



Synthesis and Structural Studies on Transition Metal Complexes Derived from 4-Hydroxy-4-methyl-2-pentanone-1*H*-benzimidazol-2-yl-hydrazone

M.NEELAMMA, P.VENKATESWAR RAO* and G.H.ANURADHA

Department of Chemistry, R B V R Reddy Women's College
Narayanguda, Hyderabad-500029, India

*Department of Chemistry, Nizam College, Hyderabad-500001, A.P., India
pallapothulav@yahoo.com

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Abstract: Transition metal complexes of Cr(III), Fe(III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) with a tridentate ligand, 4-hydroxy-4-methyl-2-pentanone-1*H*-benzimidazole-2-yl-hydrazone (H-HPBH) derived from the condensation of 2-hydrazinobenzimidazole and diacetone alcohol was synthesized. Characterization has been done on the basis of analytical, conductance, thermal and magnetic data, infrared, ¹H NMR, electronic, mass and ESR spectral data. From analytical and thermal data, the stoichiometry of the complexes has been found to be 1:1 (metal: ligand). Divalent complexes have the general formula [M(HPBH)Cl(H₂O)₂] in octahedral geometry, [M(HPBH)Cl] in tetrahedral and square planar stereochemistries and trivalent complexes [M(HPBH)Cl₂(H₂O)] in octahedral disposition. Infrared spectral data suggest that the ligand HPBH behaves as a monobasic tridentate ligand with N: N: O donor sequence towards the metal ions. On the basis of the above physicochemical data, octahedral, tetrahedral and square planar geometries were assigned for the complexes. The ligand and metal complexes were screened for their physiological activities against *E. coli* and *S. aureus*. The order of physiological activity has been found to be Cu(II) > Ni(II) > Zn(II) > Co(II) > Cr(III) > Mn(II) > Fe(III) > ligand against *E. coli* and Ni(II) > Cu(II) > Zn(II) > Mn(II) > Cr(III) > Fe(III) > Co(II) > ligand against *S. aureus*.

Keywords: 2-Hydrazinobenzimidazole, Diacetone alcohol, *E. coli*, *S. aureus*.

Introduction

The coordination chemistry of nitrogen-oxygen donor ligands is an interesting area of research¹. A great deal of attention in this area has been focused on the complexes formed by transition metal ions with hydrazones²⁻⁷. The remarkable biological activity of acid hydrazides, a class of Schiff bases, their corresponding aroyl hydrazones, and the dependence

of their mode of chelation with transition metal ions present in the living system has been of significant interest in the past⁸⁻¹¹. The chemistry of metal complexes with the Schiff base ligands has become a rapidly growing area of research¹². Schiff bases play an important role in inorganic chemistry as they form stable complexes with most transition metal ions¹³⁻¹⁴. The remarkable biological activity of aroyl hydrazones and their mode of chelation with transition metal ions present in the living systems have been of significant interest¹⁵⁻¹⁶. The coordination chemistry and physiological activities of benzimidazole derivatives and their metal complexes are of recent interest¹⁷⁻²². Hydrazones of benzimidazole are biologically active against different bacteria, fungi and microbes²³⁻²⁶. The hydrazones act as anticancer and antitumor agents²⁷⁻²⁸. The coordination compounds of aroyl hydrazones have been reported to act as enzyme inhibitors²⁹ and are useful due to their pharmacological applications³⁰. The ligand in the present investigation 4-hydroxy-4-methyl-2-pentanone-1H-benzimidazole-2yl-hydrazone (H-HPBH) is a potential chelating agent³¹. Besides its chelating ability it has promising antimicrobial activities³². In view of this, the work on the title compounds was undertaken and the results are reported here.

Experimental

Synthesis of ligand

Diacetone alcohol (0.01 mole, 0.116 g) was added to a solution of 2-hydrazine benzimidazole (0.01, 0.148 g) in methanol (100 mL). The mixture was heated under reflux for 3 h. The mixture was then poured into ice-cold water. The violet colored needles separated out were filtered and washed with small amount of aqueous alcohol. The compound was dried in vacuum and recrystallized from aqueous methanol.

Synthesis of metal complexes

The metal chelates were prepared by refluxing the methanol solutions of the respective metal chlorides (0.01 mole) and H-HPBH (0.01mole, 0.246 g) on a water bath for *ca* 4 h. The pH of the solution was adjusted to 7 by drop wise addition of 10% methanol- ammonia solution. Upon cooling the product, the metal chelates were separated out. The products were washed with methanol, followed by petroleum ether (60-80 °C) and dried in vacuum.

Materials and methods

All the metal salts and solvents used were E.Merck analar products. Mercapto benzimidazole, hydroxylamine and diacetone alcohol were Fluka reagents. All other chemicals used were of AR grade.

Elemental analyses (C, H, N, and Cl) were carried out using micro analytical techniques on Heraeus C H N rapid analyzer. Metal estimations were done on a Perkin Elmer 2380 atomic absorption spectrometer. Conductivity measurements were carried out on Elico digital conductivity meter model No: CM -180. The magnetic susceptibility measurements were done on a Faraday balance (CAHN – 7600) using Hg[Co(SCN)₄] as calibrant. TGA and DTA data were recorded on Leeds and Northrup USA instrument. Infrared spectra were recorded on a Perkin Elmer 1600 FT infrared spectrometer in nujol mull. The electronic spectra were recorded on a Shimadzu UV-160 Spectrometer. ¹H NMR data was recorded on a Varian XL 200 MHz NMR spectrometer using CDCl₃ and DMSO-d₆ as solvents. ESR spectra were recorded at room temperature on Jeol-SE-3X, ESR spectrometer using DPPH as standard.

Results and Discussion

All the metal chelates except Zn(II) complex are colored. All of them are stable towards air and moisture. They decompose at high temperatures (>723 K). The metal complexes are

insoluble in common organic solvents like benzene, chloroform, carbon tetrachloride, hexane, methanol, ethanol, acetone *etc.* They are soluble in DMF, dioxane and DMSO. The analytical data (Table 1) of the metal complexes show that all the metal chelates have 1:1 metal to ligand stoichiometry. Conductance measurements were carried out to ascertain the electrolytic/non-electrolytic nature of metal complexes. Molar conductance values of the complexes in 0.001 M DMF solutions at room temperature suggest their non-electrolytic nature. The analytical data reveal that the complexes have general formulae such as $[M(\text{HPBH})\text{Cl}(\text{H}_2\text{O})_2]$, $[M(\text{HPBH})\text{Cl}]$ and $[M(\text{HPBH})\text{Cl}_2(\text{H}_2\text{O})]$, where L = HPBH.

Table 1. Analytical and conductivity data of the ligand and metal complexes

| Ligand/ Complex | Carbon | Found (Calcd) % | | | Metal | Λ_M mhos $\text{cm}^2\text{mol}^{-1}$ |
|---|---------|-----------------|----------|----------|---------|---|
| | | Hydrogen | Nitrogen | Chloride | | |
| HPBH | 63.38 | 7.28 | 22.72 | - | - | - |
| $[\text{C}_{13}\text{H}_{18}\text{N}_4\text{O}]$ | (65.34) | (7.32) | (22.76) | | | |
| Cr(III) complex | 40.39 | 4.19 | 14.47 | 18.33 | 13.54 | 40 |
| $[\text{CrC}_{13}\text{H}_{19}\text{N}_4\text{O}_2\text{Cl}_2]$ | (40.42) | (4.92) | (14.51) | (18.39) | (13.44) | |
| Mn(II) complex | 41.96 | 5.56 | 15.04 | 9.50 | 14.70 | 18 |
| $[\text{MnC}_{13}\text{H}_{21}\text{N}_4\text{O}_3\text{Cl}]$ | (42.00) | (5.65) | (15.07) | (9.55) | (14.78) | |
| Fe(III) complex | 39.88 | 4.81 | 14.38 | 18.80 | 14.24 | 57 |
| $[\text{FeC}_{13}\text{H}_{19}\text{N}_4\text{O}_2\text{Cl}_2]$ | (40.02) | (4.87) | (14.31) | (18.21) | (14.31) | |
| Co(II) complex | 45.89 | 4.85 | 16.57 | 10.35 | 17.25 | 32 |
| $[\text{CoC}_{13}\text{H}_{17}\text{N}_4\text{OCl}]$ | (45.96) | (5.00) | (16.49) | (10.45) | (17.35) | |
| Ni(II) complex | 41.58 | 5.55 | 14.87 | 9.40 | 15.70 | 29 |
| $[\text{NiC}_{13}\text{H}_{21}\text{N}_4\text{O}_3\text{Cl}]$ | (41.53) | (5.59) | (14.92) | (9.46) | (15.64) | |
| Cu(II) complex | 45.31 | 4.89 | 16.25 | 10.22 | 18.41 | 26 |
| $[\text{CuC}_{13}\text{H}_{17}\text{N}_4\text{OCl}]$ | (45.34) | (4.94) | (16.27) | (10.31) | (18.45) | |
| Zn(II) complex | 40.80 | 5.45 | 14.62 | 9.22 | 17.00 | 11 |
| $[\text{ZnC}_{13}\text{H}_{21}\text{N}_4\text{O}_3\text{Cl}]$ | (40.85) | (5.50) | (14.66) | (9.29) | (17.10) | |

¹H NMR data

The ¹H NMR spectrum of HPBH was recorded in DMSO-d₆ solution. δ 11.3 NH proton (s, 1H, of Benzimidazole), a multiplet at δ 6.95- δ 7.25 is due to aromatic protons, δ 1.2 (s, 3H, CH₃), δ 1.3 (s, 3H, CH₃). The methyl groups are non-equivalent and are present in tautomeric form, δ 2.1 (d, 3H, CH₃), δ 2.5 (m, 2H, CH₂), δ 8.4 (s, 1H, NH) and δ 12.1 (s, 1H, OH). The ¹H NMR signals are in correlation with the expected structure³³ (Figure 1).

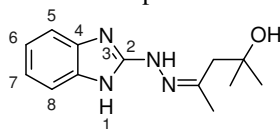


Figure 1. Structure of the ligand HPBH

Infrared spectral studies

The infrared spectrum of the free ligand shows a broad strong band in the region 3450 – 3000 cm^{-1} with stretching vibrations at 3450, 3330 and 3000 cm^{-1} which are assignable to νNH (free), νNH (ring) and νOH (hydrogen bonded). Strong bands located at 1634 and 1579 cm^{-1} can be assigned to $\nu\text{C}=\text{N}$ (exo) and $\nu\text{C}=\text{N}$ (ring). Sharp peaks at 1269 cm^{-1} can be adjudged mainly due to $\nu\text{C}-\text{O}$ (enolic) stretching frequency³⁴. The $\nu\text{N}-\text{N}$ band is located at 1018 cm^{-1} .

The IR spectra of all the complexes show a general broadening and merging of νNH and νOH bands. In the spectra of all the complexes, no band is observed around 3000 cm^{-1} which was present in the free ligand and it suggests the cleavage of intramolecularly H bonded νOH with subsequent deprotonation and coordination through oxygen of OH group. It is further substantiated by the upward shift of $10\text{-}20\text{ cm}^{-1}$ in $\nu\text{C-O}$ (enolic) frequency. This further confirms the participation of oxygen of enolic group in complex formation³⁵. A negative shift in $\nu\text{C=N}$ (exo) at 1634 cm^{-1} and $\nu\text{C=N}$ (ring) at 1579 cm^{-1} to the extent of $20\text{-}30\text{ cm}^{-1}$ in the spectra of the chelates suggest the involvement of azomethine nitrogen free as well as azomethine nitrogen benzimidazole ring in coordination³⁶.

In the spectra of all the complexes, there is an upward shift in $\nu\text{N-N}$ by $10\text{-}30\text{ cm}^{-1}$, which further supports the involvement of azomethine nitrogen of hydrazine chain in coordination³⁷. The peak at 740 cm^{-1} , which is assigned to the NH out of plane deformation vibration in the free ligand, is not altered on chelation. It reveals the non-participation of imine nitrogen in coordination.

The spectra of all the chelates show a broad band in the region $3300\text{-}3000\text{ cm}^{-1}$, which can be attributed to νOH . This supports the presence of coordinated water in Cr(III), Fe(III), Mn(II), Ni(II) and Zn(II) complexes. The rocking modes further supports the presence of coordinated water and wagging modes observed in the range $880\text{-}930\text{ cm}^{-1}$ and $670\text{-}400\text{ cm}^{-1}$ respectively³⁸⁻³⁹ further substantiates the presence of coordinated water and reveals the absence of lattice water. These non-ligand bands can be interpreted to the presence of coordinated water. The absence of vibration modes in the range of $3500\text{-}3200\text{ cm}^{-1}$ and $1670\text{-}1580\text{ cm}^{-1}$ is a clear evidence for the absence of lattice water.

The far IR spectra of all the metal chelates contain non-ligand bands around $240\text{-}280\text{ cm}^{-1}$ and these are assigned to $\nu\text{M-Cl}$. The spectra of all the complexes show non-ligand bands in the region $460\text{-}640\text{ cm}^{-1}$. These are assigned to $\nu\text{M-O}$ and $\nu\text{M-N}$ bands⁴⁰⁻⁴¹. Based on the IR data it is inferred that the ligand HPBH behaves as a monobasic tridentate ligand with O: N: N donor sequence.

Mass Spectral data

The mass spectrum of the ligand shows its characteristic peaks at m/z values: 91,104,112,118,130,133,174, 228 and 246. These are in accordance with the proposed structure of the ligand (Figure 2).

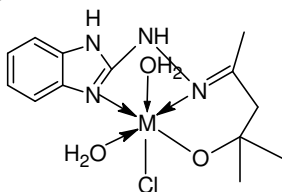


Figure 2. Structure of the metal complexes, where $\text{M}=\text{Mn(II)}$, Ni(II) and Zn(II)

Thermogravimetric and differential thermal analyses

The thermal stability and decomposition temperature of the complexes were studied in air by simultaneous recording of TG and DTA curves. The measurements were made at a heating rate of 10 K min^{-1} . 100 mg samples were heated in platinum crucibles in air in the temperature range $300\text{-}1273\text{ K}$ with TG sensitivity of 100 mg, full scale. Thermo gravimetric analysis was carried out to confirm the presence of coordinated water. It shows a weight loss of one mole of coordinated water per mole of the complex with respect to the trivalent

complexes. The loss has been found to be in one step. One endothermic peak was observed in differential thermal analysis. The loss of water occurs at 433 K for Cr(III) and at 453 K for Fe(III) complexes. In bivalent complexes Mn(II), Ni(II) and Zn(II) the weight loss is observed to be two moles of coordinated water per mole of the complex. The loss of water occurred in one step⁴². It occurs at 443 K for Mn(II), 453 K for Ni(II) and 463 K for Zn(II) complexes⁴³. The complexes have decomposition temperatures in the range 723 K-825 K. The complexes have lost small molecules like H₂O, CH₃OH, HCl and Cl₂ during the process of thermal treatment. Some phase changes in the solid state are also observed.

Magnetic susceptibility measurements

The magnetic moments are measured at room temperature *i.e.*, at 300 K. The magnetic moments are 3.85, 5.95, 5.92 and 3.42 B.M. for Cr(III), Mn(II), and Fe(III) and Ni(II) respectively. They correspond to d³, d⁵, d⁵ and d⁸ configurations of the complexes respectively⁴⁴. As the magnetic moments correlate very well with the mononuclear complexes, low temperature magnetic measurements were not carried out. Subnormal magnetic moments have not been obtained. Metal-Metal interactions were not visualized. Hence Magnetic moments were not studied at low temperatures or liquid nitrogen temperatures. The complexes are assigned with a high spin octahedral geometry. Co(II) complex exhibits a magnetic moment of 4.2 B.M. This higher value is attributed to the orbital magnetic moment. The Cu(II) complex shows a magnetic moment of 2.15 B.M. This value supports the square planar geometry⁴⁵.

Electronic spectral studies

The electronic spectrum of Cr(III) complex shows three intense bands centered at 17290, 22727 and 25000 cm⁻¹. The first band can be assigned to ⁴A_{2g} → ⁴T_{2g} (F) transition and the second band is assigned to ⁴A_{2g} → ⁴T_{1g} (F) and the third can be attributed to ⁴A_{2g} → ⁴T_{1g}(P) transition respectively. These transitions support octahedral geometry around the Cr(III) complex⁴⁴.

The Mn(II) and Fe(III) complexes with d⁵ configuration exhibit absorption bands in the region 25320, 33400 cm⁻¹ for Mn(II) complex and 27000, 37000 cm⁻¹ for Fe(III) complex. These bands may be attributed to number of spin forbidden transitions involving ⁶A_{1g} ground state and several higher energy quartet states, which are in consistent with octahedral geometry around Mn(II) and Fe(III) metal ions⁴⁵.

The spectrum of Co(II) complex shows two bands at 8348 and 18348 cm⁻¹. These bands are assigned to ⁴A_{2g} → ⁴T_{1g} (F) and ⁴A_{2g} → ⁴T_{1g}(P) transitions respectively. These transitions are consistent with the proposed tetrahedral geometry to this complex. Further the blue color of the complex supports its proposed tetrahedral disposition.

The spectrum of Ni(II) complex shows absorption bands in the region 8400, 14730 and 21000 cm⁻¹. The band at 8400 cm⁻¹ is assigned to ³A_{2g} → ³T_{2g} while the band at 14730 and 21000 cm⁻¹ are assigned to ³A_{2g} → ³T_{1g}(F) and ³A_{2g} → ³T_{1g}(P) transitions respectively in an octahedral geometry⁴⁶.

The spectrum of Cu(II) complex shows a broad band observed in the region 18181 - 21978 cm⁻¹. The position and shape of this band suggests the merging of the three electronic transitions *i.e.*, ²B_{1g} → ²B_{2g}; ²B_{1g} → ²A_{1g} and ²B_{1g} → ²E. A square planar geometry is proposed for this complex^{47,48}. The black color of the complex further supports its proposed stereochemistry.

The ESR spectrum of Cu(II) complex was recorded at room temperature. It showed only single broad signal. Hyperfine splitting signals were absent. It shows a g_{av} value 2.087, which is consistent with its square planar geometry. The shape of the curve indicates that it has isotropic symmetry.

Physiological activity

The culture medium was prepared by the addition of 1.5 g of Beef extract, 1.5 g of yeast extract, 5 g of peptone, 1 g of dextrose, 3.5 g of NaCl and 3.68 g of dipotassium hydrogen phosphate, 1.32 g of monopotassium dihydrogen phosphate and 15 g of agar agar in one liter of water. The pH of the medium was adjusted to 7.0. The medium is sterilized at 121 °C in an autoclave at 15 lbs pressure for 15-20 min. The medium was cooled to 45-50 °C. 20 mL of the medium was poured in each of the petridishes. All the apparatus were sterilized before use. *E.coli* and *S.aureus* were introduced into the petriplates and were grown.

Bacteriological testing

Bacteriological testing was done using paper disc method. Paper discs were soaked in the in DMF solutions of the ligand and the metal complexes. Solutions are prepared in two different concentrations *i.e.*, 100 µg /mL and 1000 µg /mL in DMF. Paper discs were soaked in these solutions. They were dried. The dried paper discs were introduced into the completely grown culture plates of *E.coli* and *S.aureus*. The percentage inhibition of the bacteria was calculated by the following equation:

$$\% \text{ Inhibition} = \frac{100(C - T)}{C}$$

Where C is the diameter (mm) in controlled plates after 72 h and T is the diameter in mm in treated plates after 72 h. From the data it is observed that the ligand shows 8.9 and 44.5% inhibition at 100 µg /mL and 1000 µg /mL. The complexes also show maximum % inhibition against *E.coli*. Maximum of 11- 20% in low concentrations and 46-58% in high concentrations. The order of inhibition is Bavistin > Cu(II) > Ni(II) > Zn(II) > Co(II) > Cr(III) > Mn(II) > Fe(III) > ligand. The percentage inhibition against *S.aureus* is 9.5-19% in the lower concentration and 40-51% in the higher concentration. The order of inhibition against *S.aureus* is bavistin > Ni (II) > Cu (II) > Zn (II) > Mn (II) > Cr (III) > Fe (III) > Co (II) > ligand (Table 2). The antibacterial activity of the ligand and complexes has been less than bavistin. The metal complexes show more physiological activity than the ligand.

Table 2. Fungicidal activity of the ligand and its metal complexes

| Ligand/ Complex | Percentage inhibition after 72 h, conc. in µg/mL | | | |
|--------------------|--|------|------------------------------|------|
| | <i>Escherichia coli</i> | | <i>Staphylococcus.aureus</i> | |
| | 100 | 1000 | 100 | 1000 |
| HPBH | 8.9 | 44.5 | 9.5 | 40 |
| Cr(III) complex | 15 | 50 | 12 | 45 |
| Mn(II) complex | 13 | 49 | 14 | 47 |
| Fe(III) complex | 11 | 46 | 11 | 43 |
| Co (II) complex | 16 | 52 | 10 | 41 |
| Ni (II) complex | 18 | 56 | 19 | 51 |
| Cu (II) complex | 20 | 58 | 18 | 50 |
| Zn (II) complex | 17 | 54 | 16 | 48 |
| Bavistin | 82 | 100 | 87 | 100 |

Conclusion

Octahedral geometry is proposed for Cr(III), Fe(III) Mn(II), Ni(II) and Zn(II) complexes (Figure 3). A square planar disposition is proposed for Cu(II) complex (Figure 4) and a tetrahedral geometry has been given for Co(II) complex (Figure 5).

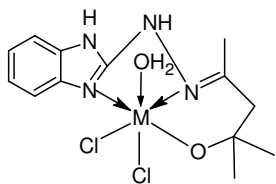


Figure 3. Structure of metal complexes, where M=Cr(III) and Fe(III)

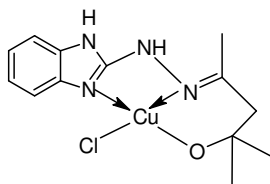


Figure 4. Structure of Cu(II) complex

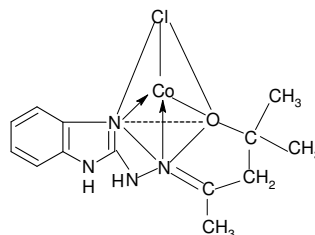


Figure 5. Structure of Co(II) complex

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