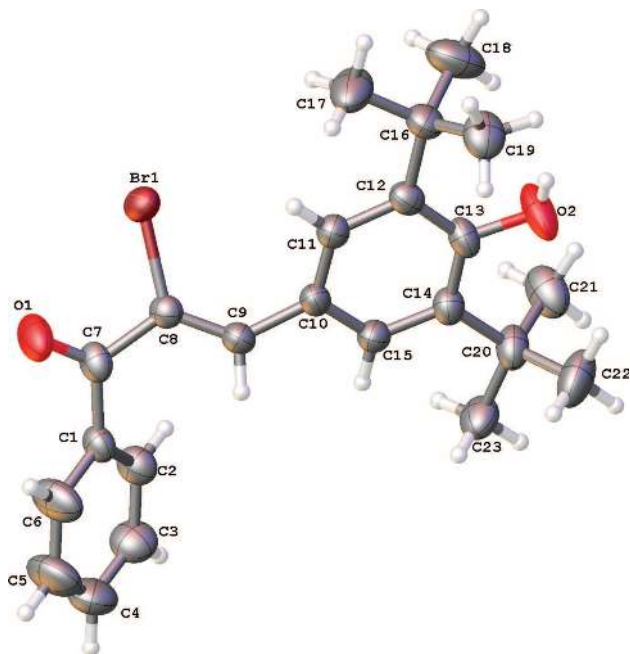


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Crystal structure of (Z)-2-bromo-3-(3,5-di-tert-butyl-4-hydroxyphenyl)-1-phenylprop-2-en-1-one, $C_{23}H_{27}BrO_2$



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Abstract

$C_{23}H_{27}BrO_2$, $P2_1/n$ (no. 14), $a = 10.3200(18)$ Å, $b = 15.905(3)(6)$ Å, $c = 12.913(2)$ Å, $\beta = 97.683(4)$, $V = 2100.6(6)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0319$, $wR_{ref}(F^2) = 0.0872$, $T = 296(2)$ K.

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Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colorless prism
Size:	0.17 × 0.15 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.97 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	27.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	21854, 4591, 0.022
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3732
$N(param)_{refined}$:	241
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
Br1	0.72766(2)	0.11800(2)	0.95063(2)	0.04809(9)
O1	0.68221(17)	0.02921(12)	0.74815(15)	0.0646(5)
O2	0.2748(2)	0.31050(11)	1.21175(16)	0.0750(6)
H2O	0.3136	0.3584	1.2133	0.113*
C1	0.4684(2)	-0.02245(13)	0.73662(15)	0.0396(4)
C2	0.3873(2)	-0.06645(14)	0.79337(17)	0.0468(5)
H2A	0.3974	-0.0614	0.8658	0.056*
C3	0.2910(3)	-0.11803(16)	0.7434(2)	0.0629(7)
H3A	0.2366	-0.1474	0.7824	0.075*
C4	0.2749(3)	-0.12634(19)	0.6372(3)	0.0729(8)
H4A	0.2095	-0.1609	0.6039	0.087*
C5	0.3556(4)	-0.0836(2)	0.5804(2)	0.0854(10)
H5A	0.3455	-0.0893	0.5081	0.102*
C6	0.4512(3)	-0.0323(2)	0.6291(2)	0.0727(8)
H6A	0.5057	-0.0036	0.5894	0.087*
C7	0.5797(2)	0.03023(13)	0.78504(16)	0.0415(5)
C8	0.56493(19)	0.08382(13)	0.87725(15)	0.0364(4)
C9	0.4505(2)	0.10827(13)	0.90392(16)	0.0388(4)
H9A	0.3790	0.0857	0.8615	0.047*
C10	0.41368(19)	0.16345(13)	0.98636(15)	0.0375(4)
C11	0.4870(2)	0.23193(13)	1.02806(16)	0.0401(4)
H11A	0.5658	0.2439	1.0036	0.048*
C12	0.4467(2)	0.28288(13)	1.10493(16)	0.0394(4)
C13	0.3266(2)	0.26307(13)	1.13903(16)	0.0422(5)
C14	0.2498(2)	0.19411(13)	1.10084(16)	0.0408(5)
C15	0.2956(2)	0.14626(14)	1.02373(17)	0.0400(4)

Table 2 (continued)

Atom	x	y	z	U _{iso} */U _{eq}
H15A	0.2458	0.1009	0.9958	0.048*
C16	0.5316(2)	0.35910(15)	1.14972(19)	0.0489(5)
C17	0.6586(3)	0.3630(2)	1.1025(3)	0.0834(10)
H17A	0.7092	0.4103	1.1310	0.125*
H17B	0.6395	0.3691	1.0281	0.125*
H17C	0.7074	0.3122	1.1187	0.125*
C18	0.5654(4)	0.3497(2)	1.2696(3)	0.0868(10)
H18A	0.5946	0.4028	1.2994	0.130*
H18B	0.6335	0.3087	1.2850	0.130*
H18C	0.4891	0.3319	1.2987	0.130*
C19	0.4611(3)	0.44145(15)	1.1238(2)	0.0626(7)
H19A	0.5225	0.4830	1.1071	0.094*
H19B	0.4212	0.4599	1.1828	0.094*
H19C	0.3949	0.4336	1.0649	0.094*
C20	0.1187(2)	0.17365(15)	1.1410(2)	0.0514(6)
C21	0.1387(3)	0.16141(19)	1.2597(2)	0.0716(8)
H21A	0.2054	0.1200	1.2783	0.107*
H21B	0.0583	0.1428	1.2820	0.107*
H21C	0.1648	0.2137	1.2932	0.107*
C22	0.0199(3)	0.24498(19)	1.1092(3)	0.0796(9)
H22A	0.0080	0.2511	1.0345	0.119*
H22B	0.0526	0.2967	1.1410	0.119*
H22C	-0.0624	0.2315	1.1322	0.119*
C23	0.0592(2)	0.09225(17)	1.0920(2)	0.0616(7)
H23A	0.1191	0.0466	1.1091	0.092*
H23B	0.0424	0.0986	1.0175	0.092*
H23C	-0.0214	0.0806	1.1188	0.092*

Source of material

The title compound was prepared by dropwise addition of bromine (1.25 mmol) to 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)-1-phenyl-propenone (1.19 mmol) in methylene chloride (10 mL) and the reaction mixture was stirred for three hours at room temperature. The reaction mixture was evaporated. The raw product was recrystallized in ethanol to get crystals. The yield of the title compound is 70% m.p. 148 °C; ¹H NMR (300 MHz, DMSO-*d*₆) δ 1.41 (s, 18H, 6CH₃), 7.48–7.84 (m, 9H, 5CH_{arom} + 2CH_{arom} + CH = +OH_{arom}). ¹³C NMR (75 MHz, CDCl₃) δ 30.92 (6CH₃), 35.05 (2C_{quat}), 119.07 (C_{arom}), 124.80 (C_{arom}), 128.52 (CH_{arom}), 129.36 (CH_{arom}), 129.82 (CH_{arom}), 132.86 (CH_{arom}), 137.32 (C_{arom}), 138.90 (C_{arom}), 145.19 (CH=), 157.21 (=C_{quat}), 191.38 (C = O).

Experimental details

H atoms were located in the difference Fourier map, but refined with fixed individual displacement parameters, using a riding model with C–H distances of 0.93 Å (for aromatic rings), 0.96 Å (CH₃ group), with *U*(H) values of 1.2*U*_{eq}(C) (for CH in aromatic moiety), and 1.5*U*_{eq}(C) (for CH₃) and O–H distance 0.8601 Å, with *U*(H) values of 1.2*U*_{eq}(O).

Comment

α-Halogen chalcones are valuable synthetic building blocks in organic synthesis [4, 5]. The existence of bulky and electron-withdrawing groups in α-position lead to an enhancement of the electrophilicity. Examples of the influence of α-modification on biological activity was presented in literature. α-Halo chalcones are shown to possess biological activity [6]. Development of chalcones and related analogues as antimitotic agents was reported [7].

In the title compound the dihedral angle between the mean planes of the aromatic rings is 40.36(12)°. A search in the latest version of the Cambridge structural database, for related compounds yielded only one structure namely 3-(3,5-di-*t*-butyl-4-hydroxyphenyl)-1-(4-hydroxy-3-methoxyphenyl)prop-2-en-1-one (REFCODE PASSOU) [8]. The geometrical and molecular parameters are very similar between both compounds and the main difference is the crystal packing. In the title compound the molecules are linked by van der Waals interactions only, whereas in the related compound, the molecules are linked by hydrogen bond interactions. The conformation about the C=C bond is *Z*, with a C7–C8–C9–C10 torsion angle of 176.26(3)°.

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