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Synthesis, Characterization and Antimicrobial Studies of 3-[(2-Hydroxy-quinolin-3-ylmethylene)-amino]-2-phenyl- 3H-quinazolin-4-one and its Metal(II) Complexes

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Abstract: A new complexes of the type ML_2 and $M'L$ [where $M=Cu(II)$, $Co(II)$, and $Ni(II)$ and $M'=Zn(II)$, $Cd(II)$ and $Hg(II)$]. $L = 3-[(2\text{-hydroxy-quinolin-3-ylmethylene)-amino]-2\text{-phenyl-3H-quinazolin-4-one}$, (HQMAPQ) Schiff base have been synthesized and characterized by elemental analysis, magnetic susceptibility, molar conductance, IR, 1H NMR, UV-Visible and ESR data. The studies indicate the HQMAPQ acts as doubly monodentate bridge for metal(II) ions and form mononuclear complexes. The complexes $Ni(II)$, $Co(II)$ and $Cu(II)$ complexes are found to be octahedral, where as $Zn(II)$, $Cd(II)$ and $Hg(II)$ complexes are four coordinated with tetrahedral geometry. The synthesized ligand and its metal(II) complexes were screened for their antimicrobial activity.

Keywords: Quinazoline, Metal(II) complexes, Antimicrobial activity, Spectral data.

Introduction

Among a wide variety of nitrogen heterocycles that have been explored for developing pharmaceutically important molecules, the quinazoline have played an important role in medicinal chemistry and subsequently have emerged as a pharmacophore¹. Recently, nitraquazone, a quinazoline derivative has been found to possess potent phosphodiesterase inhibitory activity² which is potentially useful in the treatment of asthma³. A few of the activities associated with quinazoline nucleus are hypotensive activity⁴, anticonvulsant⁵, anticoagulant⁶, anti-fibrillatory⁷, cardiac stimulant⁸, diuretic⁹, antibacterial¹⁰ and antiviral¹¹⁻¹³. Because of their biological relevance interesting to spectral and magnetic properties of ligand and its metal complexes are being used generally synthetic building blocks due to their chemical and biological relevance. The literature survey reveals that the reaction of

quinazoline hydrazide(PQH) and quinolinaldehyde(HQA) schiff base have not been reported so far. Hence it was thought to undertake such study. Therefore, it was thought worthwhile to synthesize novel quinazoline ligand (HQMAPQ) and its metal(II) complexes are characterized.

The present paper deals with the synthesis, spectral and magnatochemical studies of metal(II) complexes with the following ligand (HQMAPQ) (Fig. 1).

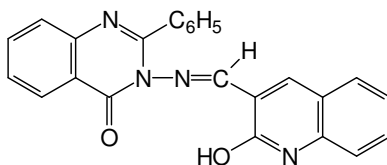


Figure 1. Structure of HQMAPQ ligand

Experimental

All the reagents used were of analytical grade and were used without further purification.

Preparation of 3-amino-2-phenylquinazoline-4-one (PQH)

A mixture of 2-phenyl-3,1-benzoxazine-4-one¹⁴ (0.1 mole) and hydrazine hydrate (0.1 mole) in ethanolic solution was refluxed for 8 hours. The excess of solvent was then distilled off, and the resulting solid was dried and recrystallised from ethanol, M.P.: 142 °C yield 72%.

Preparation of 3-[(2-Hydroxyquinoline-3-ylmethylene)-amino]-2-phenyl-3H-quinazoline-4-one (HQMAPQ)

The ligand [HQMAPA] were prepared by condensation of 3-amino-2-phenylquinazoline-4-one (PQH) (0.01 mole) and 3-formyl-2-hydroxyquinoline (HQA) (0.01 mole) in ethanol was refluxed on water bath for 6-7 h in presence of few drops of acetic acid. The reaction mixture was cooled to room temperature the separated ligand was filtered, washed and recrystallized from alcohol. The purity of the ligand (HQMAPQ) (Fig. 2) was checked by TLC.

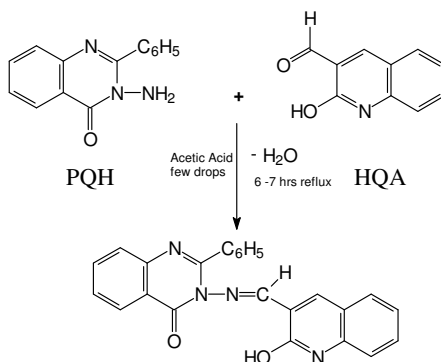


Figure 2. Synthesis of HQMAPQ

Preparation of metal(II) complexes

An ethanolic solution of ligand (HQMAPQ) (0.01 mole) was added a hot ethanolic solution of metal(II) chloride (0.01 mole) the reaction mixture was refluxed on water bath for 4 h to

get a clear solution. 0.5 g of sodium acetate was added to the reaction mixture to adjust the pH 7-8 of the solution. The reaction mixture was further refluxed for 2h until a precipitate appeared after cooling, the solid was filtered off, washed with water and then little hot ethanol apparent dryness and dried in vacuum over anhydrous calcium chloride in a desiccator (yield 60-75%).

Analysis

The complexes were analyzed for their metal and chloride contents were determined by standard methods¹⁵.

Physical Measurements

Infrared spectra of the ligand and its metal(II) complexes in KBr pellets were recorded in the spectral range 4000-350 cm^{-1} range with Perkin Elmer Spectrum one FT-IR spectrometer. UV-Visible spectra were recorded on an Elico SL-164 DOUBLE BEAM UV-Vis Spectrophotometer in the range of 200-1200 nm. Magnetic susceptibilities were measured on a Guoy Balance at a room temperature using $\text{HgCo}(\text{NCS})_4$ as calibrant. The molar conductance of the complexes were measured on ELICO CM-82 conductivity bridge in DMF solution at conc. $\sim 10^{-3}$ M. ^1H NMR spectra were recorded on AMX-400 NMR spectrometer, using TMS as internal standard and DMSO as a solvent. The elemental analysis (CHN) and ESR spectra recorded on STIC Cochin.

Results and Discussion

All the complexes were sparingly soluble in common organic solvents but soluble in DMF, DMSO and acetonitrile. The analytical data indicates that the complexes are agree well with 1:1 metal to ligand stoichiometry for Zn(II), Cd(II) and Hg(II) and 1:2 for Cu(II), Co(II) and Ni(II) complexes shown in (Table 1). The observed molar conductance (Table 1) values measured in DMF solution fall in the range ($13\text{-}20 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$). These observed values of the molar conductance are well within the expected range for non-electrolytes¹⁶.

Magnetic Susceptibility

The magnetic moments obtained at room temperature for the complexes of Cu(II), Ni(II) and Co(II) are listed (Table 1). Cu(II) complex exhibits magnetic moment 1.95 B.M. which is less than the normal value¹⁷ (1.84-2.20 B.M.). The lowered magnetic moment value observed for Cu(II) complex under present study is due to distorted octahedral geometry¹⁸. The Co(II) complex shows magnetic moment of 4.86 B.M. the spin free octahedral complex of Co(II) are reported to exhibit magnetic moment in the range of 4.46- 5.53 B.M.¹⁹. Hence observed magnetic moment for the Co(II) complex under study indicates it has an octahedral configuration. The Ni(II) complex shows magnetic moment of 2.95 B.M. The magnetic moment of octahedral Ni(II) complex are reported to exhibit magnetic moment in the range of 2.80 – 3.40 B.M.²⁰ including spin orbital coupling contribution from $^3\text{A}_{2g}$ and higher $^3\text{T}_{2g}$ states. Hence the observed magnetic moment for the Ni(II) complex suggest that it may have octahedral geometry.

Electronic spectra

The electronic spectral data of Cu(II), Co(II) and Ni(II) complexes were recorded in DMF as shown in Table 3. They have been studied with the view to obtain more information on stereochemistry of the complexes and to procure more support for the conclusion, deduced with the help of magnetic data. The light green colored Cu(II) complex exhibits a broad

asymmetric band in the region 16395-12195 cm^{-1} with maxima at 12210 cm^{-1} in an distorted octahedral geometry²¹. The broadness of the band may be due to dynamic Jahn-Teller distortion and is assigned to ${}^2\text{T}_{2g} \rightarrow {}^2\text{E}_g$ transitions.

The Co(II) complex (light purple) of the electronic absorption bands appears at 9992 and 20085 cm^{-1} , due to ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{v}_1)$ and ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})(\text{v}_3)$ transition, respectively, in an octahedral environment^{22,23}. The bands due to the ${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})(\text{v}_2)$ transition could not be observed because of its very low intensity. However the position of the v_2 band has been computed (15439 cm^{-1}) by the equation. $\text{v}_2 = \text{v}_{1+} - 10\text{Dq}$. The intense band around 30000 cm^{-1} may be a charge transfer band. The ligand field parameter such as Dq, B', β and $\beta\%$ have been calculated by using band-fitting equation given by Underhill and Billing²⁴. The crystal field splitting energy (Dq) value at 825 cm^{-1} . These values are well within the range reported are most of the octahedral Co(II) complexes. The Co(II) complex under present investigation process interelectronic repulsion parameter (B') 930 cm^{-1} . The Racha parameter (B) is less than free ion value (971) suggesting a considerable orbital overlap and delocalization of electrons on the metal ion. The nephelauxetic ratio (β) for the present Co(II) complex (0.95). This is less than one, suggesting partial covalency in the metal ligand bond. The values Dq, $\beta\%$ LFSE and v_2 / v_1 (Table 2) suggest the octahedral geometry for Co(II) complex²⁵. The electronic spectrum of Ni(II) complex shows two bands at 15151 and 25316 cm^{-1} assignable to ${}^3\text{A}_{2g} \rightarrow {}^3\text{A}_{1g}(\text{F})(\text{v}_2)$ and ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{1g}(\text{P})(\text{v}_3)$ transitions respectively, in an octahedral environment²⁶. The lowest band v_2 (10 Dq) was not observed due to limited range of the instrument used. However, it is calculated by using equation suggested by Billing and Underhill. Racha parameter B' is less than the free ion value of 1040 cm^{-1} indicating the covalent character of the complex. The ratio v_2/v_1 and $\beta\%$ are further support the octahedral geometry around the Ni(II) ion²⁷.

Infrared Spectra

The significant IR bands for the ligand (HQMAPQ) as well as for its metal (II) complexes and their tentative assignments are compiled and represented in Table 2. The broad band observed at 3435 cm^{-1} in the IR spectra of the ligand (HQMAPQ) assigned to $\nu(\text{OH})$, which were found to have disappeared in all their respective complexes, thereby indicating the involvement of phenolic oxygen is bonding with metal ions through deprotonation²⁸. A strong sharp band observed at 1659 cm^{-1} is assigned to quinazoline ring $\nu(\text{C}=\text{O})$, which was shifted to 33-57 cm^{-1} in all complexes²⁹, indicates the involvement of quinazoline ring carboxyl in complexation with metal ion³⁰, the band at 1600 cm^{-1} is assigned to the azomethine $\nu(\text{C}=\text{N})$ group³¹, lowering of $\nu(\text{C}=\text{N})$ 24-43 cm^{-1} in the complexes as compared to its ligand is due to reduction of double bond character carbon-nitrogen bond of the azomethine group³².

The band observed at 1273 cm^{-1} , of the ligand is attributed to phenolic $\nu(\text{C}-\text{O})$ in view of previous observations²¹. This band is shifted to higher frequency and is found in the region 1301-1316 cm^{-1} for the complexes. Thus, this further confirms the involvement of phenolic OH in the complex formation. The low frequency skeletal vibrations due to M-O and M-N stretching provide direct evidence for complexation. In the present investigation, the bands in the 534-525 cm^{-1} and 445-425 cm^{-1} region for $\nu_{\text{M}-\text{O}}$ and $\nu_{\text{M}-\text{N}}$ vibration respectively³³. The bands due to $\nu_{(\text{M}-\text{Cl})}$ were observed in the 364-352 cm^{-1} region and are characteristic of chlorine atom in Zn(II), Cd(II) and Hg(II) complexes is further confirmed by quantitative chloride estimation.

¹H NMR spectra

Spectrum of ¹H NMR in DMSO-d₆ solvent used. In ligand (HQMAPQ) showed sharp peak at δ 12.2 (S, 1H) due to OH at 2-position of phenyl ring of quinoline moiety has resonated, but in the case of Cd(II) complex which has been disappeared indicating the involvement of phenolic oxygen in the coordination *via* deprotonation³⁴. The peak appears at δ 8.8 (S, 1H, -CH=N) due to the azomethine group in ligand but in case of Cd(II) complex the peak observed at 8.6 (S, 1H, -CH=N). The fourteen aromatic protons due to quinazoline and phenyl rings have resonated in region δ 7.0-8.6 (m, 14H, Ar-H) as a multiplet in Cd(II) complex the fourteen aromatic protons have been observed in the region δ 6.92-8.42 (m, 14H, Ar-H) as multiplet. The Cd(II) complex suggest coordination of the phenolic OH with metal ion³⁵.

ESR Spectrum

ESR spectrum of Cu(II) complexes of ligand (HQMAPQ) scanned at room temperature (Table 4) using DPPH as a standard showed a broadened feature without hyperfine splitting due to the dipolar interaction from the ESR spectrum of a set of magnetic parameter g_{\parallel} 2.053 and g_{\perp} 2.0276. The observed ESR spectrum is characteristic of distorted octahedral geometry g value averaged to overall directions and G which is measure of extent of exchange interaction between metal ion have been calculated. In present case the value of G was found to be 4.028 according to Hathway³⁶. If G value is greater than 4, the spin exchange interaction is negligible where as G value less than 4 indicate considerable interaction between metal ions in solid complex clearly indicate that Cu(II) ion in the complex is mono-nuclear nature of the complex.

Antimicrobial Activity

Antimicrobial activity was carried out using the cup-plate method³⁷. The antimicrobial activity results of the screened compounds are given in the Table 5. The ligand (HQMAPQ) and its Cu(II), Co(II), Ni(II), Zn(II), Cd(II) and Hg(II) complexes have been tested for their antibacterial activity³⁸, against *E. coli* and *S. aureus* and antifungal activity³⁹ against *A. niger* and *A. flavous* at 100 $\mu\text{g}/0.1 \text{ cm}^3$ concentration. The standard drugs streptomycin and chlotrimazole were also tested for their antibacterial and antifungal activity at the same concentration under the conditions similar to that of the test compounds. The antibacterial activity results revealed that the ligand and its complexes shown weak to good activity. The ligand and its Cu(II) and Ni(II) complexes shows weakly active with the zone of inhibition 11-13 mm against the both organisms when compared to the standard drug streptomycin. The Co(II) and Cd(II) complexes shows active and moderate activity as compared to its ligand with zone of inhibition 14-16 and 17-18 mm respectively. The Zn(II) and Hg(II) complexes have exhibited good activity with the zone of inhibition 19 to 20 mm when compared to the standard drug streptomycin.

The antifungal activity results revealed that the ligand and its Cu(II), Co(II), Ni(II), Zn(II), Cd(II) and Hg(II) complexes have exhibited weak to good activity. The ligand and its Cu(II) and Co(II) complexes shows weak activity when compared to the standard drug chlotrimazole. The Ni(II) and Cd(II) complexes shows active and moderate activity as compared to its ligand with the zone of inhibition 15-16 and 17-18 mm respectively.

The Zn(II) and Hg(II) complexes have exhibited good activity with the zone of inhibition 19-21 mm when compared to the standard drug chlotrimazole.

Table 1 Analytical, magnetic susceptibility and molar conductance data of the ligand [HQMAPQ] and its metal (II) complexes

Ligand / Complex	Mol. Wt.	M.P. (°C)	Yield (%)	Found / (Calculated) %					μ_{eff} B.M.	Molar Conductance $\Lambda_m^{\text{cm}^2 \text{mol}^{-1}}$
				C	H	N	M	Cl		
C ₂₄ H ₁₆ N ₄ O ₂	392.41	245	80	73.21 (73.46)	4.15 (4.11)	14.12 (14.28)	-	-	-	-
[Cu(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	846.35	282	70	68.02 (68.12)	3.41 (3.57)	13.42 (13.27)	7.41 (7.51)	-	1.95	15.15
[Co(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	841.74	286	65	68.26 (68.49)	3.42 (3.59)	13.15 (13.31)	7.21 (7.00)	-	4.86	18.38
[Ni(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	841.50	291	68	68.34 (68.51)	3.72 (3.59)	13.65 (13.32)	6.69 (6.97)	-	2.95	19.49
Zn(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	492.24	284	60	58.52 (58.56)	3.02 (3.07)	11.25 (11.38)	13.25 (13.28)	7.15 (7.20)	Diamag	13.13
Cd(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	539.27	292	65	53.41 (53.45)	2.78 (2.80)	10.35 (10.39)	20.80 (20.85)	6.52 (6.57)	Diamag	16.26
Hg(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	627.44	299	75	45.90 (45.94)	2.38 (2.41)	8.91 (8.93)	31.93 (31.97)	5.62 (5.65)	Diamag	-

Table 2. Electronic spectral data and ligand field parameters of Cu(II), Co(II) and Ni(II) quinazoline complexes

Complexes	ν_1	ν_2	ν_3	Dq	B ¹	β	$\beta\%$	ν_2/ν_1	ν_3/ν_2	LFSE
[Cu(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	-	12195	16395	1429	-	-	-	-	-	24.50
[Co(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	9992	15439	20085	825	930	0.958	15.03	1.54	1.30	14.17
[Ni(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	10810	15151	25316	932	834	0.802	19.80	1.4015	1.6709	31.954

Table 3. Important spectral IR bands of the ligand [HQMAPQ] and its metal (II) complexes

Ligand/ complexes	ν_{OH}	$\nu_{\text{C=O}}$	$\nu_{\text{C=N}}$	$\nu_{\text{C-O}}$	$\nu_{\text{M-O}}$	$\nu_{\text{M-N}}$	$\nu_{\text{M-Cl}}$
C ₂₄ H ₁₆ N ₄ O ₂	3435	1659	1600	1273	-	-	-
[Cu(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	-	1608	1569	1302	530	427	-
[Co(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	-	1614	1568	1301	534	428	-
[Ni(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	-	1613	1576	1316	530	425	-
[Zn(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	-	1626	1567	1315	530	425	358
[Cd(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	-	1621	1557	1312	531	445	364
[Hg(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	-	1602	1567	1301	525	435	352

Table 4. ESR data of the Cu(II) complex of the ligand [HQMAPQ]

Complexes	g_{\perp}	g_{\parallel}	g_{av}	G
[Cu(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	2.0276	2.0532	2.0360	4.0280

Table 5. Antimicrobial activity of the ligand [HQMAPQ] and its metal(II) complexes

Compound	Antibacterial Activity of zone of inhibition, mm		Antifungal Activity of zone of inhibition, mm	
	<i>E. coli</i>	<i>S. aureus</i>	<i>A. niger</i>	<i>A. falvours</i>
C ₂₄ H ₁₆ N ₄ O ₂	11	12	12	11
[Cu(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	11	13	13	12
[Co(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	14	16	12	14
[Ni(C ₂₄ H ₁₅ N ₄ O ₂) ₂]	13	12	15	16
[Zn(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	19	20	20	19
[Cd(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	18	17	18	17
[Hg(C ₂₄ H ₁₅ N ₄ O ₂)Cl]	20	19	21	20
Streptomycin	24	21	-	-
Chlorotrimazole	-	-	24	23
DMF (Control)	0	0	0	0
Bore size	08	08	08	08

Conclusion

The elemental analysis, magnetic susceptibility, electronic, IR, ¹H NMR and ESR spectral observations projects the following structures (Fig 3 and Fig 4) for these complexes where in Cu(II), Co(II) and Ni(II) exhibit coordination number of six, Zn(II), Cd(II) and Hg(II) are four coordinated tetrahedral geometry.

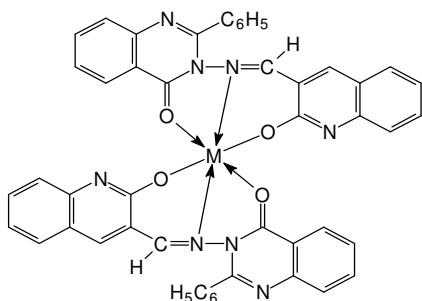


Figure 3. Structure of complex.
M = Cu(II), Co(II) and Ni(II)

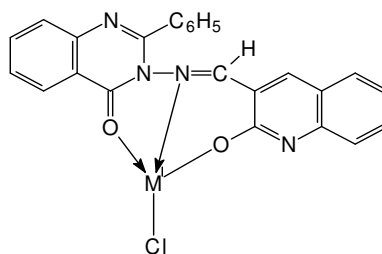


Figure 4. Structure of complex.
M' = Zn(II), Cd(II) and Hg(II)

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