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X-ray diffraction studies of Cu (II), Co (II), Fe (II) complexes with (RS)-4-(7-chloro-4-quinolyl amino) pentyldiethylamine diphosphate

Jitendra H. Deshmukh*[1] and M. N. Deshpande[2,]

[1]*Department of Chemistry, Yeshwant Mahavidyalaya, Nanded*
[2]*P.G. Department of Chemistry, NES, Science College, Nanded*

ABSTRACT

(RS)-4-(7-chloro-4-quinolyl amino) pentyldiethylamine diphosphate (CPH2) was used to synthesize Cu (II), Co (II) Fe (II) complexes. Metal complexes were characterized by elemental analysis IR, TGA.The crystal structure of complexes were further determined by X-ray diffraction method. The XRD data was used to index the compound for octahedral and tetrahedral system.

Keywords: Crystal structure, CPH2, Cu (II), Co (II) and Fe (II) complexes.

INTRODUCTION

Poly functionally rings compounds and synthesis of their metal complexes which have various biological activities and include hetero atom, have been formed in organic synthesis and coordination chemistry[1-5].Many transition metal complexes have been synthesized for analytical and commercial applications[6-7].Literature survey also reveals that the transition metal complexes generally crystallized with octahedral, tetrahedral geometry[8-10].

EXPERIMENTAL SECTION

All the chemicals used for the preparation of complexes are of BDH and AR grade mark. Metal complexes are synthesized by adding metal salt solution in appropriate solvent to the solution of the ligand. The mixture was refluxed for 3-4 hours. Then the precipitate of metal complexes was obtained. It is filtered, washed & dried in vacuum desiccators. Preparation method is given in details in earlier papers.

RESULT AND DISCUSSION

The results of ESR spectra and X-ray diffractogram of Cu (II), Co (II) Fe (II) complexes with CPH2 were obtained and summarized in following tables. All the reflections have been indexed for h, k, l values using reported literature[11]. By using Braggs equation $n\lambda=2d\sin\theta$ the d values of metal complexes were obtained. From ESR spectra of complexes the value of $g_{\perp}$, $g_{\parallel}$, and g_{av} can be determined. These values are tabulated in the table No.1

Table No.1ESR data of Fe (II), Co (II) and Cu (II) complexes

ESR spectral parameter	$[Fe (CPH2)_3]HPO_4$	$[Co (CPH2)_3] HPO_4$	[Cu (CPH2)2] HPO_4
$g_{\parallel}$	1.83	1.87	1.91
$g_{\perp}$	2.23	2.39	2.38
g_{av}	2.09	2.21	2.23

In case Fe (II) complex the g_{av} value is 2.09 which is less than 2.3. The value indicates the presence of sufficient covalence between the metal ion and donor atom[12.] In case Co (II) complex g_{av} value is found to be 2.21. This value is less than 2.3. It is assignable to the presence of covalent character in metal ion and donor atom. In case of Cu (II) complex g_{av} value is found to be 2.23. This value is less than 2.3 indicate the presence of covalent character in co-coordinated bond.

X-Ray Diffraction Study

X-ray diffraction of Fe (II) Complex: The X-ray diffraction pattern for Fe (II), Co (II) and Cu (II) complexes has been determined between 2θ range from 10^0-80^0 and data has been summarized in the following tables.

Table No. 2: Cell data and crystal parameters for $[Fe (CPH2)_3] HPO_4$ complex

a (A^0) = 21.762140 ± 0.021348
b (A^0) = 23.427150 ± 0.104918
c (A^0) = 27.591390 ± 0.21393
Standard deviation = 0.0034441%
= 0.34%
α=90° β=89.3° γ = 90°

Volume $(A^0)^3$ = 14065.86
D_{cal} = 1.0484 g/cm^3
D_{obs} = 1.0896 g/cm^3
Z =8
Crystal system = Monoclinic
Porosity (%) =3.78

I/Io	D_{obs}	D_{cal}	h	k	l
35	4.086196	4.079652	5	2	0
46	3.845215	3.837866	5	4	-2
56	3.631565	3.626787	6	0	0
98	3.310913	3.315434	5	3	4
89	3.190592	3.192186	6	3	2
91	2.975097	2.975549	4	5	5
100	2.597885	2.596681	8	2	2
84	2.411530	2.417858	9	0	0
69	2.044857	2.048483	9	6	1
75	1.908849	1.906651	10	4	5
78	1.653265	1.651666	10	5	9
73	1.450272	1.451837	10	10	-8

From the cell data and crystal lattice one can conclude that Fe (II) complex is having monoclinic crystal system.

Table No.3 Cell data and crystal parameters for [Co (CPH2) $_3$] HPO_4 complex

a (A^0) = 21.726610 ± 0.043441
b (A^0) = 23.251750 ± 0.131456
c (A^0) = 27.564500 ± 0.236385
Standard deviation = 0.0051651
= 0.5%
α=90° β=89.8° γ =90°

Volume $(A^0)^3$ = 13925.04
D_{cal} =1.0619 g/ cm^3
D_{obs} =1.0987 g/ cm^3
Z = 8
Crystal system = Monoclinic
Porosity (%) = 3.34.

I/Io	D_{obs}	D_{cal}	h	k	l
45	4.088381	4.070295	5	2	0
76	3.440960	3.457274	6	2	0
76	3.115265	3.120359	5	3	5
86	2.942080	2.940511	5	4	5
69	2.524552	2.524686	8	0	4
63	2.289217	2.287862	9	2	3
87	1.471531	1.411477	10	10	7
97	1.976285	1.974943	9	0	8
100	1.821989	1.821101	7	7	9
97	1.662780	1.664075	8	8	9
84	2.080324	2.079713	5	5	10

From above data it is clear that Co (II) complex is having monoclinic crystal system.

Table No.4 Cell data and crystal parameters for [Cu (CPH2)] Cl_2 complex

a (A^0) = 21.689170 ± 0.012974
b (A^0) = 23.189140 ± 0.040350
c (A^0) = 27.605530 ± 0.065766
Standard deviation = 0.0028465
= 0.28%
α=90° β=90° γ =90°

Volume $(A^0)^3$ = 13884.29
D_{cal} = 1.5274 g/ cm^3
D_{obs} = 1.6525 g/cm^3
Z = 16
Crystal system = Orthorhombic
Porosity (%) =10.46

I/Io	D_{obs}	D_{cal}	h	k	l
30	4.288056	4.285252	5	0	1
19	3.902708	3.897474	5	2	2
14	3.735246	3.747842	5	3	1
16	3.353157	3.347970	6	2	2
100	3.043481	3.045405	5	2	6
88	2.975110	2.975966	7	2	1
50	2.623335	2.619574	7	4	3
32	2.478043	2.480974	7	5	3
31	2.411530	2.409908	9	0	0
21	2.328537	2.331284	9	0	3
21	2.145296	2.144476	4	9	5
20	1.882965	1.883424	7	5	10
27	1.702111	1.701530	5	10	9
26	1.486129	1.485381	8	10	10

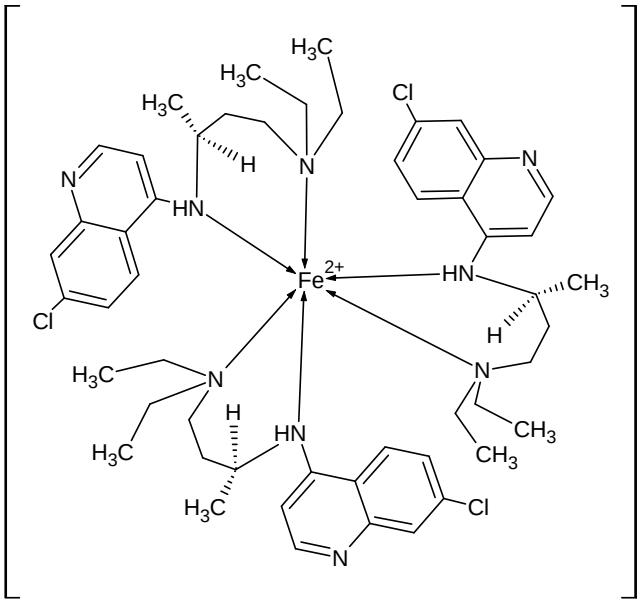
Cell data and crystal lattice parameter of Cu (II) indicates complex attributed to orthorhombic crystal system.

CONCLUSION

On the basis of magnetic moment, crystal lattice parameters and spectral data complexes of Fe (II), Co (II) exhibit octahedral structure were as Cu (II) complex exhibit tetrahedral structure.

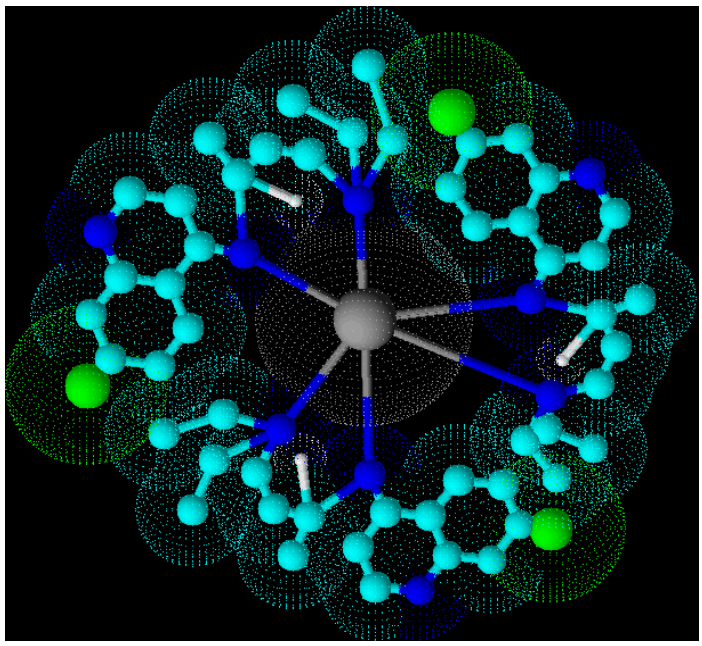
The proposed structure for Fe (II), Co (II) and Cu (II) complexes are as shown in the following figures.

Proposed structure of [Fe (CPH2) $_3$] HPO_4 complex



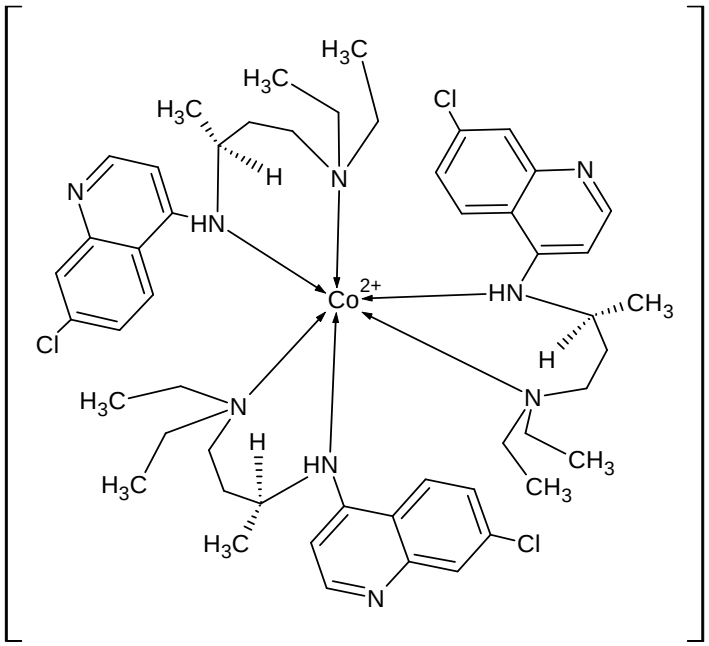
HPO_4

Molecular formula : $C_{54}H_{78}Cl_3N_9Fe\ HPO_4$ Mol. wt = 1110.5



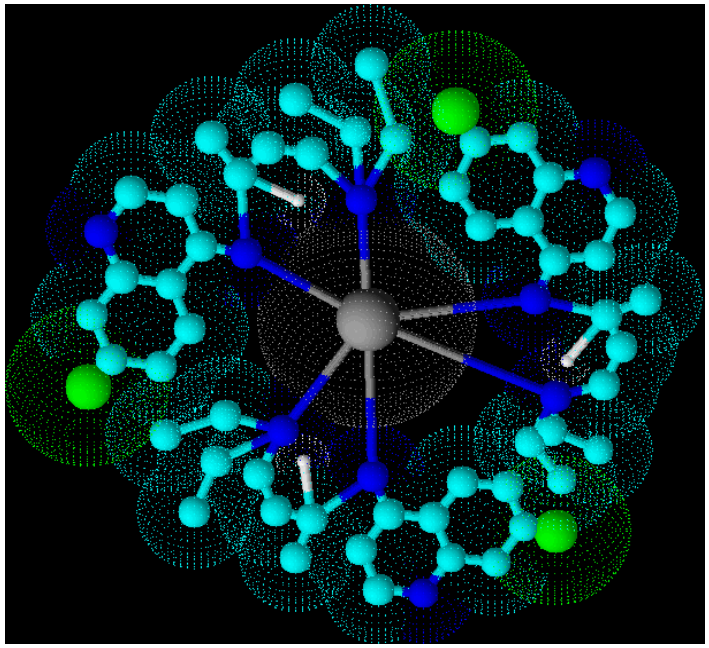
3D view of [Fe (CPH2) $_3$] HPO_4 complex

Proposed structure Co [CPH2]$_3$ HPO$_4$ complex



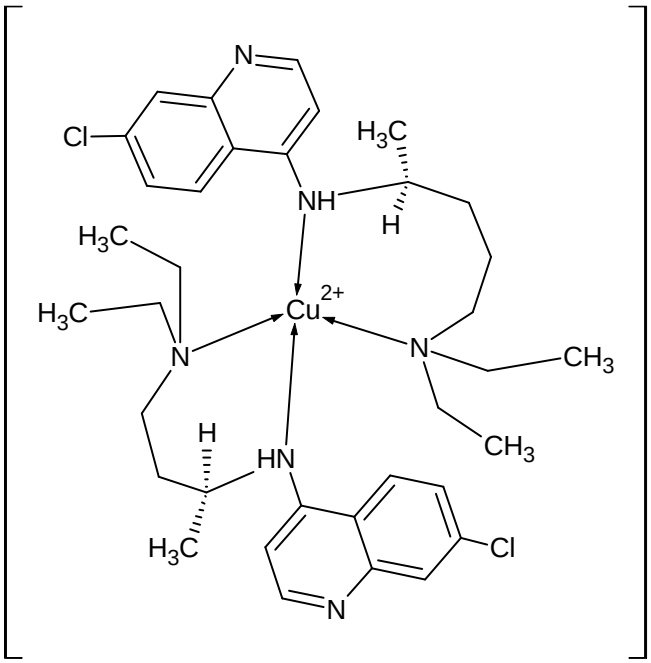
HPO_4

Molecular formula : $C_{54}H_{78}Cl_3N_9Co\ HPO_4$ Mol. wt = 1113.5



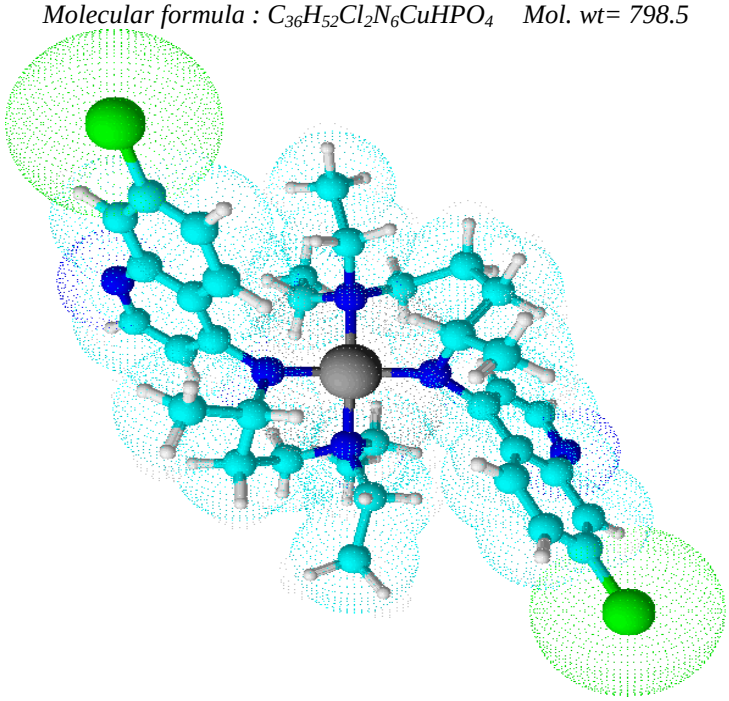
3D view of Co [CPH2]$_3$ HPO$_4$ complex

Proposed structure of [Cu (CPH2)$_2$] HPO$_4$ complex



HPO$_4$

Molecular formula : $C_{36}H_{52}Cl_2N_6CuHPO_4$ Mol. wt= 798.5



3D view of Cu [CPH2]$_2$ HPO$_4$ complex

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