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Key indicators

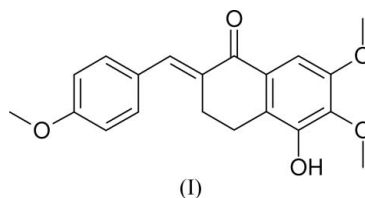
Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.045
 wR factor = 0.114
Data-to-parameter ratio = 16.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.5-Hydroxy-6,7-dimethoxy-2-[(*E*)-1-(4-methoxyphenyl)-
methylidene]-3,4-dihydro-2*H*-naphthalen-1-oneThe title compound, $\text{C}_{20}\text{H}_{20}\text{O}_5$, possesses normal geometrical
parameters. $\text{O}-\text{H}\cdots\text{O}$ bonds and possible $\text{C}-\text{H}\cdots\text{O}$
interactions are present.

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Comment

The title compound, commonly called 2(4-methoxybenzyl-
idene)-5-hydroxy-6,7-dimethoxytetral-1-one, (I), prepared by
the condensation of anisaldehyde with 5-hydroxy-6,7-
dimethoxytetral-1-one, was found to be sensitive to aerial
oxidation. Its crystal structure was determined in the hope of
identifying structural features which might explain its ready
oxidation.

All the geometrical parameters for (I) (Fig. 1 and Table 1) lie within their expected ranges (Allen *et al.*, 1995). The six-membered C9–C14 ring adopts an envelope configuration, with C9 and C11–C14 approximately coplanar [r.m.s. deviation = 0.054 Å, maximum deviation = 0.0846 (10) for atom C14] and C10 in the flap position, displaced by 0.637 (2) Å from the mean plane. The dihedral angle between the C3 and C15 benzene rings is 66.90 (4)°. The terminal methyl groups are displaced from their attached benzene ring C-atom mean planes by 0.066 (3), 0.079 (3), and –1.113 (3) Å for atoms C1, C19, and C20, respectively.

Various $\text{O}-\text{H}\cdots\text{O}$ and possible $\text{C}-\text{H}\cdots\text{O}$ interactions exist in the crystal structure of (I) (Table 2). The O3–H1 group forms a bifurcated intramolecular/intermolecular hydrogen bond. The intermolecular $\text{O}-\text{H}\cdots\text{O}$ connectivity results in chains of (I) propagating along [010]. Pairs of inversion-symmetry-related C3-benzene rings interact by $\pi-\pi^i$ [symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$] stacking with a centroid separation of 3.6516 (9) Å and an interplane separation of 3.451 Å. Overall, we cannot observe any unusual structural features in (I) that correlate with its sensitivity to oxidation.

Experimental

A stirred solution of 102 mg of 5-hydroxy-6,7-dimethoxytetral-1-one (Cooke & Robinson, 1970) and 100 mg of 4-methoxybenzaldehyde in ethanol (10 ml) was treated dropwise with concentrated sulfuric acid (1.2 ml) over a period of 10 min (slight exothermic reaction) and the pale-brown solution was left at room temperature for 5 d. The crys-

tals that had formed were collected and washed with cold ethanol. Recrystallization from ethanol gave (I) as pale-yellow needles (124 mg, 80%; m.p. 412–413 K) UV-vis: λ_{\max} 334 nm ($\epsilon = 16,100$); ν_{\max} (cm^{-1}) 3312, 1654, 1600, 1578, 1252, 1167, 1120, 1009, 928, 824; ^{13}C NMR (100 MHz): δ 21.27, 26.78, 55.33, 55.95, 61.06, 102.81, 113.92, 123.61, 128.51, 129.17, 131.66, 133.79, 136.33, 139.41, 145.70, 150.80, 159.85, and 187.28. The crystals were sensitive to oxidation, turning red on exposure to air. TLC investigations revealed a complex mixture of oxidation products. The compound was also oxidized by high-potential quinones in methanol or dioxane solution, again giving an intractable mixture of products.

Crystal data

$\text{C}_{20}\text{H}_{20}\text{O}_5$	$D_x = 1.347 \text{ Mg m}^{-3}$
$M_r = 340.36$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3939 reflections
$a = 18.3264 (5) \text{ \AA}$	$\theta = 2.9\text{--}27.5^\circ$
$b = 13.3022 (5) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 15.7673 (6) \text{ \AA}$	$T = 120 (2) \text{ K}$
$\beta = 119.1321 (16)^\circ$	Block (cut from needle), pale yellow
$V = 3357.5 (2) \text{ \AA}^3$	$0.22 \times 0.20 \times 0.12 \text{ mm}$
$Z = 8$	

Data collection

Nonius KappaCCD diffractometer	3231 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.042$
Absorption correction: multi-scan (SADABS; Bruker, 2003)	$\theta_{\text{max}} = 27.5^\circ$
$T_{\text{min}} = 0.979$, $T_{\text{max}} = 0.989$	$h = -23 \rightarrow 23$
20240 measured reflections	$k = -17 \rightarrow 17$
3856 independent reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 3.3509P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.114$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
3856 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
233 parameters	Extinction correction: SHELXL97
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0030 (5)

Table 1

Selected geometric parameters (\AA , $^\circ$).

C5–C8	1.460 (2)	C13–C14	1.4814 (19)
C9–C14	1.484 (2)		
C9–C10–C11–C12	52.21 (16)	C12–C13–C14–C9	14.32 (19)
C10–C11–C12–C13	−27.58 (18)	C10–C9–C14–C13	13.43 (18)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O3–H1 \cdots O2 ⁱ	0.86 (2)	2.02 (2)	2.8248 (15)	155 (2)
O3–H1 \cdots O5	0.86 (2)	2.29 (2)	2.7308 (15)	112 (2)
C6–H6 \cdots O5 ⁱⁱ	0.95	2.48	3.2668 (18)	140
C7–H7 \cdots O3 ⁱⁱ	0.95	2.59	3.4591 (18)	153

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

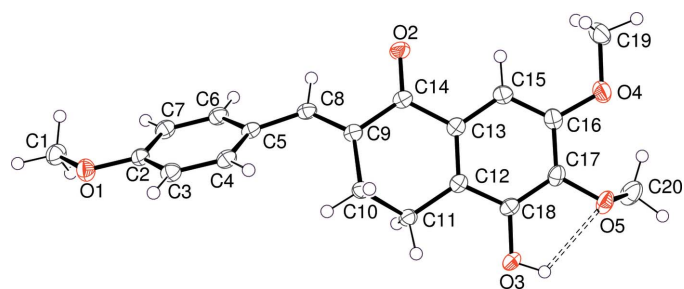


Figure 1

View of (I) (50% displacement ellipsoids; H atoms are drawn as small spheres of arbitrary radius). The dashed lines represent the O–H \cdots O intramolecular interaction.

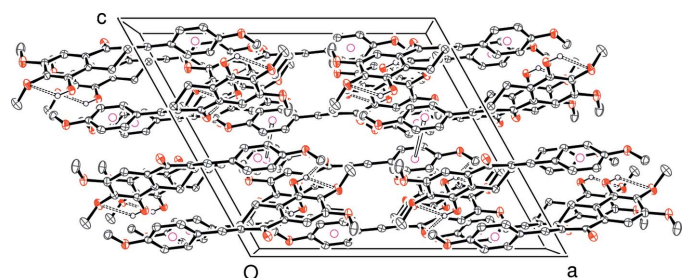


Figure 2

View showing crystal packing (all H atoms omitted except OH; pink circles used to represent the centroid of the C3 benzene ring).

The O-bound H atom was located in a difference map. Its position was freely refined with the constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$ applied. All the C-bound H atoms were placed in calculated positions ($\text{C--H} = 0.95\text{--}0.99 \text{ \AA}$) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$. The methyl groups were allowed to rotate to best fit the electron density.

Data collection: COLLECT (Nonius, 1998); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: SCALEPACK and DENZO (Otwinowski & Minor, 1997), and SORTAV (Blessing, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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