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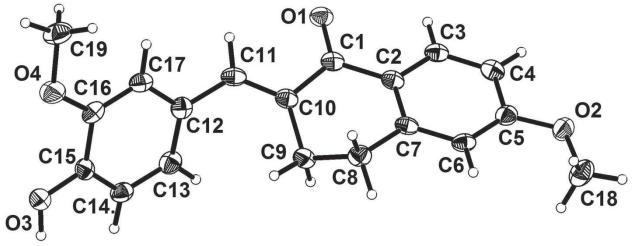
Crystal structure of (*E*)-2-(4-hydroxy-3-methoxybenzylidene)-6-methoxy-3,4-dihydronaphthalen-1(2*H*)-one, $C_{19}H_{18}O_4$



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Abstract

$C_{19}H_{18}O_4$, orthorhombic, *Pbcn*, $a = 21.1472(8)$ Å, $b = 9.7978(4)$ Å, $c = 14.5181(6)$ Å, $V = 3008.1(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0520$, $wR_{ref}(F^2) = 0.1194$, $T = 100(2)$.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Orange plate
Size:	0.56 × 0.17 × 0.09 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	55°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	39151, 3461, 0.134
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2466
$N(param)_{refined}$:	214
Programs:	SHELX [22], Bruker programs [23]

Source of material

To a stirred solution of 6-methoxy-1-tetralone (5 g, 0.0028 mol) in conc. HCl (28 mL) and glacial acetic acid (28 mL) at 0 °C, vanillin (4.3 g, 0.0028 mol) was added. The resulting mixture was further stirred at this temperature for 3 h, then at room temperature for 18 h. Diethyl ether was added and the ethereal layer was discarded. The remaining residue was treated with a water/ice mixture. The separated solid was filtered off and recrystallized from ethanol/ petroleum ether to afford the title compound (50%) as red crystals, m.p. 110–115 °C; γ_{max} **IR** (KBr)/cm^{-1} 3431, 3177, 2940, 2838, 1638, 1597, 1559, 1520, 1443, 1425, 1390, 1317, 1253, 1165; **^{1}H-NMR** (400 MHz; $CDCl_3$) δH: 2.92 (2 H, t, *J* 6.0, CH_2), 3.13 (2 H, app. td, *J* 6.0, 1.7, CH_2), 3.87 (3H, s, OCH_3), 3.92 (3H, s, OCH_3), 6.70 (1H, d, *J* 2.6, CH—Ph-tetralone), 6.87 (1H, dd, *J* 8.5, 2.6, CH—Ph-tetralone), 6.96–6.95 (2H, m, 2 × CH—Ph-vanillin), 7.02 (1H, dd, *J* 7.7, 1.7, CH—Ph-vanillin), 7.78 (1H, s, CH = C_qCO), 8.10 (1H, d, *J* 8.5, CH—Ph-tetralone); **^{13}C-NMR** (100 MHz; $CDCl_3$) δH: 27.28 (CH_2), 29.18 (CH_2), 55.40 (OCH_3), 55.94 (OCH_3), 112.21, 112.73, 113.21, 114.39, 123.64, 127.14, 128.32, 130.66, 133.70, 136.33, 145.51, 146.24, 163.46 (6 × CH—Ph-tetralone and CH—Ph, 6 × C_q-Ph-tetralone and C_q-Ph), 186.71 (C = O); δ_C (100 MHz; $CDCl_3$) 27.28 (CH_2), 29.18 (CH_2), 55.40 (OCH_3), 55.94 (OCH_3), 112.21, 112.73, 113.21, 114.39, 123.64, 127.14, 128.32, 130.66, 133.70, 136.33, 145.51, 146.24, 163.46 (14 × CH-Ar, Cq-Ar and CH = Cq CO), 186.71 (C = O).

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	0.45468(6)	0.82075(12)	−0.11321(9)	0.0172(3)
O2	0.72991(6)	1.06912(13)	−0.11627(9)	0.0207(3)
O3	0.36151(6)	0.37388(14)	0.36125(9)	0.0197(3)
O4	0.28832(6)	0.58164(13)	0.31800(9)	0.0232(3)
C1	0.49596(9)	0.81438(17)	−0.05242(12)	0.0142(4)
C2	0.55753(9)	0.88251(17)	−0.06542(12)	0.0147(4)
C3	0.57352(9)	0.93631(17)	−0.15192(12)	0.0155(4)
H3A	0.5446	0.9309	−0.2000	0.019*
C4	0.63112(9)	0.99665(19)	−0.16657(13)	0.0173(4)
H4A	0.6414	1.0304	−0.2245	0.021*
C5	0.67441(9)	1.00737(18)	−0.09393(13)	0.0167(4)
C6	0.65949(9)	0.95551(18)	−0.00765(13)	0.0155(4)
H6A	0.6882	0.9633	0.0405	0.019*
C7	0.60138(9)	0.89180(17)	0.00671(12)	0.0150(4)
C8	0.58494(9)	0.83213(19)	0.09932(13)	0.0179(4)
H8A	0.5619	0.8992	0.1352	0.021*
H8B	0.6235	0.8101	0.1323	0.021*
C9	0.54469(9)	0.70305(19)	0.08894(13)	0.0178(4)
H9A	0.5692	0.6325	0.0586	0.021*
H9B	0.5325	0.6697	0.1493	0.021*
C10	0.48615(9)	0.73407(17)	0.03296(13)	0.0155(4)
C11	0.42684(9)	0.69864(17)	0.05672(12)	0.0158(4)
H11A	0.3943	0.7351	0.0213	0.019*
C12	0.40819(9)	0.60866(18)	0.13255(12)	0.0158(4)
C13	0.44199(9)	0.49039(18)	0.15217(13)	0.0180(4)
H13A	0.4763	0.4663	0.1156	0.022*
C14.	0.42482(9)	0.40813(18)	0.22580(13)	0.0168(4)
H14A	0.4469	0.3276	0.2368	0.020*
C15	0.37567(9)	0.44399(18)	0.28277(13)	0.0154(4)
C16	0.33833(9)	0.55846(18)	0.26053(13)	0.0160(4)
C17	0.35452(9)	0.63824(18)	0.18579(13)	0.0164(4)
H17A	0.3294	0.7128	0.1705	0.020*
C18	0.77579(10)	1.0899(2)	−0.04455(14)	0.0247(5)
H18A	0.8132	1.1303	−0.0702	0.037*
H18B	0.7863	1.0037	−0.0171	0.037*
H18C	0.7585	1.1494	0.0015	0.037*
C19	0.24712(10)	0.6916(2)	0.29427(15)	0.0257(5)
H19A	0.2121	0.6937	0.3363	0.039*
H19B	0.2317	0.6789	0.2327	0.039*
H19C	0.2698	0.7763	0.2979	0.039*
H1O3	0.3905(12)	0.308(3)	0.3694(17)	0.049(8)*

approximation, with U_{iso}(H) set to 1.2U_{eq}(C) except for methyl hydrogen atoms. The H atoms of the methyl group were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density with U_{iso}(H) set to 1.5U_{eq}(C).

Discussion

2-Arylidene-1-tetralones contain the α,β-unsaturated enone fragment. This moiety is a characteristic functionality of biologically and synthetically important compounds known as chalcones. This synthon is considered as a versatile building block that can be used to construct heterocyclic rings having one or more heteroatoms and of different ring sizes such as 2-pyrazoline [1], pyrimidine [2, 3], pyran, and isooxazoline [4]. Generally chalcones and their analogs are prepared by Claisen-Schmidt reactions under basic conditions using bases such as sodium hydroxide or potassium hydroxide [5], lithium hydroxide [6] and piperidine [7]. Acid catalyzed synthesis is also well established using *p*-toluene sulfonic acid under focused microwave irradiation [8], thionyl chloride [9], dry hydrochloric acid and borontrifluoride-etherate [10]. Some chalcones are known for their biological activities as they exhibit cytotoxic [11], antibacterial [12], antifungal [13], antimalarial and antitubercular [14], anti-inflammatory [15], antimalarial [16], antiviral [17], tyrosinase inhibitor [18], antiallergenic [19], antioxidant [20] and antihyperglycemic [21] activities.

The asymmetric unit cell of the titled compound contains one independent molecule. The bond length of C10—C11 is 1.346(3) Å, which are typical C = C double bond and the configuration around this double bond is *trans*. The molecules are connected in the crystal *via* intermolecular hydrogen bonds: O3—H1O3$\cdots$O1^{i}, symmetry code: (i) $x, -y+1, z+1/2$ forming chains along the *c* axis of the choosen unit cell. Additionally, non-classical hydrogen bonds may contribute to the stability of the packing.

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