



Synthesis, crystal structure, experimental and theoretical investigations of 3-(4-ethoxy-3-methoxyphenyl)-1-phenylprop-2-en-1-one



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ABSTRACT

A chalcone derivative namely, (*E*)-3-(4-ethoxy-3-methoxyphenyl)-1-phenylprop-2-en-1-one (**1v**) has been synthesized and characterized on the basis of its spectral data. The solid state self-assembly studies of **1v** were carried out through single crystal X-ray technique to see the major non-covalent interactions responsible for molecular alignment in the solid state. Furthermore, the optimized molecular geometry, vibrational frequencies, ¹H and ¹³C NMR chemical shift (in gas and in chloroform solvent) values and the molecular electrostatic potential (MEP) surface parameters of **1v** were calculated using DFT/B3LYP/HF/M06 method with 6–311++G (d,p) basis set in ground state. All the theoretical calculations for **1v** were found in good agreement with experimental data.

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1. Introduction

Chalcones (benzylideneacetophenones) belong to one of the largest classes of plant metabolites and are precursors of flavonoids and isoflavonoids that play major role in the plant defense mechanism to protect from microorganisms, insects and animals [1–8]. Synthetically, chalcones can easily be obtained by the Claisen-Schmidt condensation reaction between substituted acetophenones and benzaldehydes. The possibility of having different substituents on the two aromatic rings and their ability to act as Michael acceptors make this class of compounds highly attractive drug scaffolds. Hence, a large variety of their derivatives have been

synthesized in recent years with interesting and diverse pharmacological activities such as anti-fungal, anti-bacterial, anti-cancer, anti-inflammatory, *anti*-plasmodial, immunosuppressive, anti-oxidant, anti-leishmanial, analgesic and anti-pyretic activities etc [9–16]. Other than their applications in medicinal chemistry, they have also been reported to show nonlinear optical and luminescent properties [17–20].

With the advent of various computational methods in recent years, the theoretical investigations for solving structure and chemical reactivity related problems are getting increased attention from computational chemistry community [21–29]. These methods are presently emerging as an efficient tool for predicting molecular structures, harmonic force fields, vibrational wavenumbers, IR intensities and Raman activities etc. of biologically important molecules, and may serve as an alternative tool to various spectral techniques, that are needed for structure elucidation, in near future. Therefore, a lot of energy and money may be saved by using these methods.

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Table 1
Crystallographic data for chalcone derivative **1v**.

Crystal data	1v
CCDC	983900
Chemical formula	C ₁₈ H ₁₈ NO ₃
M _r	296.33
Crystal system, space group	Monoclinic, P2 ₁ /c
Temperature (K)	139
a, b, c (Å)	12.4849 (2), 14.0781 (2), 8.7868 (1)
β (°)	105.787 (1)
V (Å ³)	1486.14 (4)
Z	4
Radiation type	Mo Kα
μ (mm ⁻¹)	0.09
Crystal size (mm)	0.30 × 0.16 × 0.10
Data collection	
Diffractometer	Bruker APEX-II CCD area-detector diffractometer
Absorption correction	Multi-scan (SADABS; Sheldrick, 1996)
T _{min} , T _{max}	0.670, 0.746
No. of measured, independent and observed [I > 2σ(I)] reflections	14019, 3415, 2783
R _{int}	0.023
(sin θ/λ) _{max} (Å ⁻¹)	0.650
Refinement	
R [I ² > 2σ(I ²)], wR (F ²), S	0.037, 0.102, 1.02
No. of reflections	3415
No. of parameters	192
H-atom treatment	H-atom parameters constrained
Δ _{max} , Δ _{min} (e Å ⁻³)	0.25, -0.18

In this context and as continuation of our recent research interests in organic synthesis, self-assembly and theoretical spectral investigations of small organic molecules [30–44], herein, we report the synthesis, solid state self-assembly, optimized molecular geometry, vibrational IR analysis, Nuclear Magnetic Resonance (NMR) analysis and molecular electrostatic potential (MEP) calculated at the B3LYP/6–311++G (d,p) level of (*E*)-3-(4-ethoxy-3-methoxyphenyl)-1-phenylprop-2-en-1-one (**1v**), focusing on the comparison of calculated and the experimental results.

2. Experimental

2.1. General

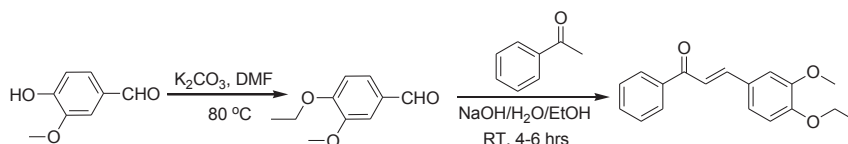
All reagents and solvents were used as purchased from the supplier or recrystallized/redistilled as required. Thin layer chromatography (TLC) was performed using aluminium sheets coated with silica gel 60 F254 (Merck). The melting point of chalcone derivative (**1v**) was determined in open capillary tubes by using Gallenkamp apparatus (MP-D) and was uncorrected. The FT-IR was recorded on NICOLET 6700 FT-IR Spectrometer (4000–400 cm⁻¹) using ATR (attenuated total reflectance) method. The ¹H and ¹³C Nuclear Magnetic Resonance (NMR) spectra of **1v** were determined on Bruker spectrometer at 300 MHz and 75 MHz in CDCl₃, respectively, using residual solvent signals as a reference.

2.2. Synthesis of a chalcone derivative (**1v**) [45]

In a 100 mL two neck-round bottom flask equipped with reflux condenser containing DMF (10 mL) was added 4-hydroxy-3-

methoxybenzaldehyde (2 g, 0.01 mol) and anhydrous K₂CO₃ (2.07 g, 0.015 mol) and heated the reaction mixture at 60–65 °C for 30 min before the dropwise addition of bromoethane (0.01 mol). After addition of bromoethane, the whole reaction mixture was heated under N₂ at 80 °C for 4–5 h. After the completion of the reaction, monitored by TLC, the reaction mixture was poured into separating funnel containing ethylacetate and washed several times with water. The organic layer was then dried over anhydrous MgSO₄, evaporated by using rotary evaporator to get 4-ethoxy-3-methoxybenzaldehyde intermediate, which was used in the next step without further purification. The 4-Alkoxy-3-methoxybenzaldehyde intermediate (0.01 mol, 1 g) was dissolved in ethanol (10 mL) in a 100 mL round bottom flask and 37% ice cooled NaOH solution (10 mL) was then added into it. After stirring the reaction mixture for 30 min at 0 °C, the acetophenone (0.01 mol) was added dropwise and stirred for another 4–6 h. The precipitates thus appeared were poured into the crushed ice, filtered and washed thoroughly with water. The obtained solid was finally recrystallized from ethanol to get pure **1v**.

1v: Yield 94%; yellow crystals; m. p. 105–108 °C; R_f = 0.47 (*n*-hexane: ethyl acetate, 4:1), IR (ν̄, cm⁻¹): 1652 (C=O), 1574 (C=C), 1237, 1035 (Ar-O-R); ¹H NMR (300 MHz, CDCl₃), 1.51 (3H, t, J = 6.9 Hz), 3.99 (3H, s), 4.17 (2H, q, J = 6.9 Hz), 6.91 (1H, d, J = 8.4 Hz), 7.18–7.28 (2H, m), 7.40 (1H, d, J = 15.6 Hz), 7.49–7.54 (3H, m), 7.78 (1H, d, J = 15.6 Hz), 8.03 (2H, d, J = 6.9 Hz); ¹³C NMR (75 MHz, CDCl₃) δ 14.07, 56.03, 64.03, 110.32, 112.16, 119.95, 123.21, 127.67, 128.45, 128.58, 132.58, 138.53, 145.17, 149.41, 150.86, 190.72; EIMS (*m/z*), 282.



Scheme 1. Synthesis of new chalcone derivative **1v**.

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