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Research Article

SYNTHESIS, SPECTRAL STUDY AND PROPERTIES OF (E)-3-(5-BROMOFURAN-2-YL)-1-(2,6-DIHYDROXYPHENYL)PROP-2-EN-1-ONE

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ABSTRACT

***(E)**-3-(5-bromofuran-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one was synthesized by Claisen Schmidt condensation method in alkaline medium. The Chalcone of 5-Bromo furfural was characterized by IR, UV-Visible, ^{1}H NMR, Mass spectra, CHO analysis and chemical tests. This is stable solid compound having yellowish brown color with molecular formula [$C_{13}H_9O_4Br$].*

***KEYWORDS:** Chalcone, acetophenone, UV-visible, IR, ^{1}H NMR, Mass spectral analysis, Claisen-Schmdit condensation method, CHO analysis.*

INTRODUCTION

The chemistry of chalcones has generated intensive scientific studies through the world. The name "chalcones" was given by Kostanecki and Tambor [1].The growing interest in chalcones and their potential use in medicinal applications are proved by the growing number of publications concerning the synthesis and biological evaluation of chalcones [2-4]. The focus is more on Chalcones synthesized by Claisen Schmidt condensation which involves the condensation between an aromatic aldehyde or ketone with an aromatic ketone or aldehyde catalysed by the presence of dilute alkali or acid to form beta unsaturated compound [5]. Chalcones are of a great interest because they have a unique structural feature of having a >C=O functional conjugation with >C=C < and the whole molecule is in conjugation [6, 7]. Chalcones and flavanones are isomeric and undergo interconversion readily, acid or alkali acts as a catalyst and the change can takes place in either direction usually in acid medium, the formation of the flavones is more favorable in alkaline medium [4, 8].

Absorption in the ultraviolet and visible region of the electromagnetic spectrum depends on the electronic structure of molecules. Infrared spectroscopy is very important technique for the identification of functional groups [9, 10]. ^{1}H NMR spectroscopy is widely used to know the environment of protons in the compound. Mass spectroscopy is the most accurate technique for the determination of molecular weight of the compound .In the mass spectroscopy technique matter is bombarded with highly energetic electrons. Then matter absorbs or ejects electrons from it. When it ejects electrons charged species are formed [11]. Importance of Chalcone: the chalcone have been found useful in elucidating structure of natural products like hemlock tannin, cynomaclurin, naringenin etc [12]. Chalcones are colored compounds because of the presence of the chromophore and auxochromes [13].

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In this paper IR, UV-Visible, ^{1}H NMR, Mass spectra, physical properties, CHO analysis and chemical tests of (E)-3-(5-bromofuran-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one are investigated.

MATERIALS AND METHODS

The chalcone of 5-Bromo furfural was synthesized by Claisen-Schmidt condensation of 2, 6-dihydroxy acetophenone and 5-Bromo-2-Furaldehyde .The chemicals used for this synthesis are of AR grade. The mixture of 2,6-dihydroxy acetophenone (0.01 mol) and 5-Bromo-2-Furaldehyde (0.01 mol) are dissolved in ethanol (20ml) and then sodium hydroxide 10ml (40%) were added to it. The mixture was stirred for overnight till yellow brown color ppt was obtained. The progress of reaction was monitored by TLC and the completion of reaction was observed. After completion of reaction the contents were poured into ice cold water and then acidified by dil. HCL. The solid obtained was filtered and crude product was recrystallized from ethanol to give the Chalcone of 5-Bromo furfural. The melting point of the chalcone of 5-Bromo furfural is determined by an open capillary tube and is unconfirmed. Infra red spectrum was measured by using FT-IR spectrophotometer, UV spectrum measured on SL159 single beam UV-VIS spectrophotometer, ^{1}H NMR on Bruker AVANCE II 400 MHz Spectrophotometer in DMSO solvent using TMS and Mass spectrum was measured on Mass spectrophotometer. The purity of the compound was checked by TLC plate which were precoated with silica gel using solvent ethyl acetate and petroleum ether (3:7).The reaction mechanism of formation of Chalcone of 5-Bromo furfural is given figure (1-a) and chemical structure in figure (1-b).

RESULTS AND DISCUSSION

Properties:

The Chalcone of 5-Bromo furfural having IUPAC name (E)-3-(5-bromofuran-2-yl)-1-(2, 6-dihydroxyphenyl) prop -2-en-1-one was synthesized by Claisen-Schmidt condensation method, its structure is stable at room temperature , insoluble in water and is soluble in organic solvent. The stoichiometry of the compound represented as 1:1, 5-Bromo-2-Furaldehyde and 2, 6-dihydroxy acetophenone ratio. The properties of 5-Bromo furfural chalcone and CHO analysis are given in table no. (1). The completion of reaction was checked by thin layer chromatography, Wilson's test, $FeCl_3$ test and $KMNO_4$ test. The reaction

between 5-Bromo-2-Furaldehyde and 2, 6-dihydroxy acetophenone is shown in figure (1-a).From the reaction mechanism it is observed that condensation of aldehyde group of 5-Bromo-2-Furaldehyde and ketone group of 2, 6-dihydroxy acetophenone takes place in the presence of alkali catalyst.

Infra red spectrum:

Infrared spectroscopy is very important technique for the identification of functional groups and bonding sequences in the compounds by the absorption of light in the infrared region of the electromagnetic spectrum. Infrared radiations are characterized by frequency (υ) and wavelength (λ) that are related by the speed of light (c) as $\upsilon = \frac{c}{\lambda}$.

Higher numbers are there to the left of the spectrum because it is the wavelength that is being scanned. The infrared spectrum of Chalcone of 5-Bromo furfural was recorded on a Perkin-Elmer Spectrum RX-IFTIR Spectrophotometer in the range 4000-400 cm^{-1} using potassium bromide pellet at CIL, Chandigarh, Punjab. The Infrared spectrum of Chalcone of 5-Bromo furfural is represented figure (2) and the stretching frequency for different groups in table no. (2). The α, β-unsaturated carbonyl group, characteristic of Chalcone of 5-Bromo furfural appears at 1627 cm^{-1} which agrees with the standard value for the chalcone 1625-1650 cm^{-1}.

UV-Visible spectrum:

Absorption in the ultraviolet and visible region of the electromagnetic spectrum depends on the electronic structure of molecules. The energy absorbed in ultraviolet region produces changes in the electronic energy of molecule resulting in the transition of valance electrons.

The ultra violet visible spectrum of Chalcone of 5-Bromo furfural shows that major absorption bands occur in the range 230 to 490 nm. The UV-Visible spectrum is given in figure (3) and corresponding data in table no. (3).

^{1}H NMR spectrum:

The chemical shift (δ) of proton depends upon the factors such as electro negativity, electron density which causes particular nuclei to appear at different chemical shift (δ).Greater the shielding effect, lower the chemical shift and opposite of this character is deshielding effect. The ^{1}H NMR spectrum Chalcone of 5-Bromo furfural is recorded on Bruker AVANCE II 400 MHz Spectrophotometer in DMSO solvent using TMS as an internal standard at SAIF, Chandigarh, Punjab are shown in figure (4) and spectral data in table no. (4) [14].

Mass Spectrum:

Mass spectroscopy is the most accurate technique for the determination of molecular weight of compound. In this technique matter is bombarded with highly energetic electrons.

$M - e^- \longrightarrow \overset{\bullet}{M}^+ + 2e^-$ leaves behind a radical cation.

$M + e^- \longrightarrow M^{\bullet -}$ leaves behind an anionic radical.

The mass spectrum is a plot representing the m/e values of various ions against their relative percent intensity. The intensity of other peaks is shown relative to the base peak. The peak at extreme right corresponds to the molecular weight of the original molecule. The molecular ion is called parent ion and usually denoted as $[M]^+$ ion. In mass spectrum $[M^+ + 1]$ and $[M^+ + 2]$ peaks are also noticed. Mass spectroscopy also separates the isotopes [15, 16]. The mass spectrum Chalcone of 5-Bromo furfural was recorded on Waters, Q-TOF Micro Mass (LC-MS).at SAIF, Chandigarh, Punjab. In the mass spectrum of the Chalcone of 5-Bromo furfural molecular ion peak is observed m/z and calculated m/z corresponding to $[M^+]$, peaks are in good agreement with chalcone structures shown in figure(5) and table(5).

Table No. 1: Properties of Chalcone of 5-Bromo furfural

Mol. Formula.	Color	Mol. Wt.	M.P °c	Found (Calculated) %					
				C	H	O	N	S	Br
$[C_{13}H_9O_4Br]$	Yellowish brown	309	241	50.49 (50.51)	2.7 (2.9)	20.69 (20.70)	---	---	25.82 (25.85)

Table No.2: Stretching frequency of functional groups in Chalcone of 5-Bromo furfural

Ligand	υ(OH) Enolic	(-CO-CH=CH-) α,β-unsaturated carbonyl group	(C-O-C) Stretching Frequency	(C=C) Stretching Frequency	Aromatic Ring (C=C) Stretching Frequency	Ar-H Stretching Frequency	Br - stretching frequency
Chalcone of 5-Bromo furfural	3286 cm^{-1}	1627 cm^{-1}	1095 cm^{-1}	1589 cm^{-1}	1516 cm^{-1}	3012 cm^{-1}	961(C-Br)

Table No. 3: UV spectral data of Chalcone of 5-Bromo furfural

Absorption spectra	Wavelength
absorbance	λ(nm)
0.0564	271.0
0.5045	346.5

Table No. 4: Chemical shift (δ) ppm and number of protons of (E)-3-(5-bromofuran-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one

Chemical Shift (δ) ppm.	Number of Protons	Multiplicity (Splitting)	Assignment
6.48-7.20	3H	m	Aromatic protons
5.0	2H	S	-OH group present on aromatic benzene ring.
7.56	1H	d	α-H on-unsaturated carbonyl system
7.90	1H	d	β-H on-unsaturated carbonyl system
7.55-7.86	2H	d	Protons on furan ring.

Table No. 5: Mass spectral analysis of Chalcone of 5-Bromo furfural

Molecule	$[M^+]$ Molecular Mass Found (Calculated)
$C_{13}H_9O_4Br$	313

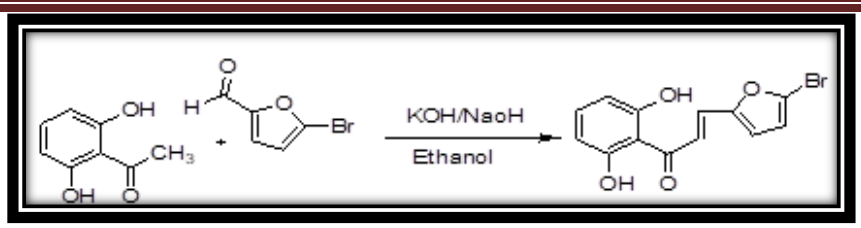

Fig. 1: (a) Reaction

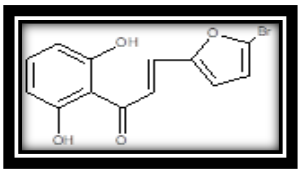

Fig. 1: (b) Chemical structure

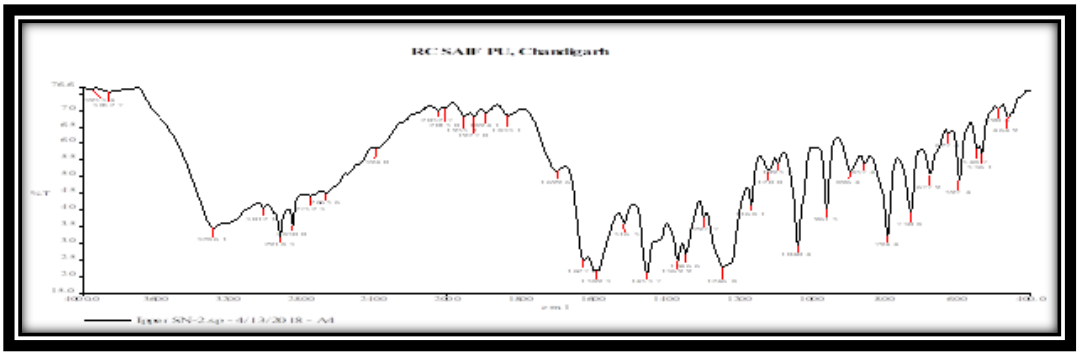

Fig. 2: IR spectrum

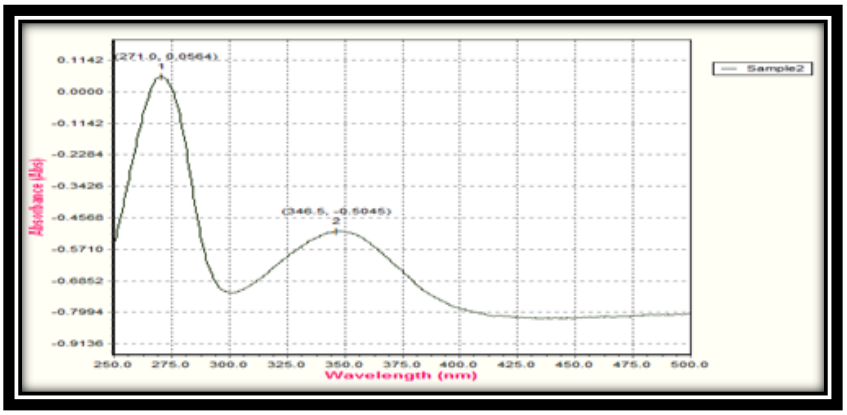

Fig. 3: UV-visible spectrum

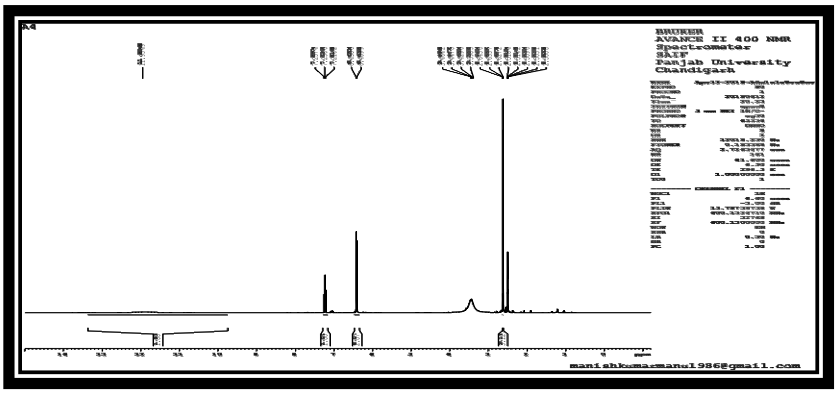

Fig. 4: ^{1}H NMR spectrum

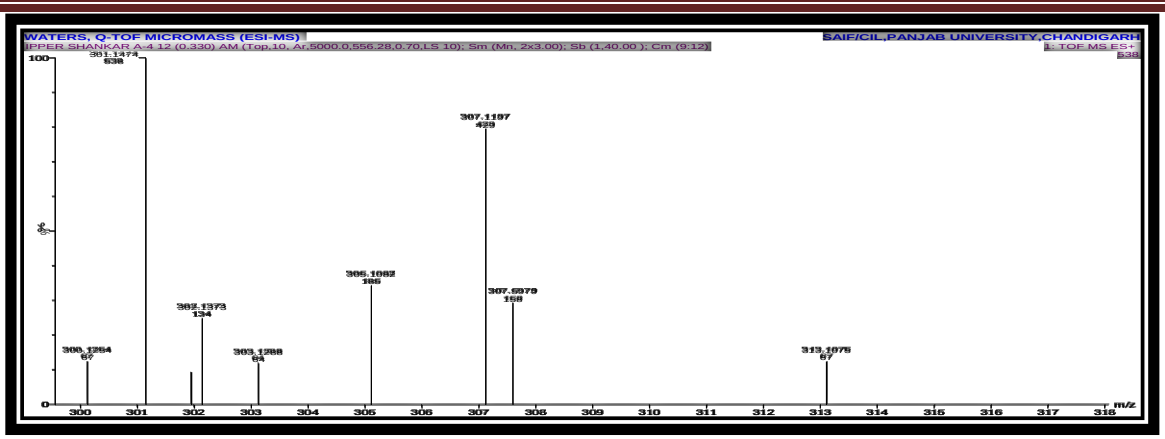

Fig. 5: Mass spectrum

CONCLUSION

(E)-3-(5-bromofuran-2-yl)-1-(2,6-dihydroxyphenyl)prop-2-en-1-one was synthesized by Claisen-Schmidt condensation method in alkaline medium. This is stable yellowish brown solid compound with molecular formula [$C_{13}H_9O_4Br$].The structure of this compound is confirmed by UV-visible, Infrared, ^1H NMR, Mass spectroscopy and CHO analysis. Elemental analysis showed that the percentage of the Bromine, hydrogen and carbon was found experimentally equivalent to the calculated values.

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