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Synthesis, spectral study and properties of Pyridine chalcone

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Abstract

Pyridine chalcone is open chain containing α, β unsaturated carbonyl group consisting of two aromatic rings. Pyridine chalcone is synthesized by Claisen-Schmidt condensation method in alkaline solution. Spectroscopic characterization using Electronic absorption, Infra red, ^{1}H NMR spectra and chemical tests has are studied for Pyridine chalcone. Physico-chemical properties, ^{1}H NMR, IR, Electronic absorption spectra and CHO analysis study of this compound shows that it is novel Pyridine chalcone.

Keywords: Pyridine chalcone, ^{1}H NMR spectrum, properties, Infra red spectrum, Electronic absorption spectrum, CHO analysis, Claisen-Schmidt condensation method.

1. Introduction

The chemistry of chalcones has generated intensive scientific studies throughout the world. Especially interest has been focused on the synthesis and biodynamic activities of chalcones. The chalcone name was firstly given by Kostanecki and Tambar. The chalcones (1, 3-di-azyl-2-propenones) and their derivatives are important intermediates in organic synthesis [1]. They serve as starting materials for the synthesis of variety of heterocyclic compounds which are due to the presence of enone functionality in chalcone moiety confers biological activity upon it, like anti-inflammatory, antifungal ,antioxidant, antimalarial .Chalcones are open chain containing α, β- unsaturated carbonyl group consisting of two aromatic rings (ring A & B) having diverse any of substituent's . In this paper we synthesize Pyridine chalcone by Claisen-Schmidt condensation method and characterize it by Infra red, Electronic absorption, ^{1}H NMR spectra, Wilson's test, $FeCl_3$ test, unsaturation test with $KMnO_4$. Pyridine chalcone is a basic heterocyclic organic compound with the chemical formula $C_{14}H_{11}NO_3$ [2-3].

2. METHOD

Pyridine chalcone is synthesized by Claisen-Schmidt condensation of pyridine-2-carbalaldehyde and 2, 6-dihydroxy acetophenone by base catalyzed followed by dehydration. The chemicals used for preparation of Pyridine chalcone are of A.R. grade. The mixture of 2, 6-dihydroxy acetophenone (0.01 mol) and pyridine-2-carbaldehyde (0.01 mol) are dissolved in ethyl alcohol (25ml) and then potassium hydroxide 10ml (40%) were added to it. The reaction mixture was heated for 3 hours till yellowish brown color ppt was obtained [4-6]. The progress of reaction was monitored by TLC. After completion of reaction the contents were poured into ice water and then acidified by dil. HCl. The solid obtained was filtered and crude product was recrystallized from ethyl alcohol to give the Pyridine chalcone [7-10]. The reaction of formation of Pyridine chalcone is given in figure (1). The melting point of the Pyridine chalcone is determined by an open capillary tube and is unconfirmed. Infra red spectrum is recorded using FT-IR spectrophotometer, ^{1}H NMR spectrum is recorded on Bruker AVANCE II 400 MHz Spectrophotometer in DMSO solvent using TMS as an internal standard at SAIF, Chandigarh ,Punjab and Electronic absorption spectrum measured on SL159, single beam UV-VIS spectrophotometer. The purity of compound is checked by TLC plate, which were precoated with silica gel using solvent ethyl acetate and petroleum ether (3:7). The IUPAC name of this compound is (E)-1-(2, 6-dihydroxyphenyl)-3-(pyridin-2-yl)prop-2-en-1-one.

3. RESULT AND DISCUSION

3.1 Properties

The Pyridine chalcone having IUPAC name 1-(2, 6-di-hydroxyphenyl)-3-(1 H-pyrrole-2-yl) prop-2-en-1-one were synthesized by Claisen-Schmidt condensation method and its structure is stable at room temperature, insoluble in water and is soluble in organic solvent(ethyl alcohol). The stoichiometry of the compound represented as 1:1 pyridine-2-

carbaldehyde and 2, 6-dihydroxy acetophenone ratio. The physico-chemical properties of Pyridine chalcone are given in table no. (1) and CHO analysis by calculation method in table no. (2).The completion of reaction was checked by thin layer chromatography, Wilson's test, $FeCl_3$ test. The reaction between pyridine-2-carbaldehyde and 2, 6-dihydroxy acetophenone is shown in figure (1).From the reaction mechanism it is observed that condensation of aldehyde group of pyridine-2-carbaldehyde and ketone group of 2, 6-dihydroxy acetophenone takes place in the presence of alkali catalyst.

3.2 Infra red spectrum

In infrared spectrum the sample is exposed to infrared radiation and the wavelength scanned across the spectrum. Whenever energy corresponding to specific wave length is absorbed, the intensity of the radiation reaching a detector momentarily decreases, and this is recorded in the spectrum. Infrared spectra are usually recorded as frequency measurement called wave number (cm^{-1}) which is the inverse of the true wavelength λ in centimeters to give convenient numbers (400-4000 cm^{-1}) [11-13]. Higher numbers are to the left of the spectrum because it is really wavelength that is being scanned. The infrared spectra of pyridine chalcone were recorded on a Perkin- Elmer Spectrum RX-IFTIR Spectrophotometer over the range 4000-400 cm^{-1} using KBr pellet at CIL, Chandigarh, Punjab. The Infrared spectrum of pyridine chalcone is represented figure (2) and the stretching frequency for different groups in table no. (3).

3.3 Electronic absorption spectrum

The absorption of ultraviolet (UV) or visible light results in a change in energy of absorbing molecule. The electronic spectrum consists of bands containing several absorption lines. Each band corresponds to a definite change in the electronic energy. The ultra violet visible spectrum of pyridine chalcone shows that major absorption bands usually occur in the range 230 to 490 nm. The electronic absorption spectrum is given in figure no. (3) and corresponding data in table no. (4).

3.4 ^1H NMR spectral study of Pyridine chalcone

Nuclear Magnetic Resonance Spectroscopy is possible due to the absorption of energy at particular frequency by atomic nuclei, characterized by a property, termed spin and this gives rise to a magnetic moment associated with that nuclei. The frequency at which proton absorbs energy depends on electronic environment present around that proton. 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. In the analysis of organic molecule, ^1H NMR spectra plays very important role. It is a most valuable technique in structural investigation [14]. The ^1H NMR spectrum of pyridine chalcone 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. (5).

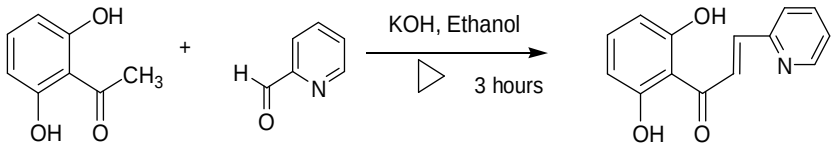

Fig. (1): Reaction of Pyridine chalcone.

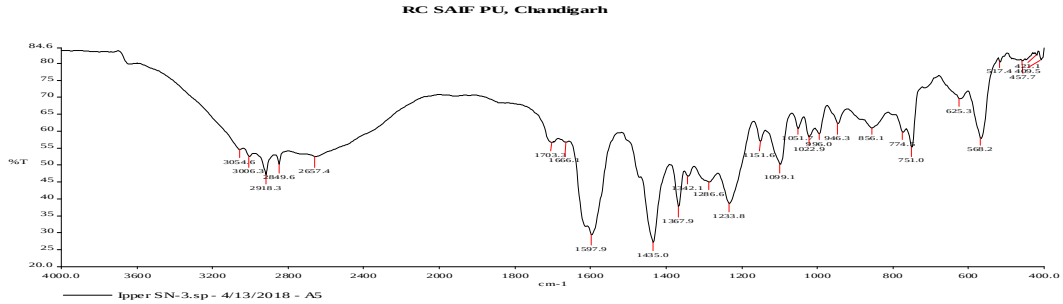

Fig. (2): Infra red spectrum for Pyridine chalcone.

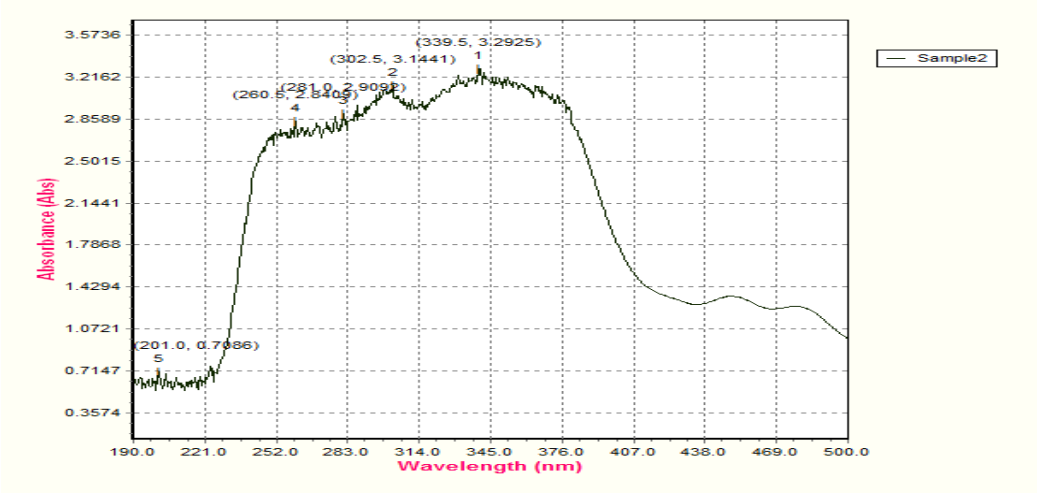

Fig. (3): Electronic absorption spectrum.

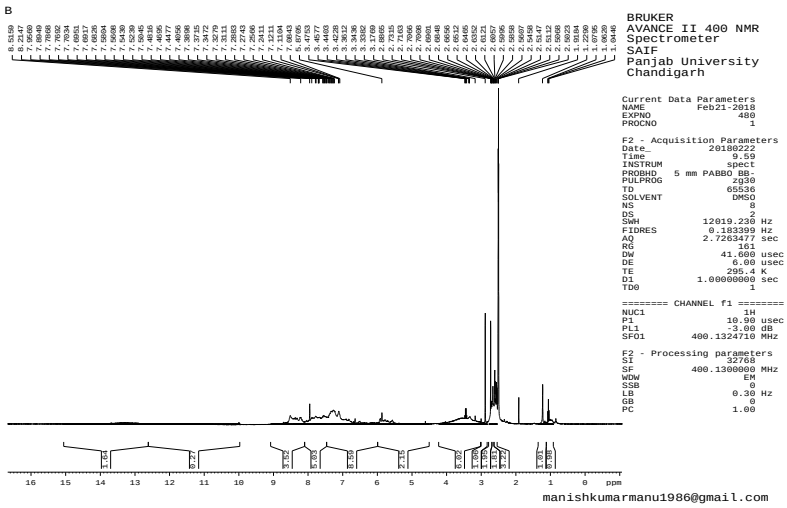

Fig.(4): ^{1}H Nuclear Magnetic Resonance Spectrum of pyridine chalcone

Table no. (1): The properties of pyridine chalcone

Property	Color	M.P. 0c	Yield	Chemical name	Chemical formula	Molecular weight	Phase
Observation	Yellow	255	90 %	Pyridine chalcone	C14H 11O3N	241	Solid(Yellowish brown crystals)

Table no. (2): Elemental analysis data of Pyridine chalcone ($C_{14}H_{11}O_3N$) and chemical structure**.**

CHO analysis Calculated in %			
C	H	O	N
68.69 (69.70)	4.61 (4.60)	20.94 (19.90)	5.80 (5.81)

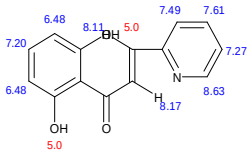

Table no. (3): The stretching frequency of different groups

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**
3055 cm^{-1}	1666 cm^{-1}	1099 cm^{-1}	1597 cm^{-1}	1435 cm^{-1}	3001 cm^{-1}

Table no. (4): Electronic absorption spectrum: absorption and corresponding wave length for Pyridine chalcone.

Absorption spectra	Wavelength
absorbance	λ(nm)
3.0392	260
3.4037	302
3.5364	329

Table no. (5): ^{1}H Nuclear Magnetic Resonance Spectral data of pyridine chalcone

Chemical Shift (δs) 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.
8.17	1H	d	α-H on-unsaturated carbonyl system
8.11	1H	d	β-H on-unsaturated carbonyl system
7.27-8.63	4H	m	Protons on pyridine ring..

4. CONCLUSION

The Pyridine chalcone having IUPAC name 1-(2, 6-di-hydroxyphenyl)-3-(1 H-pyrrole-2-yl) prop-2-en-1-one were synthesized by Claisen-Schmidt condensation method and its structure is confirmed by IR , ^{1}HNMR, UV spectra, CHO analysis, physical properties, Wilson's test, $FeCl_3$ test and unsaturation test with $KMnO_4$. This is novel Pyridine chalcone. The reaction mechanism for the formation of Pyridine chalcone shows condensation of aldehyde group of pyridine and ketone group of 2, 6-di-hydroxy acetophenone takes place.

5. ACKNOWLEDGEMENT

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