

Spectral studies of 5-({4-amino-2-[(Z)-(2-hydroxybenzylidene) amino] pyrimidin-5-yl} methyl)-2, 3, 4-trimethoxybenzene with some transition metal ions.

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Abstract: Some transition metal ions Complexes with 5-({4-amino-2-[(Z)-(2-hydroxybenzylidene) amino] pyrimidin-5-yl} methyl)-2,3,4-trimethoxybenzene were prepared and characterized by elemental analyses, Infrared, magnetic moment, electronic spectra, mass spectra, X-ray powder diffraction, molar conductance and thermal analysis (TGA). The complexes have general formulae $[ML_2 \cdot 2H_2O]$ {where $M = Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Pd(II)$ and $Pt(II)$ }. The coordination behavior of the metal ions towards to the investigated Schiff base takes place through $-C=N, -NH_2$ and $-OH$ groups. The obtained C, H and N elemental analysis data showed the Metal: Ligand ratio is 1:2 [M: L] ratio. The molar conductance data reveal that all the metal complexes are non-electrolytic in nature. From the magnetic moments the complexes are paramagnetic except Zn metal ion complexes have octahedral geometry with coordination number eight. The thermal behavior of these complexes shows that, the hydrated complexes have loses two water molecules and immediately followed by decomposition of the anions and ligand molecules in the second and third stage. The Schiff bases and metal complexes show good activity against some bacteria. The antimicrobial results indicate that, the metal complexes have better antimicrobial activity as compared to the prepared Schiff base.

Keywords: Infrared, Mass spectra, X-ray powder diffraction, TGA/DTA, Anti- microbial activity.

I. Introduction

A large number of Schiff bases and their complexes have been studied for their important properties, e.g., their ability to binds with oxygen[1-2], catalytic activity in hydrogenation of olefins[3], transfer of an amino group[3], photo chromic properties[4] and from the complexes towards some toxic metals[5-7]. The real impetus towards developing their Co-ordination Chemistry with their physico-chemical and biological properties [8-9]. Metal complexes of Schiff bases were play an important role in the development of Co-ordination Chemistry. Schiff bases were providing important and essential ways for chemical and biological activity of compounds [10]. The high affinity for the complexation of the Schiff bases towards the transition metal ions were utilized in preparation of their solid complexes. Schiff base with donors (N, O, etc.) have structural similarities with natural biological systems, imports in elucidating the mechanism of transformation, racemization reaction in biological systems physiological and pharmacological activities associated with them [11-12]. The complexes were characterized by IR, magnetic moment, electronic spectra, mass spectra, X-ray powder diffraction, molar conductance, and thermal analysis (TGA) Magnetic susceptibility.

II. Experimental

2.1 Materials and methods: All reagents and chemicals were analytical grade, used without any further purification. The drugs were obtained from various pharmaceutical companies. All solvents were purified & distilled by distillation before using.

2.2 Preparation of Schiff base and metal complexes: Schiff bases were prepared by mixing of equi molar ethanolic solutions of the 5-(3, 4, 5- trimethoxybenzyl) pyrimidine- 2, 4- diamine and 2-hydroxybenzaldehyde by condensation reaction using few drops of acetic acid. The resulting mixture was reflux for near about three hours using water condenser. After complete refluxation, the solution were cooled at room temperature, solid separates, dried at 70 °C in oven refer fig: 1. The 0.01M Solution of $MnCl_2, CoCl_2, NiCl_2, CuCl_2, ZnCl_2, PdCl_2$ and $PtCl_2$ salts solutions were prepared in double distilled water. The solutions of 0.01m Schiff base were prepared in absolute alcohol. The 40 cm³ of 0.01M solution of Schiff base and 20 cm³ solution of metal chloride were refluxed for 2 hours using water condenser, by using 8-9 drops of alcoholic ammonia solution were added to adjust the pH range of the solution in between 7.0 – 8.5 refer fig: 2. This solution kept for 24 hours in dark

place the coloured solid residue were obtained. The complexes were filtered, washed with ether and dried at 60°C in oven. The yields of complexes were obtained in range between 69-83%.

2.3 instrumental techniques: IR spectra of the schiff base and its complexes were recorded using KBr pellets by Perkin Elmer spectrophotometer at ICT Mumbai. The C, H and N of schiff base and complexes were determined by using elemental analytical methods. Electronic spectra recorded at ICT-Mumbai using Dimethyl formamide and water as a solvent. TGA/DTA spectra were recorded on Mettler Toledo Star system in the temperature range between 20-850 °C. The Mass spectra were recorded at ESI technique. Melting points of the ligand and decomposition temperature of complexes were determined in College Laboratory. The Molar conductance measurements were recorded in DMF (10^{-3} M) and water using Electronic Digital conductivity meter and cell is calibrated with saturated KCl solution. The Magnetic susceptibilities of complexes were recorded on Gouy balance using Hg [Co (SCN)₄]¹⁴ as a calibrate. X-ray powder diffraction data recorded at TIFR-Mumbai.

III. Result And Discussion:

The physical properties and analytical data of both the Schiff base and its complexes are given in Table :1. All these complexes are colored, except Zn(II) complex and stable at room temperature e.g. Over anhydrous calcium chloride in a desiccators for reasonably long period of time. The complexes are insoluble in organic solvents like toluene, methanol, ethanol, acetonitrile & chloroform but they are soluble in dimethyl sulfoxide and dimethyl formamide. They are decomposed in the range 220-250°C. The melting point and decomposition point reported in open capillary are uncorrected. The values of the molar conductance of complexes were mainly based on the mobility of free ions in the complex solution. The larger value of conductance indicates the higher mobility of the free ions and smaller value of conductance indicates lower mobility of the free ions in the solution. The conductivity values of the complexes are observed in dimethyl sulfoxide in 10^{-3} molar solution. It is non-electrolytic in nature [13].

3.1 Infrared spectra: infrared bands that provide considerable structural evidence for the formation of Schiff bases and their metal ion complexes are reported in Table: 2. the infrared spectra of complexes were compared with ligand and their substituted moieties. The schiff base shows prominent bands observed at 3320 cm^{-1} due to ν NH₂ group and hydroxyl group at 3420 cm^{-1} . The bands observed at 1690 cm^{-1} due to $>\text{C}=\text{N}-$ group. In the complexes band due to anilino NH₂ group shift to lower frequencies at the range 3170-3210 cm^{-1} , the band due to $>\text{C}=\text{N}-$ group is found at 1640-1620 cm^{-1} but there no band found for hydroxyl group due to coordination with metal ions[11-16]. Hence the complexes with 5-({4-amino-2-[(Z)-(2-hydroxybenzylidene) amino] pyrimidin-5-yl} methyl)-2, 3, 4-trimethoxy benzene indicates that, the coordination take place through nitrogen of anilino -NH₂, $>\text{C}=\text{N}-$ and hydroxyl group oxygen. The bands observed at 3450-3425 and 3510-3490 cm^{-1} is due to two coordinated water molecules. The bands observed far infrared region at 530-505 cm^{-1} due to (M \rightarrow N) linkage and 480-460 cm^{-1} for (M \rightarrow O)[17-20]. The central metal ions have eight co-coordinated numbers and ligand behaves as bidentate.

3.2 Electronic spectra: The electronic spectra of the Ni²⁺ and Cu²⁺ of Schiff base complexes have been recorded in DMF + Ethanol solutions in the wavelength range 350-900 nm^{-1} .The spectral parameters and their assignment are listed as 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 [21]. The position shapes and of Ni²⁺ and Cu²⁺ are observed in solution phase using ethanol and dimethyl sulfoxide. The Ni²⁺ and Cu²⁺ complexes have lower energies as compared to those of aqua complex. The magnitude of the bathochromic shift [22] of the bands in each case meager Nephelauxetic effect (β) [23-25], the bonding parameter ($b^{1/2}$) [26] and Sinha's parameter (δ %) [27] have been calculated. The bonding parameter reflects the participation of 3d orbital [21]. The $b^{1/2}$ value obtained for the present complexes indicates a decreasing order of 3d- orbital participation in the Ni²⁺ and Cu²⁺ complexes. The average value of Sinha's parameter (δ %) & η obtained in each case is positive and smaller, indicating the presence of weak covalent bonding character in the complexes [28].

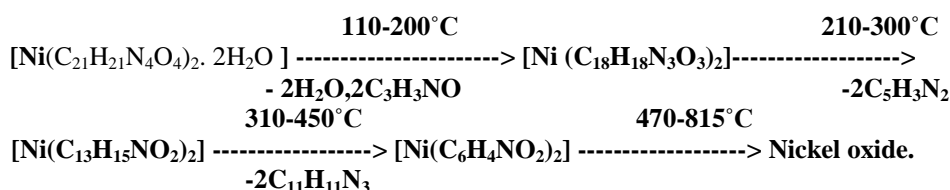
3.3 Magnetic Moment: The corrected magnetic moment (μ_{eff}) in Bohr magnetron units of the Schiff base complexes are given in Table No.1.The magnetic moments of the complexes room temperature except that of zinc (II) complex, which is diamagnetic and other complexes are paramagnetic[29-30]. This indicates slight participation of the 3d electron in bond formation. The μ_{eff} . Values of complexes are listed in Table: 1.

3.4 X-ray Powder diffraction: The complexes were examined for their powder diffraction data. The nature of spectra indicates low crystallinity of the complexes. The major reflexes were used to calculate crystal lattice parameters using Back-cal program on computer by Ito's method [31]. The observed values are reported in Table: 4 and fig.:3. The diffractogram was indexed using POWD (an Interactive Powder Diffraction Data

Interpretation and Indexing Program, Version 2.2) software. The cell parameters of pure crystals were obtained by selecting intense peaks. Calculations of cell parameters reveals that the pure crystal belongs to monoclinic crystal system [32], with unit cell parameters **a** = 28.9780 Å, **b** = 8.7652 Å, **c** = 8.3880 Å, **A** = 13.26, **B** = 77.22, **C** = 158.20 and unit cell volume is 1555.48. The experimental 'D' values are in good agreement with the calculated ones for the above cell parameters of the [Pd (C₂₁H₂₁N₄O₄)₂.2H₂O] complex. The observed value of density of the complex is 1.1740 gm/ cm³ to the best of author's knowledge, x-ray diffraction data on these crystals is limited only to the 2θ values and intensity. Cell parameters, (hkl) values and d-spacing are reported for the first time.

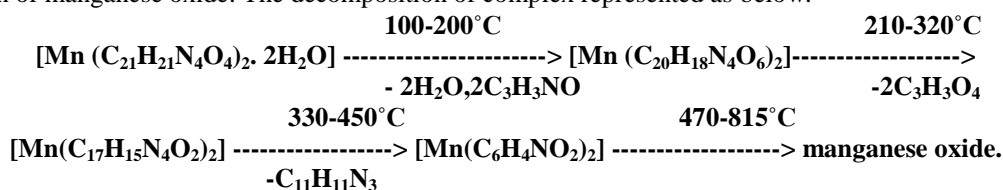
3.5 Thermal analysis: Thermogravimetric and differential thermal analysis results of [Ni (C₂₁H₂₁N₄O₄)₂. 2H₂O] and [Mn (C₂₁H₂₁N₄O₄)₂. 2H₂O] are reported. Complexes loss weight due to exothermic and endothermic process [33-34]. These complexes are thermally stable at room temperature and decompose in four steps.

3.5.1 [Ni (C₂₁H₂₁N₄O₄)₂.2H₂O] complex: Thermal analysis study of Ni²⁺ complex shows loss in weight in the temperature range 110-200°C, which is corresponds to loss of two water molecule and some part of chelating agent. The experimental percentage loss, which is calculated from thermo gravimetric analysis curve, is 14.92. This value comparable with theoretical percentage loss. The differential thermal analysis peak at this range is endothermic. In the temperature range 210-300°C, the some part of chelating agent is lost. The major part of chelating agent is lost in the temperature range of 310-450°C. The experimental percentage loss is 31.49, which is obtained from Thermogravimetric analysis curve. The experimental percentage loss value is comparable with theoretical percentage loss value i.e. 31.80. The differential thermal analysis peak is exothermic. The probable leaving part of chelate in this temperature range is 2C₁₁H₁₁N₃. The temperature range 470-815°C leading to the formation of nickel oxide. The decomposition of complex represented as below.



3.5.2 [Mn(C₂₁H₂₁N₄O₄)₂.2H₂O] complex : Thermal analysis study of Mn²⁺ complex shows loss in weight in the temperature range 100-200°C, which is corresponds to loss of two water molecule and some part of chelating agent. The experimental percentage loss, which is calculated from thermo gravimetric analysis curve, is 15.02. This value comparable with theoretical percentage loss. The differential thermal analysis peak at this range is endothermic.

In the temperature range 210-320°C, the some part of chelating agent is lost. The major part of chelating agent is lost in the temperature range of 330- 450°C. The experimental percentage loss is 31.70, which is obtained from thermo gravimetric analysis curve. The experimental percentage loss value is comparable with theoretical percentage loss value i.e. 31.81. The differential thermal analysis peak is exothermic. The probable leaving part of chelate in this temperature range is 2C₁₁H₁₁N₃. The temperature range 470-815°C leading to the formation of manganese oxide. The decomposition of complex represented as below.



3.6 Mass spectra: The ESI mass spectra of the metal complexes recorded at room temperature are used to compare their composition and are listed in table: 5 and fig: 4&5. The mass spectra of Ni (II) complex shows a molecular ion peak at m/z =455.7 Similarly Cu (II) complex shows a peak at m/z =458.7, which corresponds to molecular weight of the respective compounds. While mass spectra of Mn(II) & Co(II) complexes shows m/z=450.1, m/z= 455.2 respectively, which corresponds to [M+1]. The Zn(II) complex shows m/z=461.9 which corresponds to [M+2] peak respectively. These peaks support to the structure of the complexes. The schiff base complex shows mass peak at m/z= 391.2. 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. 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 complex shows characteristic molecular ion peak at their expected m/z values confirming their monomeric form [35-36].

3.7 Anti-Microbial Activity: Above synthesized schiff base and their complexes were screened against some bacteria by the filter paper disc method at various concentrations using nutrient agar as medium. Sterilized filter paper of 5 mm diameter were soaked in solutions of different concentrations of test samples and introduced on nutrient agar plates. These plates were incubated for 48 hours at 35°C [37-39]. The schiff base and complexes were applied on a paper disc with the help of a micropipette. The discs were left in an incubator for 48 h at 37°C and then applied on the bacteria grown agar plates. The Schiff bases and metal complexes show good activity against bacteria. The antimicrobial results also indicate that the metal complexes are better antimicrobial agents as compared to the Schiff bases.

IV. Figures and Tables

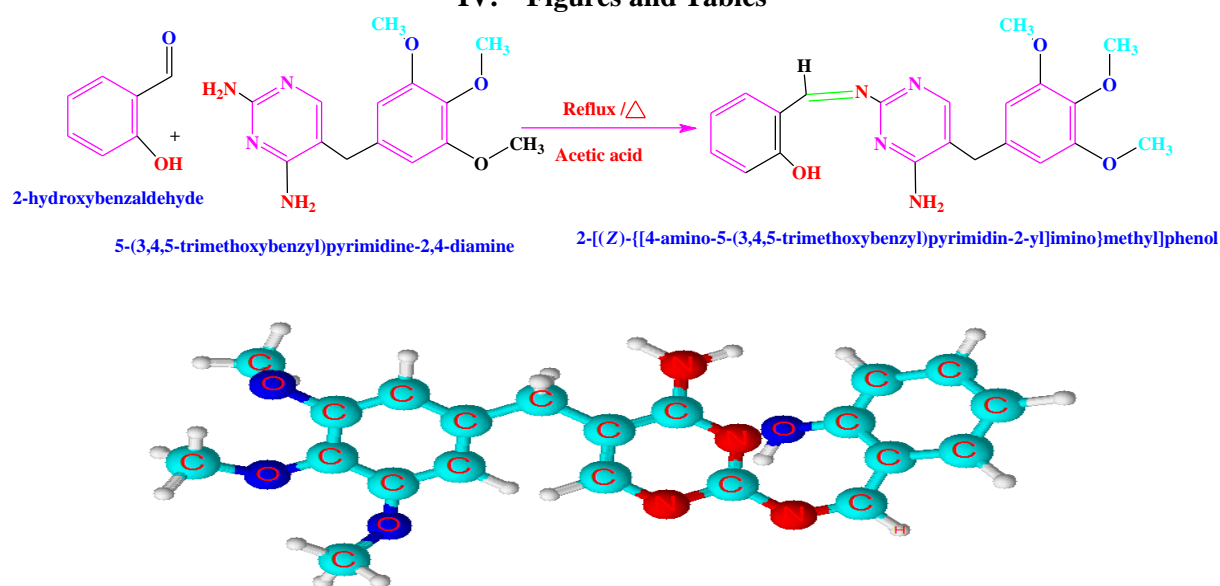


Fig.1.Synthesis method of schiff base and 3D structure of Schiff Base

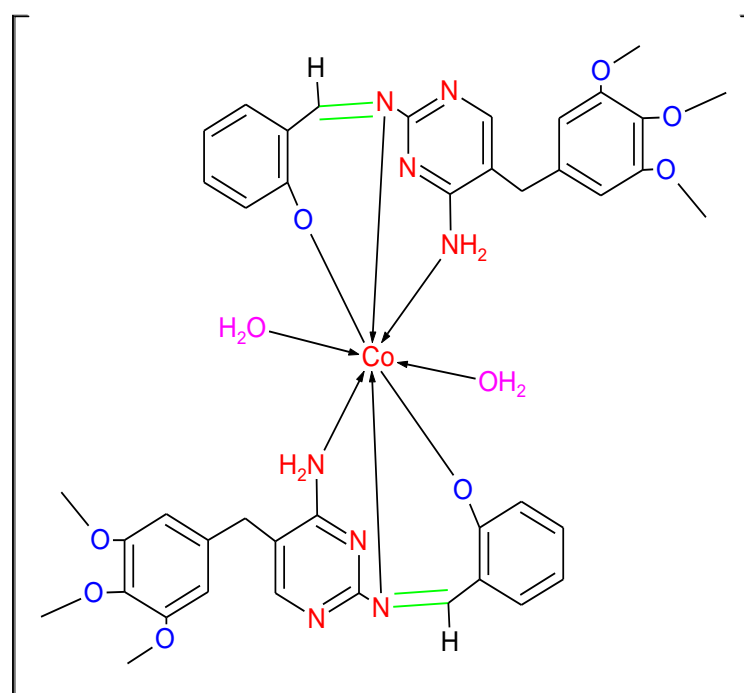


Fig. 2. Structure of $[\text{Co}(\text{C}_{21}\text{H}_{21}\text{N}_4\text{O}_4)_2 \cdot 2\text{H}_2\text{O}]$ complex.

Schiff base /Complexes	% yield	MP/DP °C	C%	N%	M%	BM.µeff
C ₂₁ H ₂₂ N ₄ O ₄	88%	272-275	63.95	14.2		
[Mn (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	73%	235-238	56.13	12.47	12.13	5.52
[Co (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	71%	225-227	55.63	12.36	13.01	4.42
[Ni (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	74%	232-235	55.66	12.35	12.95	3.18
[Cu (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	71%	221-224	55.07	12.23	13.88	1.92
[Zn (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	78%	223-226	54.85	12.18	14.22	dimag
[Pd (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	72%	218-221	50.36	11.19	21.25	
[Pt (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	77%	215-218	42.79	9.51	33.09	

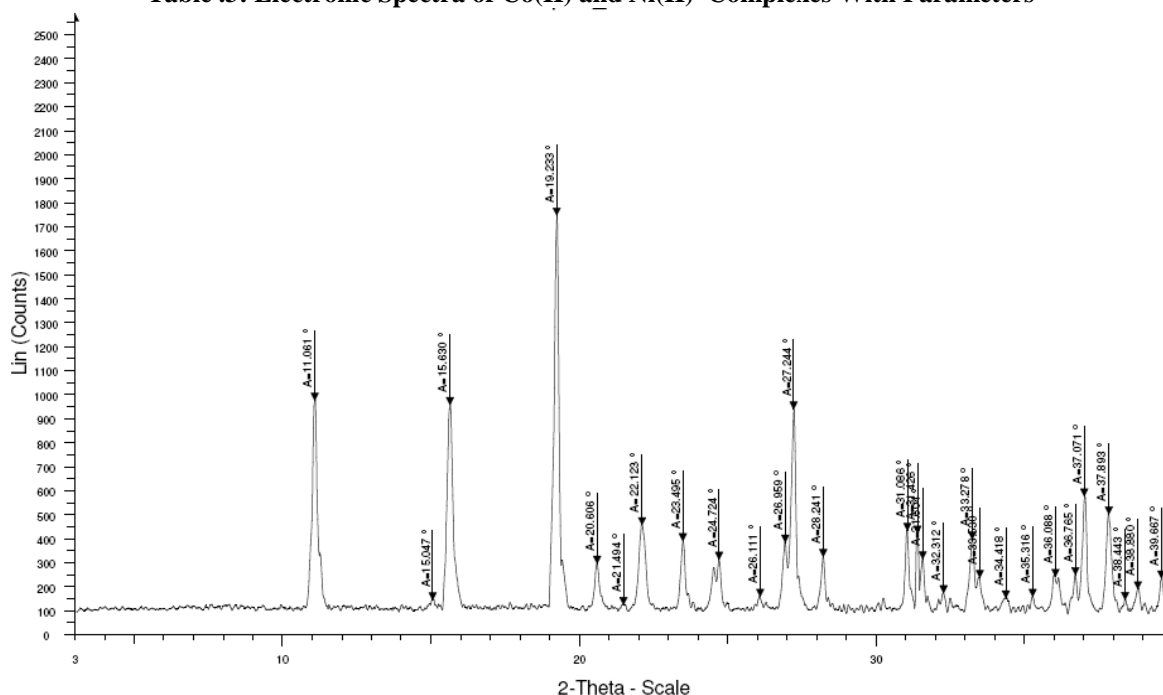
Table .1: Analytical data and Physical Data of Schiff Base and Its Complexes

Schiff base /Complexes	νC-OH	νC=N	νC-NH ₂	νM-N	νM-O	2H ₂ O
C ₂₁ H ₂₂ N ₄ O ₄	3420	1690	3320	-	-	-
[Mn (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	-	1625	3204	515	477	3435,3558
[Co (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	-	1630	3172	518	465	3447, 3542
[Ni (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	-	1632	3188	523	475	3428, 3530
[Cu (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	-	1628	3194	530	480	3442, 3540
[Zn (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	-	1638	3170	505	470	3425, 3545
[Pd (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	-	1620	3203	512	466	3432, 3490
[Pt (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	-	1640	3210	518	460	3438, 3510

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 ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	26915 15245 11242	³ A _{2g} → ³ T _{1g} (P) ³ A _{2g} → ³ T _{1g} (F) ³ A _{2g} → ³ T _{2g}	β=0.9754 δ ⁰ _{0-1.2712} b ^{1/2} = 0.0784 η=0.012
[Cu (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	15245 12735	⁴ T _{1g} → ⁴ A _{2g} (P) ⁴ T _{1g} → ⁴ T _{2g}	β=0.9658 δ ⁰ _{0-1.7912} b ^{1/2} = 0.09245 η=0.0176

Table .3: Electronic Spectra of Co(II) and Ni(II) Complexes With Parameters



SCB 2 Cuplex_21 Jun 2010 - File: SCB 2 Cuplex_21 Jun 2010.raw - Type: 2Th/Th locked - Start: 3.000 ° - End: 40.001 ° - Step: 0.020 ° - Step time: 38.3 s - Temp.: 25 °C (Room) - Time Started: 14 s - 2-Th

Fig;3, X-Ray Powder Data of [Pd (C₂₁H₂₁N₄O₄)₂.2H₂O]

Line No	d-spacing A °		% Intensity	Indices			2 Theta Deg.		Diff.
	Obs.	Calc.		h	k	l	Obs.	Calc.	
1.	7.9922	8.1187	55.8	1	0	0	11.06	10.89	.173
2.	5.8828	5.9093	8.0	2	0	0	15.05	14.98	.068
3.	4.6109	4.6068	100	-1	0	1	15.63	15.84	-.207
4.	4.3066	4.3088	10.6	2	0	0	19.23	19.25	-.017

5.	4.1307	4.1204	8.9	2	1	0	20.61	20.60	.011
6.	4.0146	4.0594	26	-2	0	1	21.49	21.55	-.054
7.	3.7832	3.8049	22.2	-3	1	1	22.12	21.88	.247
8.	3.5979	3.5545	17.7	4	0	0	23.50	23.36	.136
9.	3.4098	3.3794	21.8	1	1	1	24.72	25.03	-.306
10.	3.3044	3.3460	53.6	0	1	1	26.11	26.35	-.239
11.	3.2705	3.2860	18.4	4	1	0	26.96	26.62	-.154
12.	3.1573	3.1469	24.6	0	2	0	27.24	27.11	.131
13.	2.9677	2.9547	24	4	0	0	28.24	28.34	-.095
14.	2.8442	2.8509	9.7	-3	0	2	30.09	30.22	-.136
15.	2.7682	2.7777	22.5	-2	0	2	31.43	31.35	.076
16.	2.6900	2.7062	13.3	0	2	1	32.31	32.20	.114
17.	2.6704	2.6754	8.5	-2	1	2	33.28	33.07	-.188
18.	2.6035	2.6121	8.8	4	2	0	33.53	33.47	.064
19.	2.5393	2.5402	10.5	5	1	0	34.42	34.30	.117
20.	2.4867	2.5005	13.6	-6	1	1	36.09	35.88	.205

Table : 4, X-Ray Powder Data of [Pd (C₂₁H₂₁N₄O₄)₂.2H₂O]

Schiff base /Complexes	Expected m/z	Found m/z	Peak assigned
C ₂₁ H ₂₂ N ₄ O ₄	392.424	391.2	M
[Mn (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	449.362	450.1	M+1
[Co (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	453.357	455.2	M+2
[Ni (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	453.117	455.7	M+2
[Cu (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	457.97	458.7	M
[Zn (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	459.833	461.9	M+2
[Pd (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	500.844	502.5	M+2
[Pt (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	589.502	591.1	M+1

Table: 5. Mass spectral data of the schiff base and complexes.

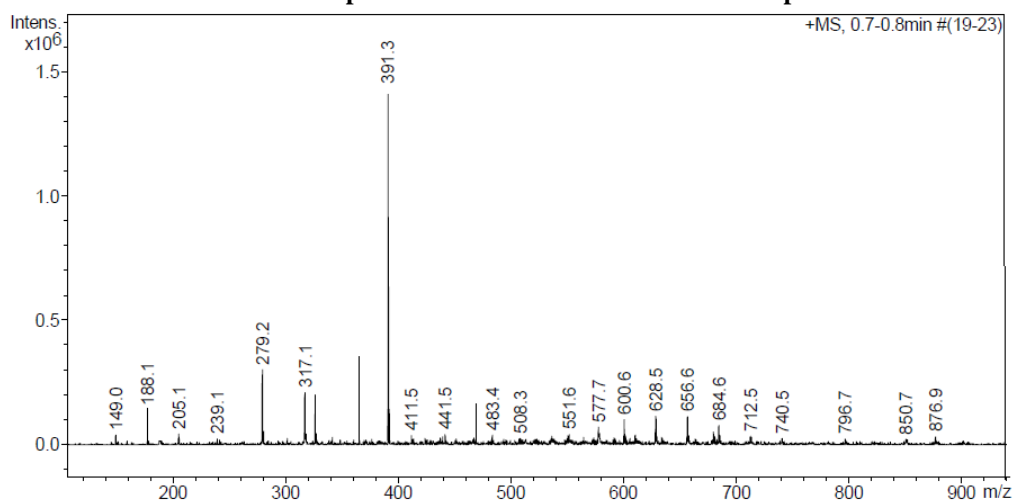


Fig: 4. Mass Spectral Data Of The Schiff Base

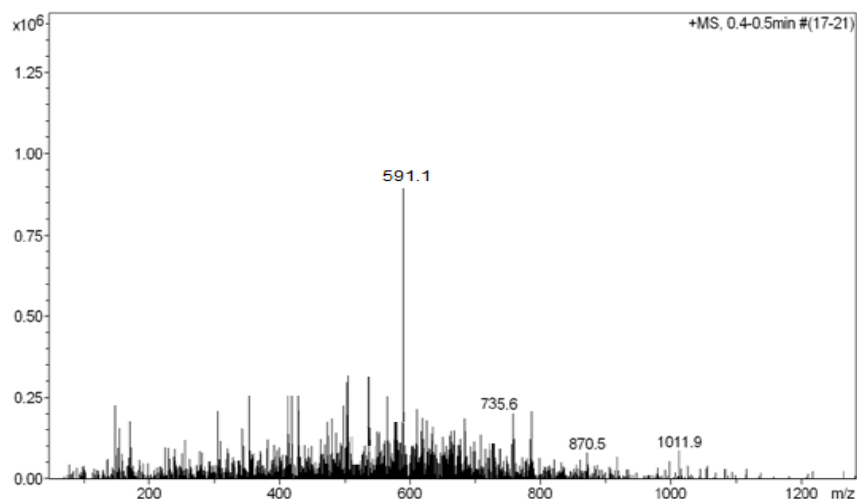


Fig: 5. Mass Spectral Data of [Pt (C₂₁H₂₁N₄O₄)₂.2H₂O]Complex

Schiff base /Complexes	E.Coli	S.Aureus	A.Nigar	F.Oxysporum
C ₂₁ H ₂₂ N ₄ O ₄	8	10	11	16
[Mn (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	12	14	17	25
[Co (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	11	15	15	22
[Ni (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	13	14	18	27
[Cu (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	12	14	16	25
[Zn (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	13	13	14	28
[Pd (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	12	15	17	27
[Pt (C ₂₁ H ₂₁ N ₄ O ₄) ₂ .2H ₂ O]	11	15	17	25

Table 6: For Antimicrobial Activity Of Schiff Base And Complexes

IV. Conclusion:

The schiff base and its metal complexes of MnCl₂, CoCl₂, NiCl₂, CuCl₂, ZnCl₂, PdCl₂ and PtCl₂ have been structurally characterized. The analytical data show that the metal- ligand ratio of stoichiometric in all these complexes is 1:2. Complexes are non-electrolytes in nature due to chloride ion are absent. The spectral data show that the ligand act as neutral and bidentate coordinating through oxygen atoms of hydroxyl group, nitrogen atom of the anilino and azomethane group and of the 5-({4-amino-2-[(Z)-(2-hydroxybenzylidene) amino] pyrimidin-5-yl}methyl)-2,3,4-trimethoxybenzene respectively. Based on analytical, molar conductance, magnetic and spectral data all these complexes are octahedral geometry with coordination number eight. The antimicrobial results also indicate that, the metal complexes are better antimicrobial agents as compared to the Schiff bases. We are proposed the following probable structure of the complex [M(C₂₁H₂₁N₄O₄)₂. 2H₂O].

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