): δ (p.p.m.) = 191.15 (CO); 147.25 (CH); 137.84 (C); 132.98, 132.98, 141.29, 131.29 (CH), 128.78 (C), 127.97 (CH), 18.67 (CH_3).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2025). **10**, x250415 [https://doi.org/10.1107/S2414314625004158]

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Crystal data

$C_{10}H_9BrO$
M_r = 225.08
Triclinic, $P\overline{1}$
a = 5.5734 (9) Å
b = 8.1618 (13) Å
c = 10.6194 (16) Å
α = 98.577 (2)°
β = 96.441 (2)°
γ = 102.546 (2)°
V = 461.00 (13) Å^3
Z = 2
$F(000)$ = 224
D_x = 1.622 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7348 reflections
θ = 2.6–28.6°
μ = 4.41 mm^{-1}
T = 150 K
Plate, colourless
0.36 × 0.23 × 0.04 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.63, T_{max} = 0.85
12194 measured reflections
2232 independent reflections
2077 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 28.0°, θ_{min} = 2.0°
h = −7→7
k = −10→10
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.051
S = 1.05
2232 reflections
110 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0243P)^2 + 0.1552P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.46488 (3)	1.00661 (2)	1.33348 (2)	0.03862 (7)
C1	0.1030 (3)	0.2949 (2)	0.52287 (17)	0.0397 (4)
H1A	−0.047264	0.328231	0.545930	0.060*
H1B	0.086027	0.173332	0.524923	0.060*
H1C	0.125813	0.316143	0.436060	0.060*
C2	0.3226 (3)	0.39689 (19)	0.61669 (15)	0.0308 (3)
H2	0.480779	0.377596	0.603523	0.037*
C3	0.3146 (3)	0.51246 (19)	0.71713 (14)	0.0274 (3)
H3	0.158932	0.534668	0.732273	0.033*
C4	0.5417 (3)	0.60755 (18)	0.80609 (14)	0.0253 (3)
C5	0.5149 (2)	0.70343 (17)	0.93299 (13)	0.0230 (3)
C6	0.7179 (3)	0.82873 (19)	1.00146 (15)	0.0284 (3)
H6	0.867758	0.852042	0.965647	0.034*
C7	0.7057 (3)	0.91961 (19)	1.12013 (15)	0.0304 (3)
H7	0.843908	1.006229	1.165242	0.037*
C8	0.4872 (3)	0.88158 (18)	1.17190 (13)	0.0260 (3)
C9	0.2838 (3)	0.75609 (19)	1.10791 (14)	0.0276 (3)
H9	0.136975	0.729938	1.145979	0.033*
C10	0.2970 (2)	0.66900 (18)	0.98747 (14)	0.0257 (3)
H10	0.156462	0.585051	0.941560	0.031*
O1	0.75060 (19)	0.60866 (15)	0.77924 (11)	0.0348 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.05075 (11)	0.03740 (10)	0.02638 (9)	0.01081 (7)	0.00651 (7)	0.00052 (6)
C1	0.0426 (9)	0.0367 (9)	0.0352 (8)	0.0058 (7)	0.0048 (7)	−0.0022 (7)
C2	0.0319 (7)	0.0300 (7)	0.0329 (8)	0.0098 (6)	0.0087 (6)	0.0068 (6)
C3	0.0246 (7)	0.0318 (7)	0.0276 (7)	0.0090 (6)	0.0066 (5)	0.0061 (6)
C4	0.0243 (6)	0.0268 (7)	0.0282 (7)	0.0091 (5)	0.0071 (5)	0.0086 (5)
C5	0.0212 (6)	0.0244 (6)	0.0259 (6)	0.0074 (5)	0.0045 (5)	0.0085 (5)
C6	0.0207 (6)	0.0317 (7)	0.0326 (7)	0.0035 (5)	0.0053 (5)	0.0083 (6)
C7	0.0256 (7)	0.0310 (7)	0.0302 (7)	0.0001 (6)	−0.0005 (5)	0.0044 (6)
C8	0.0310 (7)	0.0265 (7)	0.0219 (6)	0.0093 (6)	0.0032 (5)	0.0059 (5)
C9	0.0249 (6)	0.0307 (7)	0.0291 (7)	0.0063 (6)	0.0082 (5)	0.0083 (6)
C10	0.0210 (6)	0.0261 (7)	0.0287 (7)	0.0027 (5)	0.0049 (5)	0.0047 (5)
O1	0.0239 (5)	0.0467 (7)	0.0360 (6)	0.0114 (5)	0.0098 (4)	0.0063 (5)

Geometric parameters (Å, º)

Br1—C8	1.8925 (14)	C5—C6	1.395 (2)
C1—C2	1.490 (2)	C5—C10	1.3951 (18)
C1—H1A	0.9800	C6—C7	1.380 (2)
C1—H1B	0.9800	C6—H6	0.9500
C1—H1C	0.9800	C7—C8	1.385 (2)

C2—C3	1.326 (2)	C7—H7	0.9500
C2—H2	0.9500	C8—C9	1.382 (2)
C3—C4	1.478 (2)	C9—C10	1.385 (2)
C3—H3	0.9500	C9—H9	0.9500
C4—O1	1.2278 (17)	C10—H10	0.9500
C4—C5	1.4940 (19)		

C2—C1—H1A	109.5	C10—C5—C4	122.52 (13)
C2—C1—H1B	109.5	C7—C6—C5	121.43 (13)
H1A—C1—H1B	109.5	C7—C6—H6	119.3
C2—C1—H1C	109.5	C5—C6—H6	119.3
H1A—C1—H1C	109.5	C6—C7—C8	118.46 (14)
H1B—C1—H1C	109.5	C6—C7—H7	120.8
C3—C2—C1	125.10 (14)	C8—C7—H7	120.8
C3—C2—H2	117.4	C9—C8—C7	121.73 (13)
C1—C2—H2	117.4	C9—C8—Br1	119.11 (11)
C2—C3—C4	121.66 (13)	C7—C8—Br1	119.16 (11)
C2—C3—H3	119.2	C8—C9—C10	119.06 (13)
C4—C3—H3	119.2	C8—C9—H9	120.5
O1—C4—C3	122.00 (13)	C10—C9—H9	120.5
O1—C4—C5	119.30 (13)	C9—C10—C5	120.62 (13)
C3—C4—C5	118.69 (12)	C9—C10—H10	119.7
C6—C5—C10	118.67 (13)	C5—C10—H10	119.7
C6—C5—C4	118.78 (12)		

C1—C2—C3—C4	179.68 (14)	C5—C6—C7—C8	1.2 (2)
C2—C3—C4—O1	13.9 (2)	C6—C7—C8—C9	0.0 (2)
C2—C3—C4—C5	−165.45 (14)	C6—C7—C8—Br1	−179.19 (11)
O1—C4—C5—C6	17.9 (2)	C7—C8—C9—C10	−1.6 (2)
C3—C4—C5—C6	−162.71 (13)	Br1—C8—C9—C10	177.61 (10)
O1—C4—C5—C10	−160.10 (14)	C8—C9—C10—C5	2.0 (2)
C3—C4—C5—C10	19.2 (2)	C6—C5—C10—C9	−0.8 (2)
C10—C5—C6—C7	−0.8 (2)	C4—C5—C10—C9	177.24 (13)
C4—C5—C6—C7	−178.94 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.95	2.56	3.5094 (18)	177
C1—H1*C*···O1^{ii}	0.98	2.58	3.555 (2)	172

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