

Spectral Studies of Some Transition Metal Ion complexes with 4-[(E)-(Ferrocene-1-Methylidene) Amino] Pyridin-2-ol

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Abstract: Several new complexes of some transition metal ions with organometallic compounds were derived from ferrocencecarboxylaldehyde and 4-amino-2-hydroxy pyridine. These organometallic complexes were investigated by using some analytical techniques like infrared, mass spectra, electronic spectra, electron spin resonance, thermal analysis, magnetic moment, conductivity and antimicrobial activity. From the obtained elemental analysis data, organometallic compounds complexes shows 1:2 [M: L] ratio and general formula of the complex is $[ML_2 \cdot 2H_2O]$. These complexes revealed a non-electrolytic nature. The magnetic moment values of the complexes exhibited the paramagnetic as well as diamagnetic in nature. The coordination behavior of the metal ions towards to the investigated organometallic compounds takes place through $>C=N-$ and $-OH$ groups. The electronic spectral data shows that all the complexes are covalent in nature, octahedral structure with co-ordination number six. Organometallic compound complexes were loses two water molecules subjected to simultaneous thermo gravimetric analysis, to study their decomposition mechanism and thermal stability. Mass spectra of the organometallic compound and their complexes are matched with theoretical values of the masses. The prepared organometallic compounds and their metal complexes were screened for their antibacterial activity against some bacterial species, *Escherichia coli*, *Pseudomonas aeruginosa* and *Staphylococcus Pyogones*. The Antimicrobial activity data show the metal complexes to be more active than the parent organometallic compounds.

Key words: Ferrocencecarboxylaldehyde, 4-Amino-2-Hydroxy Pyridine, Infrared, Mass Spectra, Electronic Spectra, ESR, Thermal Analysis, Conductivity and Antimicrobial Activity.

I. Introduction

The coordination chemistry of organometallic compounds has a significant role as ligands from last two decade after its discovery [1, 2]. Organometallic compounds and its metal ion complexes are important owing to their biochemical synthesis [3], electrochemical analysis [4], analytical methods [5], antifungal, antimicrobial activities [6-9] and used as a catalysts [10-11]. Organometallic compound containing nitrogen, oxygen and sulphur atom donors have structurally identical with natural biological systems. In the biological systems due to presence of azomethane ($>C=N-$) groups [12], Presence of nitrogen ($-NH_2$) and oxygen from hydroxyl ($-OH$) or carboxylic group ($-COOH$) donor atoms properties of the complexes to a great extent as effective and stereo specific catalysts for oxidation, reduction and hydrolysis [13]. Different types of organometallic compounds with metal ion complexes have been extensively studied and exhibit wide applications, especially in biological systems [14]. The study of mono, di, tridentate metal ion metal complexes shows spectral properties, therefore these kind of complexes still continue to play a very important role in our understanding of various aspects of coordination chemistry of metals. They play significant role in numerous biological systems [15-16] due to their coordinating ability with various transition metals to form organometallic compounds. In this paper we are presenting the preparation method, spectral studies and antimicrobial studies of schiff bases organometallic compounds and their metal ion complexes.

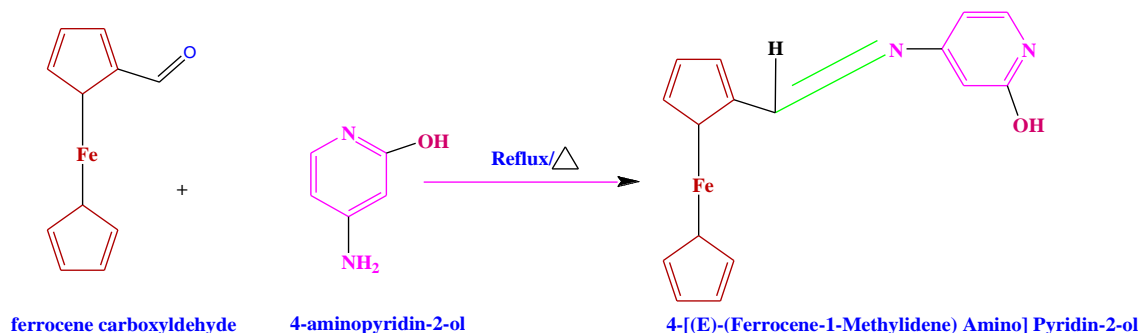
II. Experimental Methods:

All the chemicals and reagents were used for this research work in analytical grade, used without any further purification.

2.1 Preparation of organometallic compound: Ferrocencecarboxylaldehyde (0.306g) and 4-Amino-2-Hydroxy Pyridine (0.110g) were separately dissolved in 100 cm³ alcohol. The equal volume of the both the compounds were mixed in round bottom flask, few drops of acetic acid were added. Then this reaction mixture were reflux for near about five hours using water condenser, after complete refluxation, solution were cool at room temperature over night for 48 hours solid separates. This solid compound keeps for drying at 60 °C in oven. The yield of organometallic compound is 74%.

2.2 Preparation of organometallic complexes: The organometallic complexes were prepared by mixing equimolar concentration (0.01 M) of ethanolic solutions of the 4-[(E)-(Ferrocene-1-Methylidene) Amino] Pyridin-2-ol with the (0.01M) aqueous solution of Mn-acetate, Co-acetate, Ni-acetate, Cu-acetate, Zn-acetate, PdCl₂ & PtCl₂ salts. These solutions were mixed and refluxed for three hours using 304 drops of alcoholic ammonia solution. After three hours, the complexes were cooled at room temperature, filtered, washed with ether and dried at 60°C in oven. The yields of product were obtained in between 52-65%.

2.3 Reaction:



III. Result and Discussion:

The analytical and physical properties of organometallic compound and complexes are given in table: 1. The organometallic compound and their complexes were coloured, stable at room temperature for long time. The organometallic compound is soluble in alcohol but complexes were insoluble in organic solvents like toluene, methanol, ethanol, acetonitrile & chloroform. They were soluble in dimethyl sulfoxide and dimethyl formamide. They are decomposed in the range 210-240°C. The melting point and decomposition point were reported in open capillary but found uncorrected. The molar conductivity values of the complexes are observed in dimethyl sulfoxide in 10⁻³ molar solution. It is non-electrolytic in nature [17-18]. The metals contents were estimated by volumetric titration using different indicators with (0.01M) ethylene diamine tetra acetate solution expect palladium and platinum metal they are estimated by AAS method. Iron content was estimated by gravimetrically. The metal-ligand ratio is 1:2.

3.1 Infrared spectra: The infrared spectra of organometallic compound and complexes were compared with substituted moieties. The ligand shows prominent bands at 3415 cm⁻¹ due to ν-OH (hydroxyl ion) absorption. The bands observed at 1680 cm⁻¹ due to azomethane (>C=N-) group present in the organometallic compound. In the complexes band due to the -OH hydroxyl Group) is not found because there is coordination with metal ions. The band are shifted to the lower frequency in azomethane (>C=N-) group due bonding with metal ions in the range at 1630-1605cm⁻¹. The two new bands were observed in range between at 3450-3420 and 3520-3490 cm⁻¹ for two coordinated water molecules. The bands observed far infrared region at 545-530 cm⁻¹ due to (M→N) linkage and 485-465 cm⁻¹ for (M→O). The central metal ions have six co-ordinate numbers and ligand behaves as a bidentate [19-26]. IR bands of the organometallic compound and complexes are reported in table: 2.

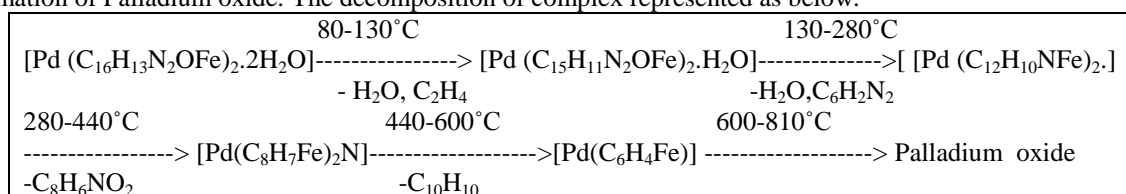
3.2 Electronic spectra: The spectral parameters and assignment are given in Table: 3. The electronic d-d transition bands normally show weak perturbation due to complexation an increase in the intensity, shift to the red region and also splitting of some bands are observed on complex formation. The position, shapes of Cu⁺² and Ni⁺² are observed in solution phase using ethanol and dimethyl sulfoxide. The Cu⁺² complexes shows two peaks and Co⁺² complexes shows three intense peak, which have lower energies as compared to those of aqua complex. The magnitude of the bathochromic shift of the bands in each case meager Nephelauxetic effect (β) [27], the bonding parameter (b1/2) [28] and Sinha's parameter (δ %) [29] have been calculated. The bonding parameter reflects the participation of 3d orbital [30]. The b^{1/2} value obtained for the present complexes indicates a decreasing order of 3d- orbital participation in the Cu⁺² and Ni⁺² complexes. The average value of Sinha's parameter (δ %) obtained in each case is positive and smaller, indicating the presence of weak covalent bonding in the complexes [31].

3.3 Magnetic Moment: The corrected magnetic moment (μ_{eff}) in Bohr magneton units of the organometallic complexes are given in Table: 1. The magnetic moments of the complexes measured at room temperature except

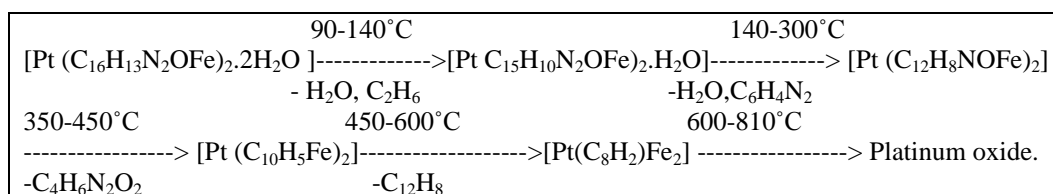
that of zinc (II) complex are diamagnetic, while other complexes are paramagnetic in nature. This indicates slight participation of the 3d electron in bond formation [32].

3.4 Thermal analysis: Thermogravimetric and differential thermal analysis results of [Pd (C₁₆H₁₃N₂OFe)₂.2H₂O] and [Pt (C₁₆H₁₃N₂OFe)₂.2H₂O] are reported as below. The complexes lose weight due to exothermic and endothermic process. These complexes are thermally stable at room temperature and decompose in different steps.

3.4.1 [Pd (C₁₆H₁₃N₂OFe)₂.2H₂O] Complex: Thermal analysis study of Pd²⁺ complex shows loss in weight in the temperature range 80-130°C, which corresponds to loss of one water molecule and some part of complex. The experimental percentage loss, which is calculated from Thermogravimetric analysis curve, is 7.78%. This value comparable with theoretical percentage loss. The differential thermal analysis peak at this range is endothermic [33]. In the temperature range 130°C-440°C, the some part of complex is lost. The major part of complex agent is lost in the temperature range of 440-600°C. The experimental percentage loss is 33.72%, which is obtained from Thermogravimetric analysis curve. The experimental percentage loss value is comparable with theoretical percentage loss value i.e. 36.17%. The differential thermal analysis peak is exothermic [34]. The probable leaving part of complex in this temperature range is C₁₀H₁₀. The temperature range 600-810°C leading to the formation of Palladium oxide. The decomposition of complex represented as below.



3.4.2 [Pt (C₁₆H₁₃N₂OFe)₂.2H₂O]Complex: Thermal analysis study of Pt²⁺ complex shows loss in weight in the temperature range 90-140°C, which corresponds to loss of water molecule and some part of the complex. The experimental percentage loss, which is calculated from Thermogravimetric analysis curve, is 7.72%. This value comparable with theoretical percentage loss. The differential thermal analysis peak at this range is endothermic. In the temperature range 140-450°C, the some part of complex is lost. The major part of chelating agent is lost in the temperature range of 450-600°C. The experimental percentage loss is 33.45%, which is obtained from Thermogravimetric analysis curve. The experimental percentage loss value is comparable with theoretical percentage loss value i.e. 36.18%. The differential thermal analysis peak is exothermic. The probable leaving part of complex in this temperature range is C₁₂H₈. The temperature range 600-810°C leading to the formation of Platinum oxide. The decomposition of complex represented as below.



3.5 Mass spectra: The ESI mass spectra of the organometallic compound, their metal complexes recorded and are listed in table: 4 and fig3 &4. The mass spectra organometallic compound shows a molecular ion peak at m/z = 322.2. The Mn (II) complex shows a molecular ion peak at m/z = 700.87 and Zn (II) complex shows a peak at m/z = 712.7 which corresponds to molecular weight of the respective compounds. While Co (II) complex shows m/z=707.12 and Cu (II) complex at m/z=711.13 which corresponds to [M+1]. The Ni (II) complex shows m/z=704.2 which corresponds to [M+2] peak respectively. The Pd (II) and Pt (II) complexes show the m/z peaks at 753.45 & 842.12 respectively. These peaks support to the structure of the complexes and confirm the stoichiometric of organometallic complexes were [ML₂.2H₂O] type. The different molecular ion peaks appeared in the mass spectra of complexes (abundance range 2-100%) are attributed to the fragmentation of the metal complex molecule obtained from the rupture of different bonds inside the molecule by successive degradation leading to many more important peaks due to formation of various radicals[35]. The spectra of complexes show molecular ion peaks in good agreement with the structure suggested by elemental analysis, spectral and magnetic studies. The spectra of the organometallic compounds and its complex shows characteristic molecular ion peak at their expected m/z values confirming their monomeric form [36].

3.6 Electron spin resonance: The Electron spin resonance spectra of organometallic complexes provide information on the basis of hyperfine and superhyperfine structures. Electron spin resonance spectra of the [Cu (C₁₆H₁₃N₂OFe)₂.2H₂O] complex have been recorded on X -band fig 5 and their g_{II}, g_I, g_{av}, Δg and G values have been calculated. The ESR parameters of [Cu (C₁₆H₁₃N₂OFe)₂.2H₂O] complex g_{II}, g_I, g_{av} and Δg have 2.01, 2.11, 2.05 and 0.08 respectively. The X-band EPR spectrum of [Cu (C₁₆H₁₃N₂OFe)₂.2H₂O] complex shows a single line at g≈2.01, which is attributed to a single s=1/2 species; this provides information about the extent of the delocalization of unpaired electron [37-39]. The g_{II} < g_I value shows the covalent nature of M:Lbond. The value of ESR parameters of [Ni (C₁₆H₁₃N₂OFe)₂.2H₂O] complex, g_{II}, g_I, g_{av}, Δg and G have 2.214, 2.322, 2.272, 0.062 and 1.64 respectively. The covalent nature of the M:L bond in the complex is further supported by the G value which is <4.0. The value g_{II} > g_I is well consistent with a primarily dx²-y² ground state. The G value of 1.64 indicates considerable exchange interaction in the complex. According to Hathway, if the value of G is greater than four, the exchange interaction is negligible; whereas when the value of G is less than 4 to considerable exchange interaction is indicated in the complex. The parameter g_{av} were obtained by equation [g_{av}=(1/3 g_{II}+ g_I)]. The g_{II} value (<2.3) indicates covalent character of the M:L bond [40-41].

3.7 Antimicrobial activity: Organometallic compound and its complexes have been screened in-vitro against the some bacteria like Escherichia coli, Pseudomonas aeruginosa and Staphylococcus Pyogones species by well diffusion method using agar as nutrient medium. Here streptomycin has been used as a standard. The microbial activity of the schiff base organometallic compound and its complexes are listed in table: 5. All the complexes shows higher antibacterial activity against Escherichia coli and Pseudomonas aeruginosa. On the other hand for Staphylococcus Pyogones bacteria shows more biological activity towards schiff base organometallic compound.

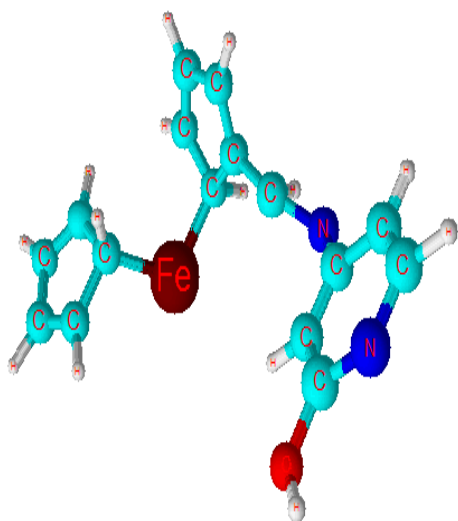


Fig: 1 . 3D View of organometallic comp.

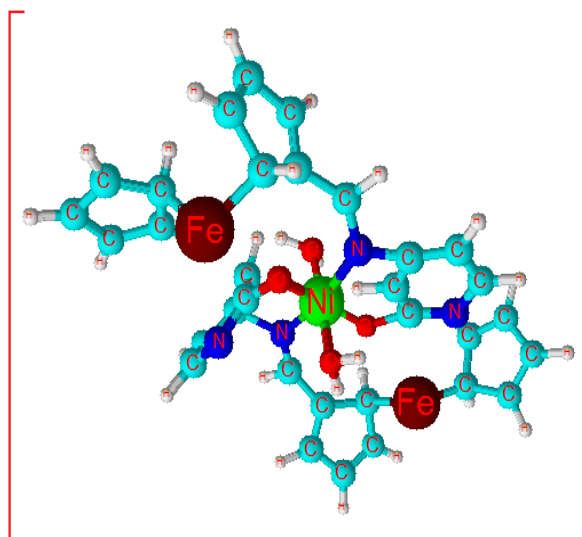


Fig:2. Structure of [Ni(C₁₆H₁₃N₂OFe)₂.2H₂O] complex

Schiff base /Complexes	% yield	MP/DP °C	C%	N%	Fe%	M%	BM.μeff
C ₁₆ H ₁₄ N ₂ OFe	74	255-258	62.77	9.15	18.24		
			62.42*	8.95*	17.98*		
[Mn (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	53	237-240	54.81	7.99	15.93	7.83	5.48
			54.34	7.55	15.59	7.45	
[Co (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	61	235-238	54.5	7.94	15.84	8.36	4.40
			54.11	7.59	15.74	8.05	
[Ni (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	54	232-235	54.52	7.95	15.84	8.33	3.21
			54.22	7.59	15.71	8.01	
[Cu (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	62	227-230	54.14	7.89	15.73	8.95	1.87
			53.85	7.58	15.42	8.70	
[Zn (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	58	221-224	54.01	7.87	15.69	9.19	dimag
			53.72	7.59	15.41	8.92	
[Pd (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	52	215-218	51.06	7.44	14.84	14.14	
			50.82	7.17	14.59	13.95	
[Pt (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	54	210-213	45.68	6.66	13.27	23.19	
			45.31	6.35	13.01	22.91	

Table .1: Analytical, Physical Data of Schiff Base organometallic and Complexes [(*) Experimental value]

Schiff base /Complexes	ν C-OH	ν C=N	ν M-N	ν M-O	2H ₂ O
C ₁₆ H ₁₃ N ₂ OFe	3415	1680			-
[Mn (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]		1608	543	472	3445,3498
[Co (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]		1614	538	467	3422, 3502
[Ni (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]		1622	527	465	3442, 3510
[Cu (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]		1630	532	472	3428, 3495
[Zn (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]		1623	538	478	3437, 3518
[Pd (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]		1614	533	480	3430, 3490
[Pt (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]		1605	525	485	3444, 3514

Table 2 : Relevant IR Spectral Data Of The Schiff Base and Its Complexes in Cm⁻¹

Complexes	Absorption bands cm ⁻¹	Assignments	Spectral Parameter
[Ni (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	20730 14200 11275	³ A _{2g} (F) → ³ T _{1g} (F) ³ A _{2g} (F) → ³ T _{1g} (F) ³ A _{2g} (F) → ³ T _{2g} (F)	β=0.9695 δ%=1.58 b ^{1/2} = 0.0873 η=0.0156
[Cu (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	25840 13345	⁴ T _{1g} → ⁴ A _{2g} (P) ⁴ T _{1g} → ⁴ T _{2g}	β=0.9610 δ%=2.05 b ^{1/2} = 0.099 η=0.020

Table 3: electronic spectra and their parameters

Schiff base /Complexes	Expected m/z	Found m/z	Peak assigned
C ₁₆ H ₁₄ N ₂ OFe	320.251	322.2	M+2
[Mn (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	701.233	700.8	M
[Co (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	705.228	707.1	M+2
[Ni (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	704.988	704.2	M
[Cu (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	709.841	711.1	M+2
[Zn (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	711.704	712.7	M+1
[Pd (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	752.715	753.4	M+1
[Pt (C ₁₆ H ₁₃ N ₂ OFe) ₂ .2H ₂ O]	841.373	842.1	M+1

Table 4. Mass of schiff base organometallic and complexes.

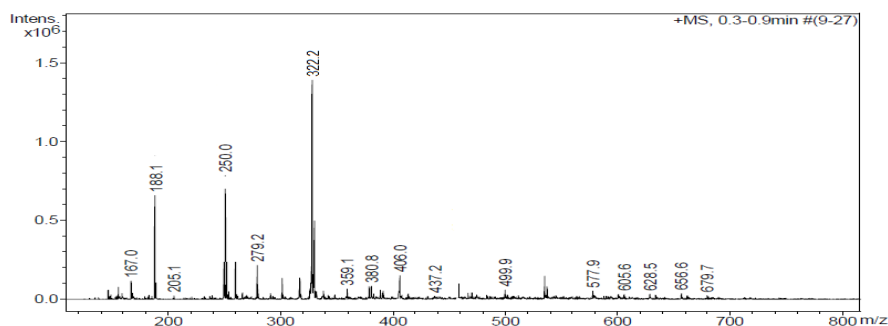
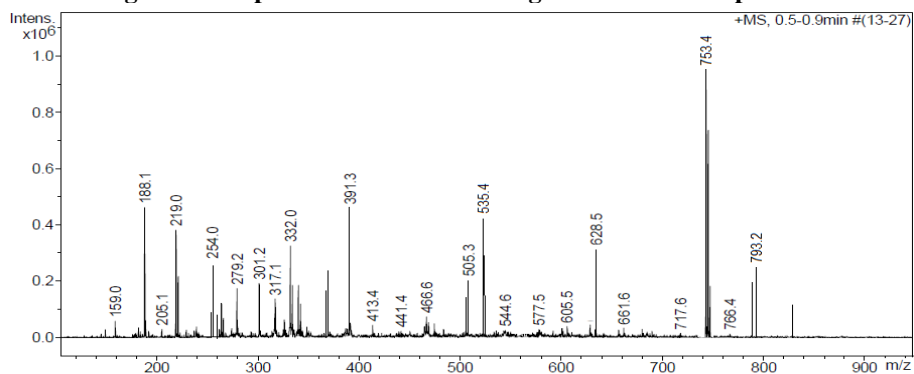


Fig 3: Mass Spectra of Schiff Base Organometallic Compound.

Fig 4: Mass Spectra Of [Pd (C₁₆H₁₃N₂OFe)₂.2H₂O] complex.

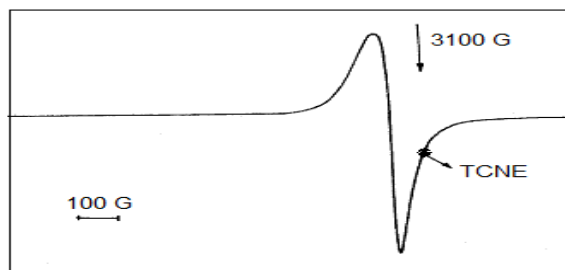


Fig 5: EPR of $[\text{Cu}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$ complex.

Schiff base /Complexes	Escherichia coli	P. aeruginosa	Staphylococcus Pyogones
$\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe}$	+	+	++++
$[\text{Mn}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$	++	+	++
$[\text{Co}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$	++	++	+
$[\text{Ni}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$	++	+++	+
$[\text{Cu}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$	+++	+++	++
$[\text{Zn}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$	++	+++	++
$[\text{Pd}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$	+++	+++	++
$[\text{Pt}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$	+++	+++	++

[Very Highly active = ++++ (inhibition zone > 20 mm); Highly active = +++ (inhibition zone > 15 mm); slightly active = ++ (inhibition zone > 10 mm); less active =+ (inhibition zone > 5 mm)]

Table: 5. Microbial activity of schiff base organometallic compound and complexes.

II. Conclusion:

The organometallic compounds were synthesized and characterized various spectral techniques. The analytical data and spectral data indicate that, metal ligand stoichiometric ratio is 1:2. Complexes are non-electrolytes in nature. The spectral data indicates that, organometallic compounds are bidentate coordinating through nitrogen atom of the azomethane and oxygen atoms of hydroxyl group of the 4-[(E)-(Ferrocene-1-Methylidene)Amino]Pyridin-2-ol. Complexes are assigned to be in octahedral geometry with coordination number is six. The pathogenic bacteria like Staphylococcus Pyogones shows more biological activity towards schiff base organometallic compound than its metal complexes. We are proposing the following probable structure of the complex $[\text{M}(\text{C}_{16}\text{H}_{13}\text{N}_2\text{OFe})_2 \cdot 2\text{H}_2\text{O}]$.

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