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**Research Article**

**Synthesis and Spectral Studies of Complexes of Co(II),  
Ni(II), Cu(II), Zn(II), Ru(II), Pd(II) & Pt(II) with 2,3-  
Disubstituted Quinazolin-3(H)-4-ones**

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**ABSTRACT**

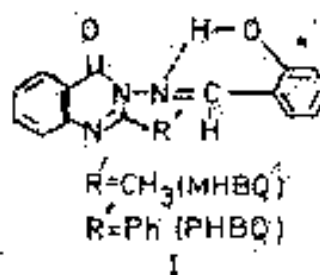
Complexes of Co(II), Ni(II), Cu(II), Zn(II), Ru(II), Pd(II) & Pt(II) with 2-methyl-3-(2'-hydroxybenzylamino)-quinazolin-(3H)-4-one (MHBO) and 2-phenyl-3-(2'-hydroxybenzylamino)-quinazolin-(3H)-4-one (PHBO) have been synthesized and characterized by analytical, conductivity, magnetic and infrared, electronic, PMR and ESR spectral data. Based on analytical data, the stoichiometry of the complexes is found to be 1:2 (metal to ligand). The infrared and PMR spectral data of the metal complexes indicate that MHBO and PHBO act as uninegative tridentate ligands towards Co(II), Ni(II), Cu(II) and Ru(II) and as uninegative bidentate ligands towards Pd(II), Pt(II) and Zn(II). The electronic spectral data suggest that all the Co(II), Ni(II) and Ru(II) complexes are octahedral, Cu(II) complexes are tetragonal, Pd(II) and Pt(II) complexes are square-planar and Zn(II) complexes are tetrahedral.

**Keywords:** Magnetic and Spectral characterisation, chelates, 2,3-Disubstituted Quinazolin- 3(H)-4-ones.

**INTRODUCTION**

Quinazolin-(3H)-4-ones possess four coordinating sites: (i) oxygen of the phenolic –OH, (ii) oxygen of the carbonyl function, (iii) nitrogen of the benzylamino group and (iv) nitrogen of the quinazolin ring (structure-I). These ligands may, therefore, act as monodentate, bidentate or tridentate ligands<sup>1,2</sup>. In order to find out the nature of interaction of these ligands with different metal ions and in continuation of our earlier work<sup>3-5</sup> on complexes of 2,3-disubstituted quinazolin-(3H)-4-ones, we report here the synthesis of Co(II), Ni(II), Cu(II), Zn(II), Ru(II), Pd(II) and Pt(II) complexes of 2-methyl-3-(2'-hydroxybenzylamino) quinazolin-(3H)-4-one (MHBO) and 2-phenyl-3-(2'-hydroxybenzylamino)-quinazolin-(3H)-4-one (PHBO). The complexes have been characterized on the basis of analytical data, conductivity measurements, magnetic susceptibility measurements

and infrared, electronic, electron spin resonance (ESR) and proton magnetic resonance spectra (PMR).



**MATERIALS AND METHODS**

Anthranilic acid, hydrazine hydrate, salicylaldehyde, acetic anhydride and other solvents used in this work were BDH reagents of AR grade. The metal salts like nickel(II) chloride, cobalt(II) acetate and zinc(II) acetate were the products of M/s. Chempure

Chemicals Limited, Bombay. Palladium(II) chloride, platinum(II) chloride and ruthenium (II) chloride were obtained from Johnson Mathey Chemicals, London. The ligands MHBQ and PHBQ were prepared as reported in literature<sup>6</sup> and their purities checked by TLC and melting point determinations. The complex  $\text{RuCl}_2(\text{DMSO})_4$  was prepared by literature method<sup>7</sup>.

#### Preparation of the complexes

In the preparation of all the Co(II), Ni(II), Cu(II) and Zn(II) complexes the following general procedure was adopted. The aqueous metal salt solution (0.1 M) was added drop-wise to a solution (0.3 M) of the ligand in acetone with constant stirring. In all the cases, the ligand concentration was kept in slight excess over that required by 1:3 (metal-ligand) molar ratio. The reaction mixture was refluxed on a water bath for 60-90 min. The complex separated out on cooling. It was filtered off and washed several times with hot acetone until the washings were free from excess ligand. The complexes were finally dried *in vacuo* over fused calcium chloride.

#### Ruthenium(II) complexes

The complex, dichlorotetrakis(dimethyl sulphoxide) ruthenium(II)  $[\text{RuCl}_2(\text{DMSO})_4]$  is a convenient starting material for the preparation of a variety of mixed ligand ruthenium(II) complexes. It was prepared using the method of Wilkinson *et al.*<sup>7</sup>. The complex,  $[\text{RuCl}_2(\text{DMSO})_4]$  (0.2 g), was suspended in toluene (30 ml) and refluxed for one hour with the appropriate ligand in acetone (30 ml). In all the cases, 1:4 (metal to ligand) ratio was maintained. During the period of refluxing, the whole suspension dissolved, resulting in a clear solution which was then evaporated under reduced pressure to remove the excess of solvent. The residue was treated with diethyl ether when the solid complex precipitated. This was filtered off, washed several times with diethyl ether, dried and recrystallized from acetone-ether to get the pure complex. Finally the complexes were dried *in vacuo* over fused calcium chloride.

#### Palladium(II) and platinum(II) complexes

To Pd(II) / Pt(II) salt solution, was added the ligand in acetone (50 ml) drop by drop with constant stirring. In all the cases the ligand concentration was kept in slight excess over that required by 1:3 (metal to ligand) molar ratio. The reaction mixture was refluxed on a water bath for about 40 min. The complex separated out in neutral medium on cooling. It was filtered and washed several times with hot water and acetone until the washings were free from the excess ligand and chloride ions. The complexes were dried *in vacuo* over fused calcium chloride.

The analytical data (C, H, N) for the ligands and their metal complexes were obtained from the Micro-analytical Laboratory, Calcutta University, Calcutta. The metal contents of the complexes were determined using standard procedures after heating these to decomposition. Molar conductivities of the complexes in DMF were measured using a Digisun digital conductivity meter, model DI-909. Magnetic susceptibilities were measured at room temperature by the Gouy method using  $\text{Hg}[\text{Co}(\text{SCN})_4]$  as the calibrant. Diamagnetic corrections were applied using Pascal's constants. Infrared spectra of the ligand and the complexes in the region  $4000\text{-}200\text{ cm}^{-1}$  were recorded in nujol on a Perkin Elmer infrared spectrophotometer model-283. Electronic spectra of the complexes were recorded in DMF on a Shimadzu multipurpose recording spectrophotometer model-MPS-5000. The solid state ESR spectra of Cu(II) complexes at liquid nitrogen temperature were recorded on a Varian E-4, X-band instrument at RSIC, IIT, Bombay. 100 MHz PMR spectra of the ligands and their Ru(II), Pd(II), Pt(II) and Zn(II) complexes were recorded in  $\text{DMSO-}d_6$  on a Jeol instrument at I.I.Sc., Bangalore.

#### RESULTS AND DISCUSSION

All the complexes are stable at room temperature, non-hygroscopic, insoluble in water and some common organic solvents, but are soluble in DMF and DMSO. The analytical data of the complexes (Table 1) indicate that the metal to ligand ratio is 1:2. The molar conductances of  $1 \times 10^{-3}\text{ M}$  solutions of the complexes in DMF are in the range  $5\text{-}20\text{ ohm}^{-1}\text{ cm}^2\text{ mol}^{-1}$  indicating their non-ionic nature<sup>8</sup>. 2-Substituted-3-aminoquinazolin-(3H)-4-ones, which are structurally similar to the present ligands, are reported to coordinate in a bidentate manner through oxygen of the carbonyl group and nitrogen of the amino group<sup>9</sup>. In the IR spectra of the present ligands, a strong band appearing around  $1680\text{ cm}^{-1}$ , attributed to  $\nu(\text{C}=\text{O})$ , is shifted to lower wave numbers by  $35\text{-}50\text{ cm}^{-1}$  in the spectra of all the complexes except in the complexes of Zn(II), Pd(II) and Pt(II), indicating that the carbonyl oxygen is involved in coordination<sup>10</sup>. However, the complexes of Zn(II), Pd(II), Pt(II) exhibit a band at  $1680\text{ cm}^{-1}$ , indicating the non-involvement of carbonyl oxygen in complex formation. The appearance of  $\nu(\text{-OH})$  in the lower region ( $3200\text{-}3100\text{ cm}^{-1}$ ) than expected ( $3600\text{-}3400\text{ cm}^{-1}$ ) in the spectra of both the ligands can be attributed to the hydrogen bonding of OH with nitrogen of the benzylamino group<sup>11</sup>. The disappearance of this band in all the metal complexes indicates complex formation by deprotonation of OH group.

The ligands MHBQ and PHBQ have two C = N groups, one belonging to quinazolin ring and the other to benzylamino group. The IR absorptions at 1640 and 1610  $\text{cm}^{-1}$  are assigned to  $\nu(\text{C}=\text{N})$  of quinazolin ring and benzylamino group, respectively<sup>12</sup>. The band at 1640  $\text{cm}^{-1}$  remains unchanged in the spectra of the metal complexes, but the one at 1610  $\text{cm}^{-1}$  undergoes shift to lower wave numbers ( $\Delta\nu$  40-50  $\text{cm}^{-1}$ ) indicating that the nitrogen of benzylamino group is involved in coordination with the metal ion<sup>12</sup>. The absence of a  $\nu(\text{S}=\text{O})$  band in the region 1050-1100  $\text{cm}^{-1}$  in all Ru(II) complexes indicates that DMSO is not coordinated with the metal ion<sup>13</sup>. The participation of oxygen and nitrogen in coordination in all the complexes is further supported by the appearance of  $\nu(\text{M}-\text{O})$  and  $\nu(\text{M}-\text{N})$  at 400 and 500  $\text{cm}^{-1}$ , respectively, in the far infrared region<sup>14,15</sup> (Table 2).

#### Electronic spectra

The electronic spectral data of the complexes along with their assignments are presented in Table 3. All the ligands exhibit strong bands around 3300  $\text{cm}^{-1}$  with a shoulder at 29000  $\text{cm}^{-1}$ , assignable to  $\pi^* \leftarrow \pi$  and  $\pi^* \leftarrow n$  transitions, respectively. Electronic spectra of Co(II) complexes display three bands around 8000, 16000 and 19500  $\text{cm}^{-1}$  which are characteristic of octahedral geometry around Co(II)<sup>5</sup>. Electronic spectra of Ni(II) complexes exhibit three bands around 9000, 15000 and 24000  $\text{cm}^{-1}$  which are characteristic of octahedral geometry around Ni(II)<sup>16</sup>. The octahedral geometry of Co(II) and Ni(II) complexes is further supported by the value of  $\nu_2/\nu_1$  ratio which lies around 2.0 and 1.60, respectively<sup>16</sup>. Various ligand field parameters, such as the ligand field splitting energy (10  $Dq$ ), Racah inter-electronic repulsion parameter (B), covalency factor ( $\beta$ ) and ligand field stabilization energy (LFSE) have been calculated for all the Co(II) and Ni(II) complexes<sup>17</sup>. The calculated 10  $Dq$  values of Co(II) and Ni(II) complexes suggest for these ligands a place between water and ammonia in the spectrochemical series. The B-values for the complexes are lower than the free ion value which is an indication of orbital overlap and delocalization of d-orbitals. The  $\beta$ -values obtained are less than unity suggesting considerable amount of covalent character of the metal-ligand bonds. The  $\beta$  values for Co(II) complexes are less than those for the Ni(II) complexes indicating the greater covalent nature of Co(II) complexes. The electronic spectra of Cu(II) complexes exhibit one broad band around 16000  $\text{cm}^{-1}$ . This is characteristic of tetragonal geometry around Cu(II)<sup>18</sup>. The two spin-allowed  $d-d$  transitions are observed in

the Ru(II) complexes at 19200 and 24500  $\text{cm}^{-1}$  which are characteristic of octahedral geometry<sup>19</sup>. The absence of splitting of lower energy band in the electronic spectra of Ru(II) complexes indicates that there is no tetragonal distortion. The bands observed for the Pd(II) complexes at 15000 and 20000  $\text{cm}^{-1}$  indicate for them square-planar geometry<sup>20,21</sup>. Electronic spectra of Pt(II) complexes show two bands around 20000 and 24000  $\text{cm}^{-1}$  which are characteristic of square-planar geometry<sup>22</sup>.

#### Magnetic moments

The experimental and calculated magnetic moments of the complexes are given in Table 3.

The values obtained for all the complexes of Co(II) and Ni(II) are in the respective expected ranges for octahedral geometry<sup>23,24</sup>. The Cu(II) complexes possess magnetic moment value (1.82 B.M.) corresponding to one unpaired electron. All the Ru(II), Pd(II), Pt(II) and Zn(II) complexes are diamagnetic in nature.

#### PMR spectra

The aromatic protons in the PMR spectra of ligands appear in the region<sup>25</sup>  $\delta$  7.0-8.5 ppm. The methyl protons of MHBQ appear at  $\delta$  2.8 ppm. The signal due to the proton present on benzylamine carbon atom is overlapped by the signals of aromatic protons. In complexes, this signal is shifted to  $\delta$  9.6 indicating a decrease in the electron density at benzylamine carbon atom, which also supports the participation of nitrogen of benzyl-amino group in coordination<sup>26</sup>. A signal present at  $\delta$  10.5 in both the ligands indicates that hydroxyl proton is hydrogen bonded. The disappearance of this signal in the complexes confirms complex formation after deprotonation.

#### ESR spectra

The  $g_{\parallel}$  and  $g_{\perp}$  values have been found to be 2.24 and 2.06 respectively for Cu(II) complex of MHBQ by Kneubuhl's method<sup>27</sup> from which  $|g|$  is calculated. The trend  $g_{\parallel} > g_{\perp}$  is characteristic of tetragonal geometry with  $d_{x^2-y^2}$  as the ground state. The axial symmetry parameter (G) for the complexes is found to be greater than 4. This shows the absence of interaction between copper centers in the solid state<sup>28</sup>. The ESR spectrum of Cu(II) – MHBQ complex is magnetically dilute in the solid state as evidenced by the presence of four peaks in the  $g_{\parallel}$  region. The  $g_{\parallel}$  value is 2.24 and  $A_{\parallel}$  value is 195 G. Further, the  $g_{\perp}$  region is split with  $g_y = 2.07$  and  $g_x = 2.05$  suggesting a rhombic structure.

Table 1: Analytical and Physical Data of the Complexes

Complex	Colour	m.p. (°C) <sup>a</sup>	Analysis (%) <sup>b</sup>			
			M	C	N	H
[Co(MHBQ) <sub>2</sub> ]	Brown	290	9.43 (9.09)	62.54 (62.03)	13.68 (13.30)	3.90 (3.10)
[Co(PHBQ) <sub>2</sub> ]	Brown	286	7.83 (7.21)	68.29 (67.92)	11.38 (11.08)	3.79 (3.12)
[Ni(MHBQ) <sub>2</sub> ]	Brown	295	9.44 (9.07)	62.54 (62.25)	13.68 (13.06)	3.90 (3.10)
[Ni(PHBQ) <sub>2</sub> ]	Dark Brown	295	7.85 (7.23)	68.29 (67.63)	11.38 (11.03)	3.79 (3.02)
[Cu(MHBQ) <sub>2</sub> ]	Brown	300	10.17 (9.90)	62.03 (61.86)	13.57 (13.12)	3.87 (3.22)
[Cu(PHBQ) <sub>2</sub> ]	Green	310	8.47 (8.09)	67.83 (67.02)	11.30 (10.89)	3.77 (3.12)
[Zn(MHBQ) <sub>2</sub> ]	Colourless	280	10.46 (10.03)	61.83 (61.02)	13.52 (13.0)	3.86 (3.20)
[Zn(PHBQ) <sub>2</sub> ]	Colourless	278	8.72 (8.23)	67.65 (67.09)	11.27 (10.82)	3.76 (3.19)
[Ru(MHBQ) <sub>2</sub> ]	Green	285	-	58.44 (58.08)	12.78 (12.43)	3.65 (3.22)
[Ru(PHBQ) <sub>2</sub> ]	Snuff	290	-	64.53 (64.02)	10.75 (10.20)	3.58 (3.07)
[Pd(MHBQ) <sub>2</sub> ]	Dark Yellow	270	-	58.00 (57.18)	12.68 (11.17)	3.62 (3.05)
[Pd(PHBQ) <sub>2</sub> ]	Snuff	272	-	64.12 (63.65)	10.68 (9.98)	3.56 (3.20)
[Pt(MHBQ) <sub>2</sub> ]	Snuff	270	-	51.13 (50.92)	11.18 (10.96)	3.19 (3.00)
[Pt(PHBQ) <sub>2</sub> ]	Snuff	275	-	57.6 (56.55)	9.60 (9.01)	3.27 (3.00)

<sup>a</sup> All the complexes decomposes above the temperature cited<sup>b</sup> Calculated values in parenthesis.Table 2: Some IR Spectral Bands (cm<sup>-1</sup>) of 2,3-Disubstituted Quinazolin-(3H)-4-ones and Their Complexes

Compound	$\nu(\text{C=O})$	$\nu(\text{C=N})$	$\nu(\text{OH})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$
MHBQ	1680	1610	3150	-	-
PHBQ	1670	1610	3200	-	-
[Co(MHBQ) <sub>2</sub> ]	1630	1560	-	400, 430	480
[Co(PHBQ) <sub>2</sub> ]	1640	1560	-	410, 430	480
[Ni(MHBQ) <sub>2</sub> ]	1620	1570	-	420, 450	500
[Ni(PHBQ) <sub>2</sub> ]	1640	1560	-	410, 430	490
[Cu(MHBQ) <sub>2</sub> ]	1640	1570	-	400, 450	500
[Cu(PHBQ) <sub>2</sub> ]	1640	1570	-	410, 440	480
[Zn(MHBQ) <sub>2</sub> ]	1640	1560	-	400, 430	-
[Zn(PHBQ) <sub>2</sub> ]	1620	1550	-	400, 420	-
[Ru(MHBQ) <sub>2</sub> ]	1630	1575	-	400, 450	500
[Ru(PHBQ) <sub>2</sub> ]	1620	1570	-	390, 420	490
[Pd(MHBQ) <sub>2</sub> ]	1630	1565	-	380, 430	-
[Pd(PHBQ) <sub>2</sub> ]	1620	1570	-	410, 440	-
[Pt(MHBQ) <sub>2</sub> ]	1630	1580	-	400, 450	-
[Pt(PHBQ) <sub>2</sub> ]	1630	1570	-	400, 450	-

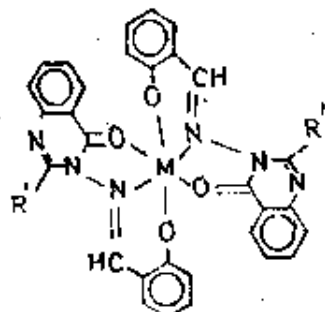
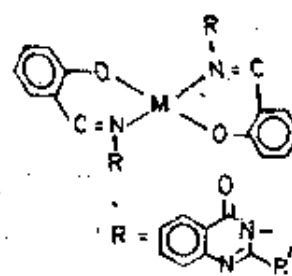
**Table 3: Magnetic Moment and Electronic Spectral Data and Relevant Ligand Field Parameters of Complexes**

Complex	$\mu_{\text{eff}}$ (B.M.)	$\nu_1$ ( $\text{cm}^{-1}$ )	$\nu_2$ ( $\text{cm}^{-1}$ )	$\nu_3$ ( $\text{cm}^{-1}$ )	$\nu_2/\nu_1$	$10 Dq$ ( $\text{cm}^{-1}$ )	B ( $\text{cm}^{-1}$ )	$\beta$	LFSE (kcal $\text{mol}^{-1}$ )
[Co(MHBQ) <sub>2</sub> ]	4.99 (5.01)	8000	16000	19500	2.00	8000	766.7	0.68	18.29
[Co(PHBQ) <sub>2</sub> ]	5.03 (5.01) <sup>b</sup>	8200	16100	19600	1.96	7900	740.0	0.65	18.05
[Ni(MHBQ) <sub>2</sub> ]	3.20 (3.15)	9050	15100	24000	1.67	6050	796.7	0.76	20.76
[Ni(PHBQ) <sub>2</sub> ]	3.28 (3.16)	9000	15000	24200	1.67	6000	813.3	0.78	20.57
[Cu(MHBQ) <sub>2</sub> ]	1.82 (1.81)	-	16000 <sup>a</sup>	-	-	-	-	-	-
[Cu(PHBQ) <sub>2</sub> ]	1.83 (1.82)	-	16000	-	-	-	-	-	-
[Ru(MHBQ) <sub>2</sub> ]	-	-	19200	24500	-	-	-	-	-
[Ru(PHBQ) <sub>2</sub> ]	-	-	19100	24450	-	-	-	-	-
[Pd(MHBQ) <sub>2</sub> ]	-	-	15000	20000	-	-	-	-	-
[Pd(PHBQ) <sub>2</sub> ]	-	-	15200	20000	-	-	-	-	-
[Pt(MHBQ) <sub>2</sub> ]	-	-	20000	24000	-	-	-	-	-
[Pt(PHBQ) <sub>2</sub> ]	-	-	20100	24050	-	-	-	-	-

<sup>a</sup> Borad band indicates the overlapping of different transitions<sup>b</sup> Calculated values in parenthesis.For Co(II) complexes:  $\nu_1 = {}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ ,  $\nu_2 = {}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ ,  $\nu_3 = {}^4T_{1g}(F) \rightarrow {}^4T_{2g}(P)$ For Ni(II) complexes:  $\nu_1 = {}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ ,  $\nu_2 = {}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ ,  $\nu_3 = {}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ For Cu(II) complexes:  $\nu_2 = {}^2E_g \rightarrow {}^2T_{2g}$ For Ru(II) complexes:  $\nu_2 = {}^1A_{1g} \rightarrow {}^1T_{1g}$ ,  $\nu_3 = {}^1A_{1g} \rightarrow {}^1T_{2g}$ For Pd(II) complexes:  $\nu_2 = {}^1A_{2g} \rightarrow {}^1A_{2g}$ ,  $\nu_3 = {}^1A_{1g} \rightarrow {}^1B_{1g}$ For Pt(II) complexes:  $\nu_2 = {}^1A_{1g} \rightarrow {}^1B_{1g}$ ,  $\nu_3 = {}^1A_{1g} \rightarrow {}^1E_g$ 

## CONCLUSION

In this report, the complexes have been characterized on the basis of analytical data, conductivity measurements, magnetic susceptibility measurements and infrared, electronic, electron spin resonance (ESR) and proton magnetic resonance spectra (PMR). Based on their stoichiometry, conductance, magnetic and IR, electronic, PMR and ESR spectral data, the complexes have been assigned structures IIa and IIb.

II(a) M = Co(II), Ni(II),  
Cu(II) and Ru(II)

II(b) M = Pd(II), Pt(II) and Zn(II)

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