### FLUORESCENCE STUDY OF ZN (II), CU (II), NI (II) AND LA (III) COMPLEXES OF 3-{(*E*)-[(4-IODOPHENYL)IMINO]METHYL}QUINOLIN-2-OL

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### Abstract :

A simple and regioselective synthesis of 2-chloro-3-formylquinoline (1) by the cyclisation of Narylacetamide has been reported by the Vilsmeier Haack reaction/cyclisation which is further undergoes hydroxylation to 3-formyl-2-hydroxyquinoline by using acetic acid. In 3-formyl-2hydroxyquinoline, the formyl group shows condensation with p-iodoaniline and forming schiff base as  $3-\{(E)-[(4\text{-iodophenyl}))\text{imino}]$ methyl $\}$ quinolin-2-ol. It acts as 1,5-bidentate ligand and forming complex of the type [ML<sub>2</sub>] where M is Zn, Cu, Ni and La which are further subjected to fluorescence study. The Schiff base shows weak emission at 461 nm (weak) for the absorption wavelength 387 nm whereas its complexes shows strong emission at 462 nm (moderate) [ZnL<sub>2</sub>, at 387 nm], 478 nm (strong) [CuL<sub>2</sub>, at 387 nm], 465 nm (moderate) [NiL<sub>2</sub>, at 387 nm] and 461 nm (moderate) [LaL<sub>2</sub>, at 387 nm]. The complexes having very high quantum efficiency than the schiff base.

Key words: p-Iodoaniline, Formylation, Vilsmeier Haack reaction, quinoline, Schiff bases.

# 1. INTRODUCTION

Quinolines and benzo/hetero fused analogues have attracted great attention of medicinal and synthetic chemist because of their presence in natural products and have good physiological activities. The Vilsmeier Haack reagent has been proved to be versatile synthetic reagent and used for the various synthetic transformations<sup>I</sup>. It has variety of synthetic applications such as used for formylation<sup>II</sup>, cyclohaloaddition<sup>III</sup>, cyclisation<sup>IV</sup>, condensation, ring annulations<sup>V</sup>, etc. There are many routes have been developed for the functionalization of quinolines, the Vilsmeier approach is found to be most efficient for achieving useful transformation and heteroannulations. The versatile application in the synthesis has been proved by the synthesis of 4-(N,N-dimethylaminomethylene)-2-alkyl/aryl-2-oxazolin-5-one<sup>VI</sup> from the N-acyl derivatives of  $\alpha$ -amino acid esters and  $\alpha$ -aminoacetanilides. Wall J et al has been developed novel quinoline based fused heterocyclic system as potential anticancer agents<sup>VII</sup>. Harrowven D C et al suggested that quinoline nucleus with different substituents at 2- and 3- positions was versatile synthem for the heteroannulation<sup>VIII</sup>. Ambika Srivastava et al has been carried out the transformation of the 2-chloro and 3-formyl groups into different functionalities<sup>IX</sup>. T Suresh et al has been carried out the annulations of 3-formyl-2-hydroxycoumarin and its derivatives.

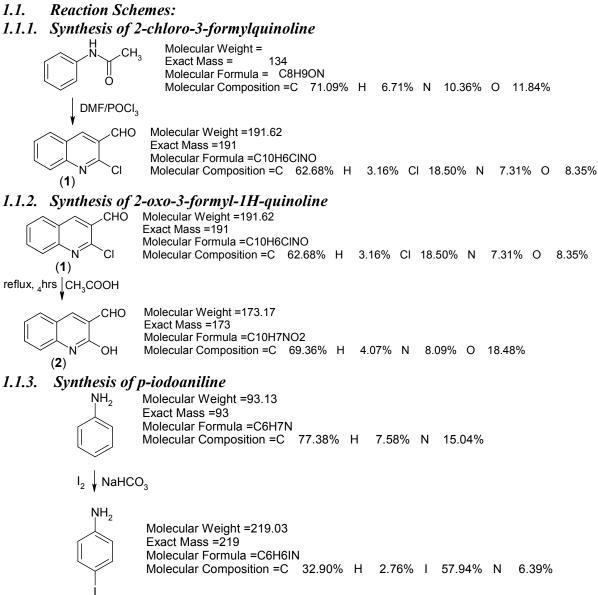
The word phosphor was invented in the early 17<sup>th</sup> century. There are some characteristics of typical phosphor as- must survive hazardous chemical environment, cannot be water soluble, durable, easy to apply, not easily detected or noticed without specialized equipment, etc. Phosphors become technologically and industrially important with the introduction of fluorescent lamps in 1938. Thermometry was suggested in the German patent in 1938. First peer-reviewed article, to our knowledge appeared in 1949. Between 1950 to 1980, it was not widely used. Its most common use was aerodynamic applications. Advances in lasers, microelectronics, and other supporting technologies enable additional commercial as well as scientific use. The physics and chemistry of luminescence materials and their applications become and still is the core area covered by Luminescence symposia.

In the small molecules organic light emitting diodes, the family of carbazoles<sup>X</sup> could be extended to be suitably fit for red<sup>XI-XII</sup>, green<sup>XIV-XVI</sup>, and blue light<sup>XVII-XIX</sup> triplet emitters and therefore, they can be used in full color displays<sup>XX-XXIII,XXV-XXVIII</sup>. More recently studies of Thompson, Forrest and co-workers shows that the use of electron blocking layers (EBLs) consisting of Ir<sup>III</sup> complexes with picolinate ligands produced improved color purities in the case of blue light emitting device<sup>XXIX</sup>. Some of the organic molecules are used as EBLs as- fluorinated phenylenes<sup>XXIV</sup>, and oxadiazole as well as triazole containing molecules such as trimer of Narylbenzimidazoles (TPBi)<sup>XXX-XXXI</sup>, 2-tert-butylphenyl-5-biphenyl-1,3,4-oxadiazole (PBD)<sup>XXXII</sup>, 3-phenyl-4-(1'-naphthyl)-5-phenyl-1,2,4-triazole (TAZ)<sup>XXXIII-XXXIV</sup>, 1,8-Naphthalimides<sup>XXXV</sup>, polyquinolines<sup>XXXVI</sup>, or carbon nanotubes doped in PPV<sup>XXXVII</sup> were also found to be useful as hole blocking layers (HBLs). Metal complexes of the heavy metals such as Gold, Tb(III), Eu(III), Ln(III), Y(III), Gd(III), Pt(II) and Ir(III)(can be used as efficient phosphorescent emitters)<sup>XXXVIII-XLVII</sup> are used as organic phosphors. Zhao et al developed white light emitting electroluminescent devices using lanthanide binuclear complexes, Tb<sub>(1-x)</sub>Eu<sub>x</sub>(aca)<sub>3</sub>(phen)<sup>XLVIII</sup>.

Searching for highly efficient fluorescence organic compounds (Schiff bases) is a topic of current interest. The aromatic based ligand having electron donating or withdrawing groups has been increased or decreased the intensity of absorption or shifted absorption wavelength on either side. Synthesis and photophysical properties (luminescence) of transition metal complexes [Cu(II) and Zn(II)] of 2-{[(E)-(2-hydroxyphenyl)methylidene]Amine}benzoic acid<sup>L</sup> are studied. Luminescence properties of the various Schiff base of 3-formyl-2-hydroxyquinoline and its Zn, Cu, Ni and La metal complexes have been checked by using Spectrofluorometer model number RF5301. A Xe laser lamp was used for excitation and emission spectra were scanned from the range 220 nm to 750 nm. The emission of Schiff bases at each excitation wavelength has been checked.

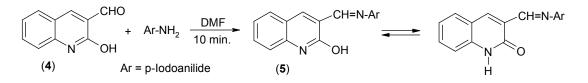
In this paper we tried to synthesis Schiff bases of 3-formyl-2-hydroxyquinoline of p-iodoaniline which has name as  $3-\{(E)-[(4-iodophenyl)imino]methyl\}$ quinolin-2-ol (4) & their metal complexes as Zn, Cu, Ni & La and study their fluorescence properties.

#### **EXPERIMENTAL SECTION:**

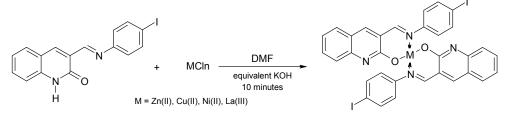


#### 1.1.4. Synthesis of 3-{(E)-[(4-iodophenyl)imino]methyl}quinolin-2-ol.

(3)



1.1.5. Synthesis of ML<sub>2</sub> complexes of 3-{(E)-[(4-iodophenyl)imino]methyl}quinolin-2-ol.



The required acetanilide (pure state) was synthesized from the reaction of aniline and acetic anhydride in aqueous medium. The Vilsmeier cyclisation of acetanilide was carried out by slowly adding POCl<sub>3</sub> (with constant stirring) to a substance in DMF at  $0-5^{\circ}$ C followed by heating to 90°C to obtain 2-chloro-3-formylquinoline (1) in good yield. The 2-chloro-3-formylquinoline (1.93 g, 0.01 mol) reacts with 70% dilute acetic acid gave 2-oxo-3-formyl-1H-quinoline (2). The structures of the compound (1) and (2) can be confirmed by spectral data and chromatographic technique. The p-iodoaniline (3) can be synthesized by following known methods (referring Vogel's Organic Chemistry practical book, 5<sup>th</sup> edition). The formation of Schiff bases (4) can be confirmed by using NMR data. It is good 1,5-bidentate ligand and forming metal complexes with Zn, Cu, Ni and La by using metal halide.

# 2.2. Experimental Procedures:

# 2.2.1. Synthesis of 2-chloro-3-formylquinoline (1)

To a solution of acetanilide (5 mmol) in dry DMF (15 mmol) at 0 - 5 <sup>0</sup>C, POCl<sub>3</sub> (60 mmol) was added dropwise with stirring and mixture was then stirred at 80 – 90 <sup>0</sup>C for 9 hrs. The resulting mixture was poured in crushed ice, stir for 5 min and resulting solid was filtered, washed well with water and dried. The compound was recrystalized from ethyl acetate. The m.p. and yield of the compound is 148 <sup>0</sup>C and 78% respectively.

PMR (DMSO-D<sub>6</sub>): 10.4 (s, 1H), 8.75 (s, 1H), 8.15 (m, 1H), 7.97 (m, 1H), 7.92 (m, 1H), 7.78 (m, 1H).

# 2.2.2. Synthesis of 2-oxo-3-formyl-1H-quinoline (2)

The suspend 2-chloro-3-formylquinoline (1.93 g, 0.01 mol) reacts with 70% dilute acetic acid (10 ml) and heated under reflux for 6 hrs. The completion of reaction was confirmed by taking TLC time to time. Cool the reaction mixture, a solid product 2-oxo-3-formyl-1H-quinoline was precipitated out, filters and washed with water, dried and purified by recrystallisation from DMF. Record m.p.  $303-04^{\circ}C$  and yield 93%.

PMR (DMSO-D<sub>6</sub>): 12.24 (bs, 1H, OH, D<sub>2</sub>O exchange); 10.24 (s, 1H); 8.54 (s, 1H); 7.92 (d, 1H); 7.66 (t, 1H); 7.38 (d, 1H); 7.28 (t, 1H).

# 2.2.3. Synthesis of p-Iodoaniline

Into a 1-liter beaker provided with a mechanical stirrer place 37 g (36 ml, 0.4 mol) of aniline, 50 g (0.6 mol) of sodium hydrogen carbonate and 350 ml of water; cool to 12 - 15 <sup>0</sup>C by addition of a little crushed ice. Stir the mixture, and introduce 85 g (0.33 mol) of powdered resublimed iodine in proportion of 5 - 6 g at interval of 2 - 3 min so that all the iodine is added during 30 min. continue stirring for 20 - 30 min, by which time the color of free iodine in the solution has practically disappeared and the reaction is complete. Filter the crude p-iodoaniline with suction on a Buckner funnel, drain as completely as possible and dry it in the air. Save the filtrate for the recovery of iodine.

Place the crude product in a 750 ml round bottom flask fitted with reflux double surface condenser, add 325 ml of light petroleum ether (b.p. 60 - 80 <sup>0</sup>C), and heat in a water bath maintain at 75 - 80 <sup>0</sup>C. Shake the flask frequently and after about 15 min, slowly decand the clear hot solution into a beaker set in a freezing mixture of ice and salt, and stirs constantly. The p-iodoaniline crystallizes almost immediately in almost colorless needle; filter and dry the crystal in the air. The yield of p-iodoaniline is 60 g (82%), m. p. 62-63 <sup>0</sup>C.

PMR (DMSO-D<sub>6</sub>): 3.60 (bs, 2H), 6.36 (t, 2H), 7.64 (t, 2H).

# 2.2.4. Synthesis of 3-{(E)-[(4-iodophenyl)imino]methyl}quinolin-2-ol

Dissolve 0.1 mol of 2-hydroxy-3-formylquinoline derivative in 300 ml of absolute alcohol in 500 ml round bottom flask fitted with spiral reflux condenser and calcium chloride guard tube. Add equivalent amount of aromatic amine (0.1 mol) and catalytic amount of acetic acid. Reflux the reaction mixture in water bath for about 4-8 hours (depending on the nature of quinoline derivative and aromatic amine). Cool the reaction mixture and filter the resulting solid product by using suction pump and Whatmann filter paper number 41 and recrystalized by using ethyl acetate. Dry it in hot air oven and record m.p.  $(230^{0}C)$  and yield (79%).

FTIR (vibration frequency in cm<sup>-1</sup>): 3436, 2852, 1662, 1552 cm<sup>-1</sup>.

<sup>1</sup>HNMR (CDCl<sub>3</sub>, ð in ppm): 10.9 (bs, 1H), 8.9 (d), 8.4 (d), 7.7 (t), 7.5 (m), 7.3 (m), 7.2 (m), 7.0 (m), 6.4 (t). Mass spectra- m/z = 375, 358, 248, 230.1, 219.4, 171.3, 145.4, 126, 128, 117.

# 2.2.5. Synthesis of metal complexes of Cu (II), Zn(II), Ni(II) and La(III) complexes of Schiff bases 3-{(E)-[(4-iodophenyl)imino]methyl}quinolin-2-ol.

Dissolve 1.50 mmol of Schiff base in 30 ml of DMF in 100 ml round bottom flask fitted with reflux condenser and calcium chloride guard tube. Add 0.75 mmol of corresponding metal salt (CuCl<sub>2</sub>, NiCl<sub>2</sub>, ZnCl<sub>2</sub>, LaCl<sub>3</sub>) and stir the reaction mixture, add 1.50 mmol potassium hydroxide in it. Heat the reaction mixture about 10 minutes in water bath so the color of reaction mixture change. Cool the content and poured it into 100 g crushed ice. Filter the solid separated, record yield.

# 2. LUMINESCENCE PROPERTIES:

Luminescence properties of the Schiff base of 3-formyl-2-hydroxyquinoline with various piodoaniline have been checked by using Spectrofluorophotometer model number RF5301. A Xe laser lamp was used for excitation and emission spectra were scanned from the range 220 nm to 750 nm. For fluorescence study of the Schiff bases, dimethylformamide is used as solvent and reference material. The excitation of the molecule is occurred due to the  $n-\pi$  \* and  $n-\pi$  \* electronic transitions.

Slit width: Excitation and emission is 10 mm; Concentration of solution is 500 ppm; Solvent used is DMF.

Quantum efficiency of metal complexes over Schiff base is calculated by using following equation-

# Quantum efficiency = Intensity of emission/Intensity of excitation.

Quantum efficiency of ligand is very low whereas is higher or increased in case of complexes which can be calculated by using above values of intensities.

# 3. **RESULT AND DISCUSSION**

All the synthesized compounds (2-5) were purified by successive recrystallization using ethanol. The purity of the synthesized compounds was checked by performing TLC. The structures of the

synthesized compounds were determined on the basis of their <sup>1</sup>HNMR data. The hydroxyl proton was observed at about 10.9 ppm (bs, exchange with  $D_2O$ ) confirming presence of phenolic –OH group. Azomethine proton (-CH=N-, IR frequency at 1662 cm<sup>-1</sup>) shows signal at 8.9 ppm. The aromatic protons of both benzene rings shows signal in the region 8.7 to 6.4 ppm. The mass spectra of Schiff base showed  $[M+1]^+$  molecular ion peak at 375 confirm the condensation of 3-formyl-2-hydroxyquinoline and 4-iodoaniline. The peak observed at 358 is due to [M - OH] fragmentation and peak at 248 is due to [M - I] fragmentation.

The excitation and emission spectra of  $3-\{(E)-[(4-iodophenyl)imino]methyl\}$ quinolin-2-ol and its Zn(II), Cu(II), Ni(II) & La (III) metal complexes are studied and is shows good result in DMF. The ligand does not shows any intense transitions but the complexes shows intense transitions. The Schiff base shows weak emission at 461 nm (weak) for the absorption wavelength 387 nm whereas its complexes shows strong emission at 462 nm (moderate) [ZnL<sub>2</sub>, at 387 nm], 478 nm (strong) [CuL<sub>2</sub>, at 387 nm], 465 nm (moderate) [NiL<sub>2</sub>, at 387 nm] and 461 nm (moderate) [LaL<sub>2</sub>, at 387 nm]. The complexes having very high quantum efficiency than the Schiff base. In complexes, the coordinated aromatic ligand may greatly enhance the luminescence by absorbing energy and transferring it to the central ion. The singlet state of ligand is transfer energy to triplet state and further to the metal ion is effective and therefore strong émission of these chelates are observed. The La(III) complex is showing strong emission in the blue light region than the other complexes.

# 4. ACKNOWLEDGMENT:

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# 5. FIGURES, TABLES AND SPECTRA:

Sample	Absorption [Wavelength	Emission at 387 nm	Quantum efficiency
Code	(intensity)]	[Wavelength (intensity)]	-
L <sub>2</sub>	316(1.065), <b>387</b> (1.189)	461(7.264)	6.11
ZnL2	312(0.429), <b>387</b> (0.459)	462(22.876)	49.84
NiL2	311(0.856), <b>387</b> (0.855)	465(13.321)	15.58
LaL2	312(0.421), <b>387</b> (0.442)	461(27.164)	61.46
CuL2	315(1.006), <b>387</b> (0.871)	478(56.342)	64.68

 Table 1: The excitation and emission wavelength with intensity:

Where L is 3-{(*E*)-[(4-iodophenyl)imino]methyl}quinolin-2-ol

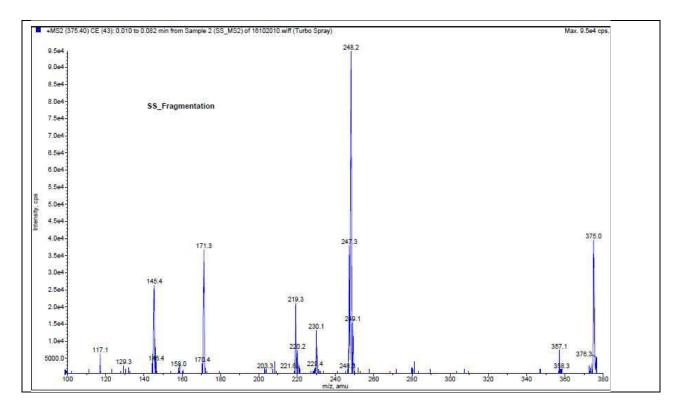


Fig 1: Mass Spectra of 3-{(*E*)-[(4-iodophenyl)imino]methyl}quinolin-2-ol

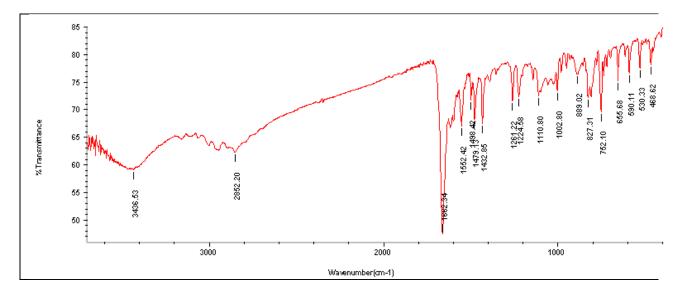


Fig 2: FTIR spectra of 3-{(*E*)-[(4-iodophenyl)imino]methyl}quinolin-2-ol

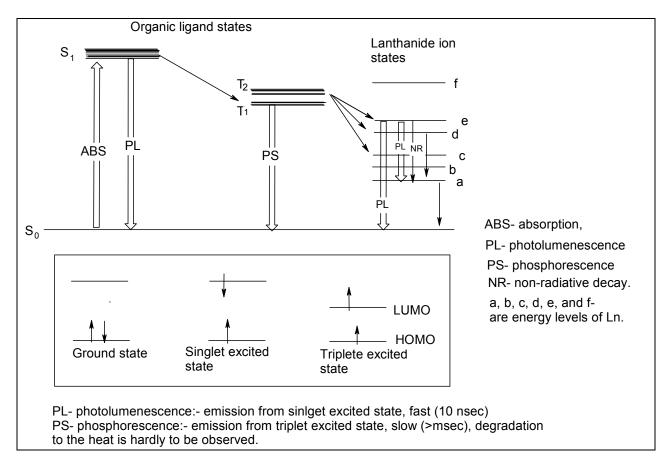


Fig 3: The mechanism of luminescence

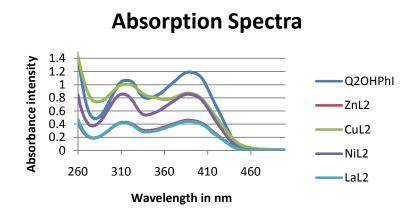


Fig: 4. Excitation/absorption spectra of  $3-\{(E)-[(4-iodophenyl)imino]methyl\}$ quinolin-2-ol and their metal complexes.

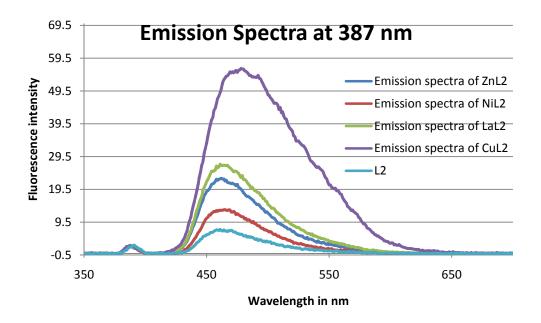


Fig: 5. Emission spectra of  $3-\{(E)-[(4-iodophenyl)imino]methyl\}$ quinolin-2-ol and their metal complexes

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