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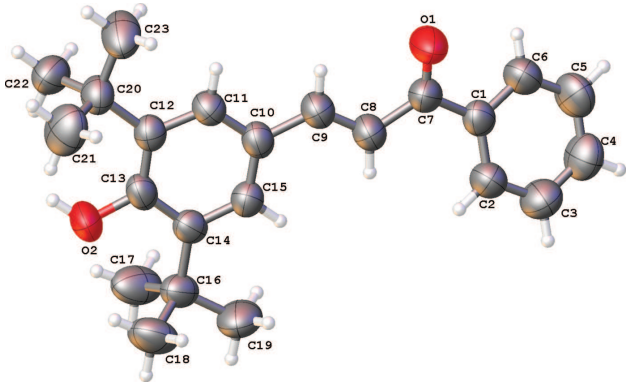
Crystal structure of (*E*)-3-(3,5-di-tert-butyl-4-hydroxyphenyl)-1-phenylprop-2-en-1-one, $C_{23}H_{28}O_2$



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Abstract

$C_{23}H_{28}O_2$, monoclinic, $P2_1/n$ (no. 14), $a = 8.9114(3)$ Å, $b = 17.7151(6)$ Å, $c = 12.6087(5)$ Å, $\beta = 100.0520(10)$, $V = 1959.93(12)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.061$, $wR_{ref}(F^2) = 0.171$, $T = 296(2)$ K.

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Crystal data, data collection and structure refinement details are summarized in Table 1.

Source of material

The title compound was prepared by the refluxing of 3,5-di-*tert*-butyl-benzaldehyde (4.3 mmol) with acetophenone (4.3 mmol) in methanol (10 mL) in presence catalytic amount of sulfuric acid during 3 h. Then the reaction mixture was cooled down. After one day the precipitated yellow single crystals were collected, washed with distilled water and dried in desiccator. The yield of 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)-1-phenyl-propenone is 67%. The analysis of the title compound was described in literature [4].

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Table 1: Data collection and handling.

Crystal:	Prismatic, red
Size:	0.2 × 0.2 × 0.2 mm
Wavelength:	Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å)
μ:	0.071 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, Φ and ω-*scans*
$2\theta_{max}$, completeness:	27.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	19602, 4266, 0.0193
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3308
$N(param)_{refined}$:	229
Programs:	Bruker programs [1], ShelX [2], OLEX2 [3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.1656(2)	0.06016(10)	0.22861(18)	0.0780(6)
O2	0.2734(2)	0.29775(9)	−0.20358(14)	0.0684(5)
H2	0.2502	0.3454	−0.2181	0.103
C1	0.0180(2)	−0.03502(11)	0.26970(16)	0.0427(4)
C2	0.1167(3)	−0.08460(13)	0.23132(18)	0.0527(5)
H2	0.1498	−0.0743	0.1668	0.063
C3	0.1659(3)	−0.14910(14)	0.2884(2)	0.0630(6)
H3	0.2303	−0.1826	0.2615	0.076
C4	0.1205(3)	−0.16394(14)	0.3843(2)	0.0620(6)
H4	0.1543	−0.2074	0.4225	0.074
C5	0.0255(3)	−0.11506(15)	0.42444(19)	0.0641(7)
H5	−0.0043	−0.1250	0.4902	0.077
C6	−0.0258(3)	−0.05131(13)	0.36761(18)	0.0553(6)
H6	−0.0910	−0.0185	0.3951	0.066
C7	−0.0478(3)	0.03280(11)	0.20867(18)	0.0499(5)
C8	0.0309(3)	0.06601(12)	0.12732(18)	0.0492(5)
H8	0.1129	0.0405	0.1074	0.059
C9	−0.0124(3)	0.13181(12)	0.08141(18)	0.0485(5)
H9	−0.0990	0.1535	0.1005	0.058
C10	0.0597(2)	0.17406(11)	0.00475(16)	0.0424(4)
C11	−0.0012(2)	0.24332(11)	−0.03236(16)	0.0427(4)
H11	−0.0876	0.2607	−0.0080	0.051
C12	0.0617(2)	0.28766(11)	−0.10444(16)	0.0406(4)
C13	0.1930(2)	0.25954(11)	−0.13756(16)	0.0425(4)
C14	0.2586(2)	0.18879(11)	−0.10338(16)	0.0407(4)
C15	0.1880(2)	0.14789(11)	−0.03310(16)	0.0418(4)
H15	0.2277	0.1010	−0.0100	0.050
C16	0.4052(2)	0.16003(12)	−0.13895(18)	0.0489(5)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C17	0.5378(3)	0.21243(16)	−0.0951(3)	0.0704(8)
H17A	0.6304	0.1924	−0.1128	0.106
H17B	0.5472	0.2162	−0.0183	0.106
H17C	0.5190	0.2616	−0.1267	0.106
C18	0.3852(3)	0.15416(17)	−0.2624(2)	0.0709(7)
H18A	0.4787	0.1370	−0.2823	0.106
H18B	0.3593	0.2028	−0.2938	0.106
H18C	0.3053	0.1189	−0.2881	0.106
C19	0.4494(3)	0.08123(15)	−0.0932(3)	0.0729(8)
H19A	0.5409	0.0649	−0.1168	0.109
H19B	0.3685	0.0463	−0.1183	0.109
H19C	0.4663	0.0832	−0.0160	0.109
C20	−0.0105(2)	0.36464(12)	−0.14150(18)	0.0478(5)
C21	−0.0531(3)	0.36999(16)	−0.2669(2)	0.0749(8)
H21A	−0.0933	0.4193	−0.2868	0.112
H21B	−0.1285	0.3325	−0.2928	0.112
H21C	0.0363	0.3615	−0.2982	0.112
C22	0.0945(3)	0.42986(12)	−0.0929(2)	0.0599(6)
H22A	0.0494	0.4772	−0.1180	0.090
H22B	0.1920	0.4249	−0.1146	0.090
H22C	0.1073	0.4280	−0.0157	0.090
C23	−0.1616(3)	0.37747(16)	−0.1010(3)	0.0730(8)
H23A	−0.2029	0.4257	−0.1255	0.109
H23B	−0.1437	0.3763	−0.0237	0.109
H23C	−0.2326	0.3385	−0.1285	0.109

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 (CH_3 group), with U(H) values of 1.2U_{eq}(C) (for CH in aromatic moiety), and 1.5U_{eq}(C) (for CH_3) and O—H distance 0.88 Å, with U(H) values of 1.2U_{eq}(O).

Discussion

Being of 1,3-diphenyl-2-propen-1-one derivatives, chalcones are simple scaffold found in many plant sources. Different synthesis methods and biological activities of chalcones were summarized in recent review article [5]. Also 3,5-di-*tert*-butyl-4-hydroxy styrene derivatives demonstrate antiinflammatory activity [6], gastroprotective effect [7], and antifungal activity [8].

The conformation about the C=C bond is *E*, with a C7—C8—C9—C10 torsion angle of 175.88(5)°. In the title compound the dihedral angle between the mean planes of the aromatic rings is 21.77(10)°. In the crystal, the molecules are linked by strong O—H···O hydrogen bonds into chain with graph-set notation C(10) along the [101] direction [9]. These supramolecular properties and the geometric and molecular parameters are very similar to 3-(3,5-di-t-butyl-4-hydroxyphenyl)-1-(4-hydroxy-3-methoxyphenyl)prop-2-en-1-one, an close related compound [10].

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