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Crystal structure of (2*E*)-1-(4-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one

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ABSTRACT



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The compound, C₁₆H₁₄O₃, (except H atoms) is almost planar [r.m.s. deviations for all non-H atoms = 0.001 Å] and the dihedral angle between the aromatic rings is 9.35 (7)°. In the crystal, molecules are linked by intermolecular O—H...O and C—H...O hydrogen bonds, forming a three-dimensional network structure. Furthermore, a weak π-π stacking interactions [centroid-to-centroid distance = 3.7055 (9) Å] contributes to the stabilization of the molecular packing. Crystal Data for C₁₆H₁₄O₃ (*M* = 254.27 g/mol): Orthorhombic, space group *Pbca* (no. 61), *a* = 13.4563(16) Å, *b* = 11.4986(14) Å, *c* = 16.720(2) Å, *V* = 2587.1(5) Å³, *Z* = 8, *T* = 296(2) K, μ(MoKα) = 0.090 mm⁻¹, *D*_{calc} = 1.306 g/cm³, 61402 reflections measured (4.88° ≤ 2θ ≤ 56.76°), 3240 unique (*R*_{int} = 0.0334, *R*_{sigma} = 0.0120) which were used in all calculations. The final *R*₁ was 0.0438 [*I* ≥ 2σ(*I*)] and *wR*₂ was 0.1228 (all data).

KEYWORDS

Chalcones
 π-π Stacking
 Aromatic ring
 Methoxyphenyl
 Crystal structure
 Geometric parameter

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1. Introduction

Chalcones, 1,3-diaryl-2-propene-1-ones, are major component of many natural products as well as important precursors for many synthetic manipulations [1-3]. Chalcones as well as their synthetic analogues display enormous number of biological activities such as anticancer, antimalarial, antibacterial, anti-inflammatory, antifungal, antioxidant, anti-HIV, antiprotozoal and carbonic anhydrase inhibiting activities [1-8].

The presence of double bond in conjugation with carbonyl functionality is believed to be responsible for the biological activities of chalcones, as removal of this functionality make them inactive [1,3,5,7]. They have the tendency to exist both in *cis*- and *trans*-forms, and can easily be cyclized to form flavanones via Michael addition. A number of synthetic routes have been reported for synthesis of chalcones while their general synthesis involves Claisen-Schmidt condensation

under homogeneous conditions in the presence of acid or base [1-8].

In this paper, we are reporting the synthesis and structural characterization of (2*E*)-1-(4-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one (**I**) (Scheme 1).

2. Experimental

2.1. Synthesis and crystallization

An aqueous solution of sodium hydroxide (10% w:v, 10 mL) was added into the mixture of 4-methoxybenzaldehyde (0.02 mol) and 4-hydroxyacetophenone (0.02 mol) in ethanol (6 mL). After stirring it at room temperature overnight, it was poured into the water in a beaker (100 mL). This mixture was neutralised with HCl (10%) and the precipitate formed was filtered, washed with cold water and dried.

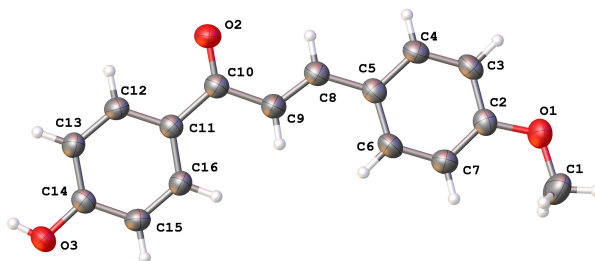
Table 1. Crystal data and structure refinement for the title compound.

CCDC no	1842374
Empirical formula	C ₁₆ H ₁₄ O ₃
Formula weight	254.27
Temperature (K)	296(2)
Crystal system	Orthorhombic
Space group	<i>Pbca</i>
a (Å)	13.4563(16)
b (Å)	11.4986(14)
c (Å)	16.720(2)
Volume (Å ³)	2587.1(5)
Z	8
ρ _{calc} (g/cm ³)	1.306
μ (mm ⁻¹)	0.090
F(000)	1072.0
Crystal size (mm ³)	0.668 × 0.380 × 0.158
Radiation	MoKα (λ = 0.71073)
2θ range for data collection (°)	4.88 to 56.76
Index ranges	-17 ≤ h ≤ 17, -15 ≤ k ≤ 15, -22 ≤ l ≤ 22
Reflections collected	61402
Independent reflections	3240 [R _{int} = 0.0334, R _{sigma} = 0.0120]
Data/restraints/parameters	3240/0/174
Goodness-of-fit on F ²	1.041
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0438, wR ₂ = 0.1104
Final R indexes [all data]	R ₁ = 0.0590, wR ₂ = 0.1228
Largest diff. peak/hole (e.Å ⁻³)	0.23/-0.16

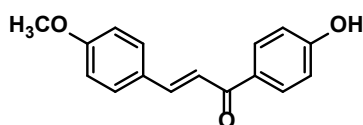
Table 2. Hydrogen-bond parameters (Å, °) for compound I.

D—H...A	D—H	H...A	D...A	D—H...A
O3—H3...O2 ⁱ	0.82	1.88	2.6926 (17)	169
C8—H8...O1 ⁱⁱ	0.93	2.54	3.4383 (18)	161

Symmetry codes: (i) $x+1/2, -y-1/2, -z+1$; (ii) $-x+1, y-1/2, -z+3/2$.

**Figure 1.** View of the molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

The solid was crystallized from ethanol-water. M.p.: 182-184 °C, Yield: 86% [2-5,7,9,10].



Scheme 1

2.2. X-ray crystallography

Data collection and cell refinement for compound I were carried out using a diffractometer Bruker APEX-II CCD [11] with graphite monochromated MoKα radiation at 296 K and Bruker SAINT [11], respectively. The absorption correction was applied using multi-scan SADABS [12]. The structure was solved by using SHELXS-2014 [13] and refined by using SHELXL-2014 [13]. The structure was drawn with ORTEP-3 for Windows [14] and Software used to prepare material for publication with PLATON [15]. The crystal data, conditions of data collection and refinement are reported in Table 1.

2.3. Refinement

All H-atoms were positioned geometrically (C—H = 0.93 Å for aromatic H, C—H = 0.96 Å for methyl H, O—H = 0.82 Å), and were included in the refinement in the riding model approximation, with $U_{iso}(H) = 1.2 U_{eq}(C)$ for aromatic H atoms, and $U_{iso}(H) = 1.5 U_{eq}(C, O)$ for methyl and hydroxyl H atoms.

3. Result and discussion

3.1. Description of crystal structure of compound I

As shown in Figure 1, whole molecule (except H atoms) of the title compound is almost planar [r.m.s. deviations for all non-H atoms = 0.001 Å]. The aromatic rings (C2–C7 and C11–C16) makes a dihedral angle of 9.35 (7)° with each other. The values of the geometric parameters are normal and comparable with those previously reported for the related structures in literature [16-18].

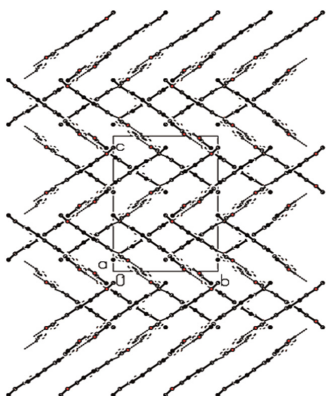
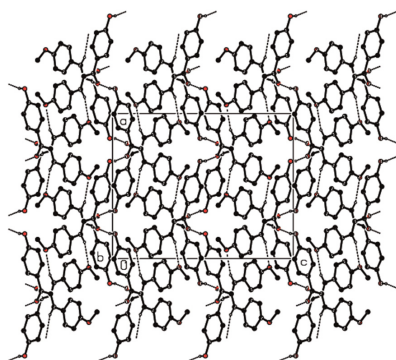
Intermolecular O—H...O and C—H...O hydrogen bonds link the adjacent molecules, forming a three-dimensional network structure (Table 2, Figures 2-4). In addition, π-π stacking interactions [$Cg2 \cdots Cg2$ ($2-x, -y, 1-z$) = 3.7055 (9) Å; where $Cg2$ is a centroid of the C11–C16 benzene ring] also contribute to the stabilization of the crystal structure.

Table 3. Atomic coordinates and equivalent isotropic thermal displacement parameters for non-hydrogen atoms (\AA^2 ; $U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* (a_i a_j)$).

Atom	x	y	z	U_{eq}
O1	0.56044 (8)	0.43745 (10)	0.86953 (7)	0.0606 (4)
O2	0.70623 (7)	-0.13924 (10)	0.58111 (7)	0.0577 (4)
O3	1.17050 (7)	-0.17719 (10)	0.51465 (7)	0.0577 (4)
C1	0.63246 (14)	0.50062 (15)	0.91428 (13)	0.0709 (6)
C2	0.59188 (10)	0.34586 (12)	0.82428 (8)	0.0430 (4)
C3	0.51834 (10)	0.28209 (13)	0.78629 (9)	0.0499 (4)
C4	0.54337 (10)	0.18849 (12)	0.73899 (9)	0.0457 (4)
C5	0.64222 (10)	0.15542 (11)	0.72790 (8)	0.0404 (4)
C6	0.71418 (10)	0.22069 (13)	0.76745 (9)	0.0507 (5)
C7	0.69045 (10)	0.31462 (13)	0.81497 (9)	0.0502 (5)
C8	0.66677 (10)	0.05684 (12)	0.67640 (8)	0.0433 (4)
C9	0.75684 (11)	0.02032 (14)	0.65808 (9)	0.0511 (4)
C10	0.77635 (10)	-0.08013 (12)	0.60571 (8)	0.0431 (4)
C11	0.88015 (9)	-0.10679 (11)	0.58359 (8)	0.0387 (3)
C12	0.90093 (10)	-0.19947 (12)	0.53215 (9)	0.0450 (4)
C13	0.99669 (10)	-0.22530 (12)	0.50884 (9)	0.0439 (4)
C14	1.07523 (9)	-0.15835 (11)	0.53666 (8)	0.0410 (4)
C15	1.05667 (10)	-0.06654 (12)	0.58833 (9)	0.0469 (4)
C16	0.96075 (10)	-0.04144 (12)	0.61111 (8)	0.0441 (4)

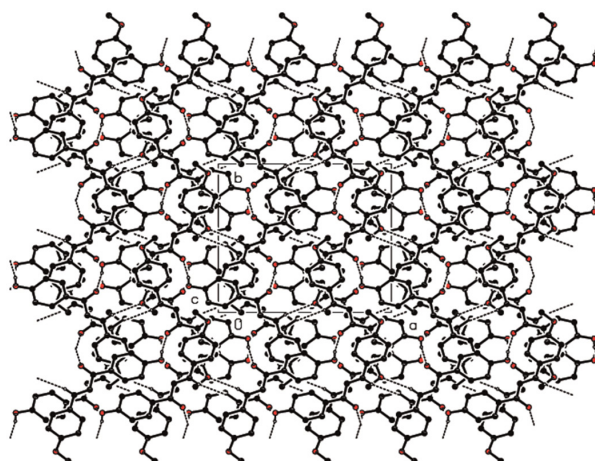
Table 4. Selected geometric parameters (\AA , $^\circ$) for compound I.

O1—C1	1.424 (2)
O2—C10	1.234 (2)
C6—C7	1.378 (2)
C14—C15	1.387 (2)
C1—O1—C2	118.32 (12)
C5—C6—C7	122.35 (13)
O3—C14—C13	122.84 (12)
C1—O1—C2—C3	175.62 (14)
O2—C10—C11—C16	-179.63 (13)
C16—C11—C12—C13	-0.5 (2)

**Figure 2.** The molecular packing and hydrogen bonding viewing down *a*-axis. H atoms not involved in hydrogen bonding have been omitted for clarity.**Figure 3.** The molecular packing and hydrogen bonding viewing down *b*-axis. H atoms not involved in hydrogen bonding have been omitted for clarity.

The atomic coordinates of the non-hydrogen atoms with their equivalent temperature factors are presented in Table 3.

The experimental geometric parameters of the title molecule are listed in Table 4. The values of the geometric parameters are normal and comparable with those previously reported for the related structures in literature [16-18].

**Figure 4.** The molecular packing and hydrogen bonding viewing down *c*-axis. H atoms not involved in hydrogen bonding have been omitted for clarity.

4. Conclusions

In this study, we have determined the crystal structure of (2E)-1-(4-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one using single crystal X-ray diffraction analysis. In the crystal, molecules are linked by intermolecular O—H...O and C—H...O hydrogen bonds, forming a three-dimensional

network structure. Furthermore, a weak π - π stacking interactions [centroid-to-centroid distance = 3.7055(9) Å] contributes to the stabilization of the molecular packing.

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Supplementary information

CCDC-1842374 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>, or by e-mailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.

Author contributions: All authors contributed equally to this work.

Ethical approval: All ethical guidelines have been adhered.

Sample availability: Samples of the compounds are available from the author.

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