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Synthesis and Characterization of new Schiff Base Derived from 1,2-Di (indol-2-yl) - 2-hydroxyethanone.

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ABSTRACT

The reaction of two moles of some aromatic aldehyde to form a new carbon-carbon bond by catalytic effect of cyanide ion in alcoholic solvents is known as the benzoin condensation. The benzoin (α-Hydroxy ketone) having both a secondary alcohol and a ketone functional group, the react of ketone group with primary amine a Schiff base is produced, which is a compound containing azomethine group, a compound containing azomethine group, *R*–*C*=*N*–. (Holm et al., 1966; Hobday and Smith, 1972).

Keywords: Aromatic aldehydes, Benzoin, amine, α-Hydroxy ketones

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INTRODUCTION

The synthesis of α-Hydroxy carbonyl derivatives has been of continuous interest to organic chemists since the beginning of the century. Because chiral α-Hydroxy ketones are important reagents for the synthesis of complex optically active natural products and are useful stereodirecting groups [1]. There are both chemical and biotechnological methods for the synthesis of α-Hydroxy ketones. Indol-2-carbaldehyde under the influence of sodium cyanide in aqueous alcohol undergone a dimolecular condensation reaction and gave benzoin. The Schiff base compounds were synthesis by reaction of 1,2-Di(indol-2-yl)-2-hydroxyethanone with many of aromatic amine derivatives. Many biologically important Schiff bases have been reported in the literature possessing, antibacterial, antifungal, antimicrobial, anticonvulsant, antiHIV , anti-inflammatory, antitumor [1-13].

EXPERIMENTAL SECTION

General

All chemicals are commercially available and used as received. Melting points were determined on a stuart melting point SMP30 apparatus and are uncorrected. The FTIR spectra were recorded on Shimadzu FTIR -8400S spectrophotometer using KBr disc. The 1H-NMR spectra were recorded on a Fourier transform Bruker spectrometer operating at 300 MHz in detoured chloroform with tetramethylsilane as internal standard in DMSO- d [6] . The compounds were analyzed for elemental analysis and the percentage of elements were found to be very near that of the calculated values

General Procedure for the Synthesis ofα-Hydroxy ketone

A solution of sodium cyanide (2 mmol, 0.098 g) in H_2O (2 ml), was added to a stirred solution of a Indol-2-carbaldehyde (10 mmol) in ethanol solvent (10 ml) . The mixture was then refluxed. The progress of reaction was monitored by TLC using chloroform as eluent. The solvent was then removed by evaporation under reduced pressure. The residue was washed with water and ethanol.

General Procedure for the Synthesis ofSchiff base

The solid intermediate **(1)** (0.01mol) was, added to an ethanolic solution (20ml) of various amines (0.01 mol). The mixture was, refluxed for about 4 hours. The reaction was, followed using TLC. The contents were, poured in to crushed ice. The crystals were washed with ethanol. The solid product **(2_{a-d})** was, separated, filtered and recrystallized from ethanol.

1,2-Di(indol-2-yl)-2-hydroxyethanone (1)

Compound **1** was synthesized according method **a** .yellow solid, Yield: % 86.18 (3.8 g). melting point : 193-194 C^o, IR(KBr): ν (cm-1) = 3359 (O-H), 3032 (C-H_{arom}), 2902-2816 (C-$H_{aliphatic}$), 1635 (C=O), 1558 (C=C), 1242 (C-O). 1H-NMR (DMSO-d_6, ppm): δ=2.42 ppm (d, H, OH), δ=4.43 ppm (d, H, CH-O), δ=6.11 ppm (s, H, CH=C), δ=6.26-6.93 ppm (m, 10H. CH $_{arm}$), δ=9.10 ppm (s, 2H, NH).

(2-Hydroxy-1,2-di(indol-2-yl)ethylideneamino) benzoic acid (2 a)

Compound **2a** was synthesized according method **b**.brawn solid, Yield: % 64.2 (0.09 g). melting point : 153 C^o, IR(KBr): ν (cm-1) = 3363 (O-H), 3100 (O-H $_{carboxylic}$), 3024- 2921(C-H_{arom}), 2885 (C-H $_{aliphatic}$), 1624 (C=O), 1573 (C=N), 1513 (C=C), 1296 (C-O), 1212 (C-N). 1H-NMR (DMSO-d_6, ppm): δ=2.02 ppm (d, H, OH), δ=3.36 ppm (d, H, CH-O), δ=6.21 ppm (s, H, CH=C), δ=7.21-8.53 ppm (m,14H. CH_{arm}), δ=8.98 ppm (s, 2H, NH), δ=10.25 ppm (s, H,COOH).

(4-nitrophenylimino)-1,2-di(indol-2-yl)ethanol (2 b)

Compound **2b** was synthesized according method **b**.yellow solid, Yield: % 64.2 (0.09 g). melting point : 174-176 C^o, IR(KBr): *ν* (cm^{-1}) = 3470 (O-H), 3047- 2931(C-H_{arom}), 2885 (C-H $_{aliphatic}$), 1635 (C=O), 1519 (C=N),

1442 (C=C), 1388 (NO_2), 1303 (C-O), 1212 (C-N),763 (NO), 1H-NMR (DMSO-d_6, ppm): δ=3.01 ppm (d, H, OH), δ=3.20 ppm (d, H, CH-O), δ=6.58 ppm (s, H, CH=C), δ=7.23-8.92 ppm (m,14H. CH_{arm}), δ=9.02 ppm (s, 2H, NH).

(2,4-dichlorophenylimino)-1,2-di(indol-2-yl)ethanol (2 c)

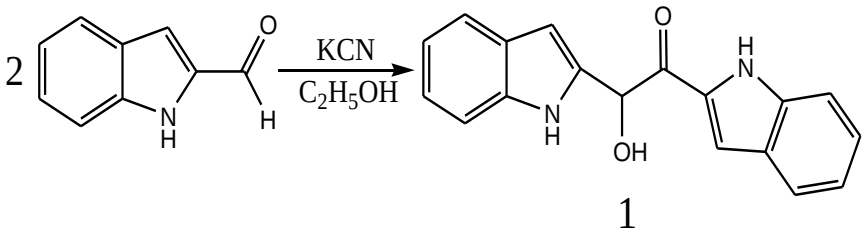
Compound **2c** was synthesized according method **b**.pink solid, Yield: % 57.14 (0.08 g). melting point : 188.8 C^o, IR(KBr): *v* (cm^{-1}) = 3317 (O-H), 3101-2929 (C-H_{arom}), 2862 (C-$H_{aliphatic}$), 1640 (C=O), 1573 (C=N), 1442 (C=C), 1388 (C-O), 1290 (C-N), 700 (C-Cl), 1H-NMR (DMSO-d_6, ppm): δ=2.01 ppm (d, H, OH), δ=4.52 ppm (d, H, CH-O), δ=6.10 ppm (s, H, CH=C), δ=6.81-7.32 ppm (m,13H. CH_{arm}), δ=9.32 ppm (s, 2H, NH).

(4-methylphenylimino)-1,2-di(indol-2-yl)ethanol (2 d)

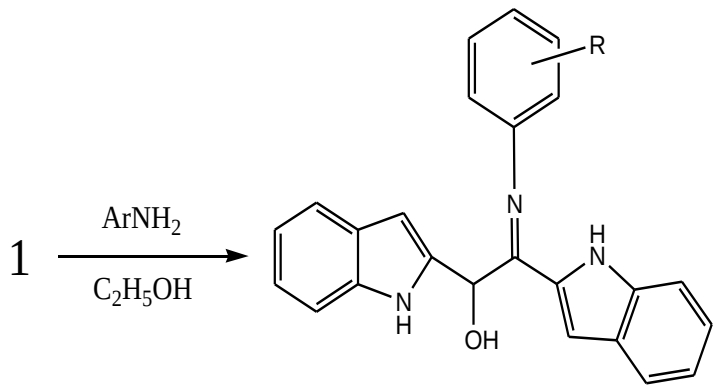
Compound **2d** was synthesized according method **b**.brawn solid, Yield: % 69.4 (0.09 g). melting point : 174 C^o, IR(KBr): *v* (cm^{-1}) = 3394 (O-H), 3109-2039 (C-H_{arom}), 2931-2816 (C-$H_{aliphatic}$), 1638 (C=O), 1570 (C=N), 1445 (C=C), 1380 (C-O), 1242 (C-N). 1H-NMR (DMSO-d_6, ppm): δ=2.01 ppm (d, H, OH), δ=2.52 ppm (s, 3H, CH_3), δ=4.22 ppm (d, H, CH-O), δ=6.74 ppm (s, H, CH=C), δ=6.88-7.36 ppm (m,14H. CH_{arm}), δ=9.39 ppm (s, 2H, NH).

RESULTS AND DISCUSSION

In ethanol solution the Indol-2-carbaldehyde was refluxed in the presence of catalytic amounts of KCN converted to unknown (α-Hydroxy ketone) 1,2-Di(indol-2-yl)-2-hydroxyethanone **(1)**. The Schiff bases were obtain by reaction of **(1)** with various amines in alcoholic solution. (Scheme 1)



1



2

2	R
a	COOH
b	NO_2
c	Cl
d	CH_3

(Scheme 1)

The IR spectrum of compound 1 show bonds of medium intensity at 1242, 1636 and 3359 cm^{-1} which are for υ (C-O), υ (C=O) and υ (O-H), respectively. The IR spectra of entries 2a-d also show υ OH at 3363, 3479, 3317 and 3394 cm-1, respectively. The N-H proton of appeared in the 1H NMR spectrum as singlet at 9.1 ppm as shown in Figure 3.The COOH proton of **2a** appeared in the 1H NMR spectrum as singlet at 10.2 ppm and the CH3 proton of **2d** appeared in the 1H NMR spectrum as singlet at 2.29 ppm.

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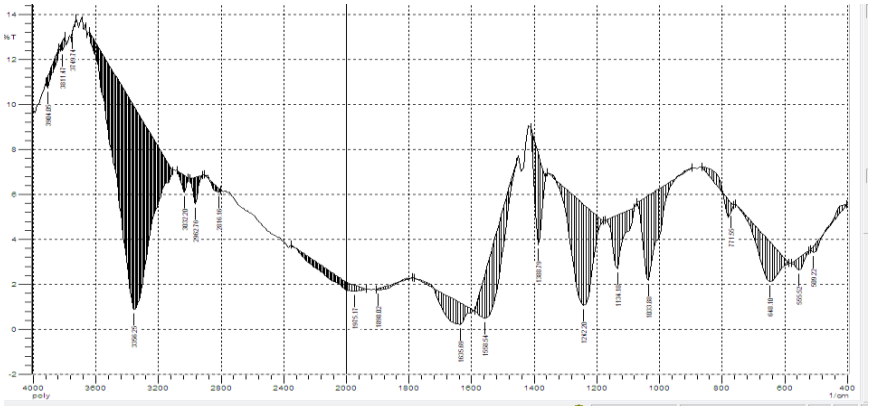

Figure 1: IR spectra for 1 compound

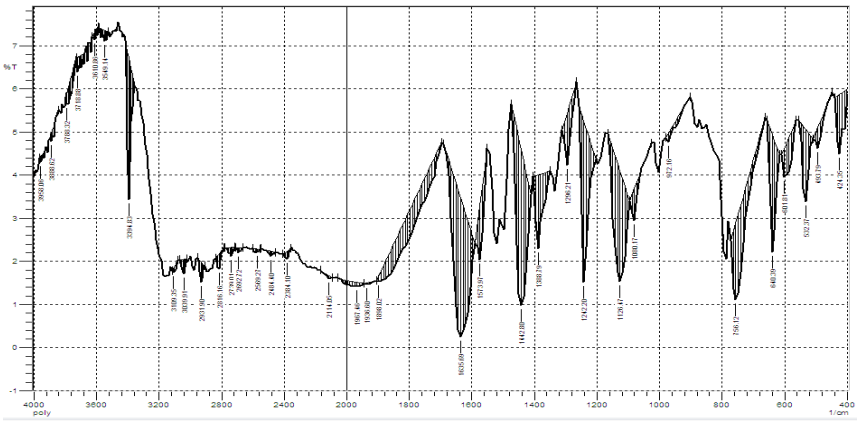

Figure 2: IR spectra for 2d compound

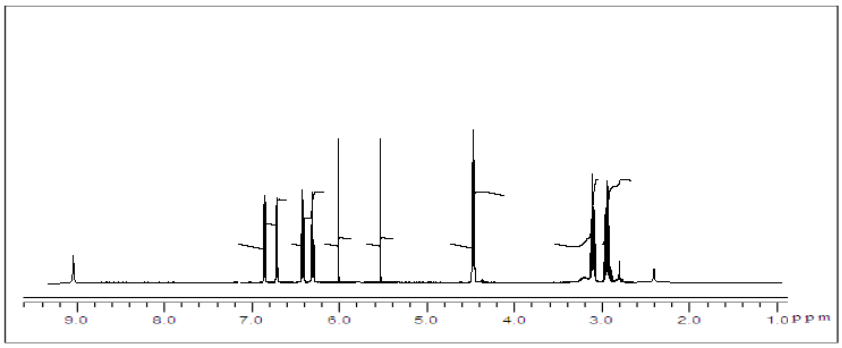

Figure 3: HNMR spectra for 1 compound

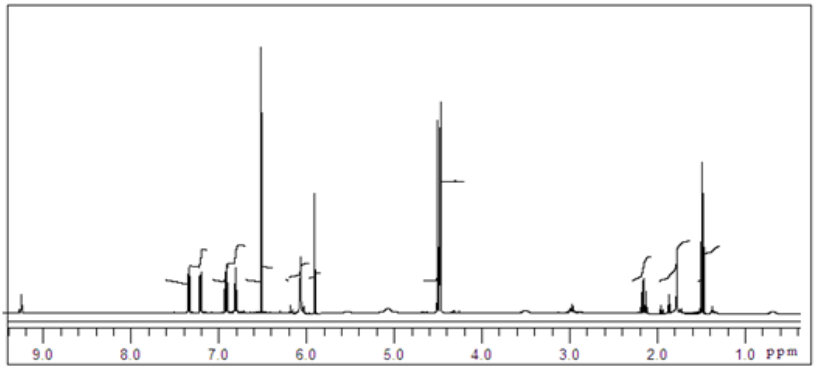

Figure 4: HNMR spectra for 2c compound

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CONCLUSION

Unknown α-hydroxy ketone was synthesis and use it tosynthesis of new Schiff base by using versus amines.

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