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1-(4-Methacryloyloxyphenyl)-3-(3-bromophenyl)prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.011 Å
R factor = 0.069
wR factor = 0.169
Data-to-parameter ratio = 16.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title molecule, $C_{15}H_{15}BrO_3$, has an *s-cis* conformation for the ketone system. The two phenyl rings form a dihedral angle of 11.0 (4)°, and the methacryloyloxy group is inclined to the attached phenyl ring by 61.5 (4)°. The structure is stabilized by C—H$\cdots\pi$ interactions and van der Waals interactions.

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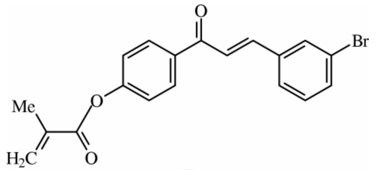
Comment

Chalcones, which are α,β-unsaturated ketones, possess multi-protecting biochemical activities, including antibacterial, antifungal, sedative, germicidal, cardiovascular, antifertility *etc*. It has been proven that halogen substitution at the benzene nucleus enhances greatly the activity of chalcones. In recent years, the synthesis of polymers having a photosensitive functional group has been an active field of research in polymer science. Monomers having structures similar to the title compound, $C_{15}H_{15}BrO_3$, have been polymerized in sol ution using free radical initiators (Balaji & Nanjundan, 2001; Subramanian *et al.*, 2001). These polymers undergo cross-linking upon irradiation with UV light or an electron beam and are being used as photoresistors (Hyder Ali & Srinivasan, 1997; Rehab & Salahuddin, 1999). These photosensitive polymers find application in the fields of integrated circuit technology and photocurable coatings (Nagamatzu & Inui, 1977). The structure determination of the title compound was undertaken as part of our study of chalcones.



(I)

In the title molecule, the spatial arrangement of the keto group and the olefenic double bond about the linking single bond is *s-cis*, as seen from the C7—C8—C9—O1 torsion angle of −7.7 (13)° (Fig. 1). A similar conformation has been reported previously for related structures (Raj *et al.*, 1996, 1997; Jeyabharathi *et al.*, 2002). The unsaturated ketone system is not strictly planar. The two phenyl rings and the unsaturated ketone system together form a curved structure, with a dihedral angle of 11.0 (4)° between the two phenyl rings. This deviation from planarity affects the π-electron conjugation. The mean plane through the methacryloyloxy group forms a dihedral angle of 61.5 (4)° with the attached phenyl ring. The widening of the C5—C6—C7 angle to 124.1 (7)° and the C6—C7—C8 angle to 128.7 (7)° can be ascribed to the short interatomic contact between atoms H5 and H8 (2.35 Å). Also, the strain induced by the short

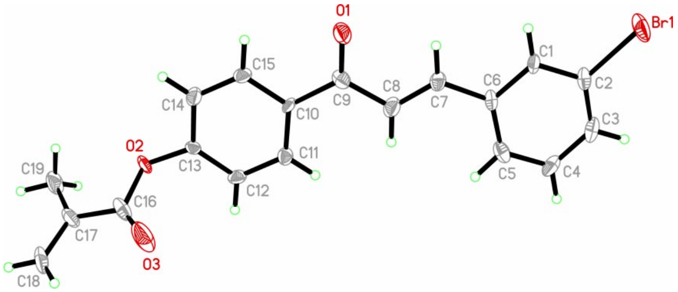


Figure 1
A view of the title molecule, with the atomic numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

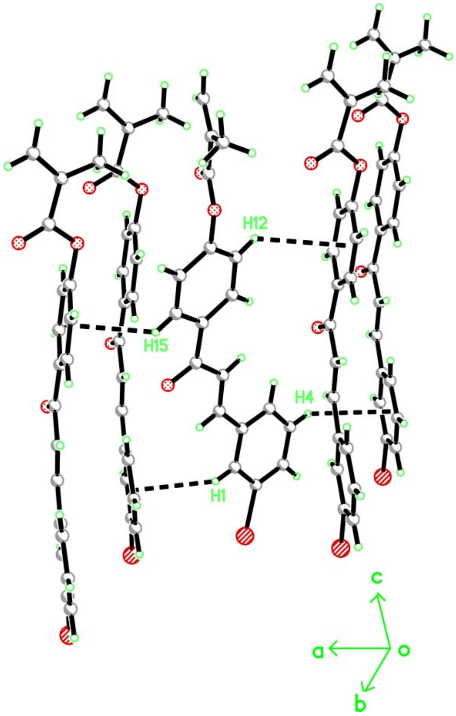


Figure 2
A view of the C—H···π interactions in the title compound.

H8···H11 [2.17 Å] contact has resulted in a slight opening of the C9—C10—C11 angle to 123.2 (6)°. In the methacryloyloxy group, the two double bonds are in the *s-cis* conformation, whereas in two closely related structures, they adopt an *s-trans* conformation (Jeyabharathi *et al.*, 2002). Steric interactions cause the bond angles around atoms C16 and C17 to deviate significantly from 120°. Weak intramolecular C—H···O interactions involving the ketone O atom, O1, are observed. The molecular packing in the solid state is stabilized by weak C—H···π interactions involving H1, H4, H12 and H15 and the phenyl rings of symmetry-related molecules (Table 2). A view of the C—H···π bonded molecular chain is shown in Fig. 2. In Table 2, *CgA* and *CgB* denote the centroids of the phenyl rings C1–C6 and C10–C15, respectively.

Experimental

The synthesis of the title compound consisted of three steps. Methacryloyl chloride was prepared according to the method of Stampel *et al.* (1950). 3-Bromostyryl-4′-hydroxyphenyl ketone was prepared by the Claissen–Schmidt condensation of 3-bromobenzaldehyde in aqueous alcohol in the presence of sodium hydroxide. The title compound was prepared by reacting 3-bromostyryl-4′-hydroxyphenyl ketone with methacryloyl chloride in methyl ethyl ketone in the presence of triethylamine. Single crystals suitable for X-ray diffraction were grown from a 1:1 methanol–chloroform mixture.

Crystal data

$C_{19}H_{15}BrO_3$
$M_r = 371.22$
Orthorhombic, $Pna2_1$
$a = 7.8096$ (2) Å
$b = 5.6168$ (2) Å
$c = 37.212$ (1) Å
$V = 1632.30$ (8) Å^3
$Z = 4$
$D_x = 1.511$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3329 reflections
$\theta = 3.3$–28.3°
$\mu = 2.53$ mm^{-1}
$T = 203$ (2) K
Slab, colorless
0.40 × 0.28 × 0.04 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.431$, $T_{\max} = 0.906$
9099 measured reflections
3381 independent reflections
1860 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.129$
$\theta_{\max} = 28.3°$
$h = -9 \rightarrow 10$
$k = -6 \rightarrow 7$
$l = -49 \rightarrow 43$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.169$
$S = 0.93$
3381 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0757P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.68$ e Å^{-3}
$\Delta\rho_{\min} = -1.04$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0196 (19)
Absolute structure: Flack (1983), 1345 Friedel pairs
Flack parameter = 0.02 (2)

Table 1
Selected geometric parameters (Å, °).

C5—C6—C7	124.1 (7)	C14—C13—O2	115.3 (6)
C8—C7—C6	128.7 (7)	O3—C16—O2	123.3 (7)
C15—C10—C9	117.1 (6)	O3—C16—C17	127.2 (7)
C11—C10—C9	123.2 (6)	O2—C16—C17	109.4 (7)
C12—C13—C14	122.4 (7)	C18—C17—C19	124.8 (8)
C12—C13—O2	122.2 (7)	C18—C17—C16	115.2 (8)
C1—C6—C7—C8	175.1 (8)	C7—C8—C9—C10	172.6 (7)
C6—C7—C8—C9	−179.0 (7)	C8—C9—C10—C15	−176.5 (7)
C7—C8—C9—O1	−7.7 (13)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O1	0.93	2.38	2.748 (10)	104
C15—H15···O1	0.93	2.44	2.757 (10)	100
C1—H1···*CgA*$^{\rm i}$	0.93	2.95	3.690 (8)	138
C4—H4···*CgA*$^{\rm ii}$	0.93	2.87	3.541 (8)	130
C12—H12···*CgB*$^{\rm iii}$	0.93	3.00	3.670 (8)	130
C15—H15···*CgB*$^{\rm iv}$	0.93	2.90	3.582 (8)	131

Symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}-y, z$; (ii) $x-\frac{1}{2}, -\frac{1}{2}-y, z$; (iii) $x-\frac{1}{2}, \frac{1}{2}-y, z$; (iv) $\frac{1}{2}+x, \frac{3}{2}-y, z$.

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 or 0.96 Å and $U_{\rm iso}$(H) = 1.5 $U_{\rm eq}$(C) for methyl H and 1.2 $U_{\rm eq}$(C) for all others. Rotating group refinement

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was used for the methyl group. The reflections (019) and (1,2,31) were removed during refinement, as the observed and calculated structure factors showed large disagreement. The high $R_{\rm int}$ value (0.129) and low precision of the geometric parameters may be a result of the poor diffraction quality of the crystal. The deepest residual electron density hole occurs at 0.96 Å from Br1.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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