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SYNTHESIS, SPECTROSCOPIC INVESTIGATION AND BIOLOGICAL STUDIES OF P-TOULIC HYDRAZIDE AZOMETHINE METAL COMPLEXES

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ABSTRACT

Two new Schiff base complexes derived from Cu(II) andLa(III) with p-ToulicHydrazide and 2-Hydroxy Benzaldehyde was prepared and investigated using some elemental analysis such as infrared, nuclear magnetic resonance, UV,ESR spectroscopy, molar conductivity measurements, thermo gravimetric analysis, magnetic moments and Powder XRD . The elemental analysis for C, H, and N showed the formation of 1:2 [M:L] complexes. The molar conductivity measurements revealed that the complexes are nonelectrolyte in nature. The thermogravimetric analysis data exhibited the presence of coordinated and hydrated water molecules. The magnetic moment results showed paramagnetic phenomena for Copper and Lanthanum complexes. The infrared spectral data of the prepared complexes displayed the proper coordination sites of the present Schiff base towards the metal ions. The electronic absorption spectral data of the prepared complexes confirmed the electronic transitions and the chemical structures. All the complexes are non-electrolytic in nature as suggested by molar conductance measurements. In addition the authors have been screened the compounds for biological activity. It was found that the compounds have shown activity against the organisms like Escherichia coli, Bacillus *subtills* and Enterococcus faecails.

INTRODUCATION

Although there are wide applications of Schiff bases and their metal chelates in biological systems,[1,2]cataly-sis,[3,4]dying processes,[5,6]and analytical applications[7].Hydrazones, RR'C=N–NR''R''', are used as inter-mediates in synthesis[8], as functional groups inmetal carbonyls [9], in organic compounds[10,11]. Which are among others employed in dinuclear catalysts[12]. Furthermore, hydrazones exhibit physiological activities in the treatment of several diseases such as tuberculosis. This activity is attributed to the formation of stable chelate complexes with transition metals which catalyze physiological processes[13,14]. They also act as herbicides, insecticides, nematocides, rodenticides, plant growth regulators, sterilants for houseflies, among other applications[15,16,17]. In analytical chemistry hydrazones find applications as multidentate ligands for transition metals in colorimetric or fluorimetricdeterminations[18]..In the present paper, the synthesis and characterization of the ligand and its complexes with Cu(II), La(III) are being reported.

MATERIALS AND METHODS

Instrumentation:

The percentage compositions of the elements (CHNO) for the compounds were determined using an element analyzer CHNO model Fison EA 1108.The Infrared spectra were recorded as potassium bromide (KBr) discs using a Perkin-Elmer Spectrum 100.The ^{1}H (400Hz) nuclear magnetic resonance spectra were recorded using the ACF200 Bruker Germany Spectrometer. Ultraviolet Spectra were recorded using Perkin-Elmer lab India UV-Vis Spectrometer. The Electron spin resonance spectra were recorded using the JES-FA Series and TG-DTA spectra were recorded using the SPTQ600 PA, Thermo gravimetric analyses of the metal complexes were carried out by using the Perkin Elmer STA 6000 system in thermal analysis center SKU, Anantapur. Melting points were measured on a unmelt capillary melting point apparatus. All materials used in this investigation were purchased from Sigma/Aldrich and AR (Merck). Solvents used were of reagent grade and purified before use by thestandard methods.

Prepation of Schiffbase(OHBAPTH) and their metal complexes:

p-Toluichydrazide and O-Hydroxybenzaldehyde Schiff Base (OHBAPTH):

Equimolar concentrations (0.01 moles) of p-Toluichydrazide and 2-hydroxy benzaldehyde were dissolved in 50 ml of methanol in 250 ml round bottom refluxing flask. Then added a few drops of Con.H_2SO_4. Then the mixture was refluxed for 3 hours on water bath. On cooling the above mixture pale yellow color (OHBAPTH) crystal products were obtained. The compound was recrystallized from methanol. Yield of the compound is 89% and melting point of the newly synthesized azomethine (OHBAPTH) is 190-192 ^{o}c, as shown in Fig.1.

Synthesis of Copper(II) and Lanthanum(III) metal complexes

2:1 ratio of OHBAPTH Schiff base and metal salts (Cu, La) (0.01 moles) were dissolved separately in 50 ml methanol in 250 ml clean round bottom refluxing flask and refluxed the mixture for half an hour on a water bath. Then 5ml sodium acetate solution was added and refluxing was continued for 6 hours. The reaction mixtures were poured in excess of cold water. Green (OHBAPTH-Cu), yellow (OHBAPTH-La) colored precipitates of metal complexes were obtained with good yield. It was separated by filtration and washed several times with hot water and methanol to free unreacted metal salt ligand respectively and finally with ether and dried in vacuum desiccators.Ligands and metal complexes analytical data was tabulated in Table-1.

Interpretation of OHBAPTH ligand and its metal complexes:

The Infrared spectrum of the OHBAPTH ligand is compared with the spectra of its Cu(II) and La(III) complexes. The important and specific IR spectral frequencies along with their assignments are given in Table.2. The IR spectra's are shown in Fig.(2-4).

A strong band exhibited at 1647 cm^{-1} in the IR spectrum of the ligand has been assigned to the (C=N) Stretching vibration of the azomethine group. On complexation this band is shifted to 1629$cm^{-1}$and 1624cm^{-1}, for Cu (II) and La(III) complexes respectively[19]. This shift to lower wave numbers supports the participation of the azomethine group of this ligand in binding to the metal ion. The coordination of azomethine nitrogen to the metal atom would be expected to reduce the electron density in the azomethine group and thus cause for a reduction in C=N stretching frequency. Bands appeared at 3620 and 1357 cm^{-1} due to the stretching[20] and bending vibrations of phenolic OH respectively. These bands are disappeared in spectra of complexes indicating the deprotonating of phenolic OH. This is further confirmed by the appearance of new bands in the region 681-690 cm^{-1} and 470-490 cm^{-1}, which are assigned to the stretching frequencies of M-N and M-O of the metal ligand bands[21] respectively for Cu (II) and La(III) complexes.

The IR spectrum of the ligand has shown a band in the region 1415-1548 cm^{-1} due to the C=C stretching vibrations. A weak band observed around 3055 cm^{-1} in ligand and 3015 cm^{-1}, 3020 cm^{-1}complexes could be assigned to the aromatic C-H stretching frequency [22]. A broad band exhibited at 3330 cm^{-1} in the IR spectrum of the ligand due to N-H stretching vibration. On complexation this band shifted to 3325cm^{-1} and 3308 cm^{-1} for Cu (II) and La(III) complexes respectively. The IR spectrum of the ligand has shown a sharp band at 1710 cm^{-1} due to C=O stretching vibration. On complexation this band shifted to 1708 cm^{-1} and 1680 cm^{-1} for Cu (II) and La (III) complexes respectively. These results indicate the formation of complex. The IR spectra of Cu (II) and La (III) complexes exhibited broad bands at 3439 cm^{-1} 3434 cm^{-1}

respectively which can be assigned to the OH stretching vibration of the coordinated water molecules[23]. These results indicate that the ligand coordinate with the metal ion through the azomethine nitrogen and the oxygen of the deprotonated hydroxyl group. The suggested structures of the complexes are given in Fig.17.

Interpretation of NMR spectra of OHBAPTH ligand and its metal complexes:

Fig.(5-7) shows the NMR spectra of the OHBAPTH ligand and its Cu (II) and La(III) complexes. Table.3 contains the important chemical shift values along with their assignments.

A typical NMR Spectra of OHBAPTH ligand was presented in the Fig.4., a singlet observed at 2.42 ppm for ^{1}H NMR spectrum of the OHBAPTH ligand is assigned to the azomethine (H-C=N) proton[24] .The singlet appeared at 0.92 ppm is attributed to the methyl protons of the phenyl ring. A multiplet is observed in the region 6.91-7.75 ppm due[25] to the aromatic protons of phenyl rings. A singlet appeared at 11.39 ppm is attributed to the hydroxyl proton attached to the phenyl ring in the ligand. The singlet appeared at 8.72 ppm due to N-H proton of ligand.

In the ^{1}H NMR spectrum of the OHBAPTH-Cu complex as shown Fig.5., a signal appeared due to azomethine proton has been shifted to 2.52 ppm compared to 2.42 ppm in the case of ligand[26]. This down field shift indicates the deshielding of azomethine proton on coordination through nitrogen atom of azomethine group. The signal observed at 0.92 ppm due to the methyl protons in the ligand is shifted to 0.91 ppm for the Cu complex. The singlet appeared at 11.39 ppm due to phenolic hydroxyl proton is absent in the NMR spectrum of Cu complex indicating the deprotonation of hydroxyl group and the involvement of that oxygen in coordination. A new signal is observed as a singlet at4.23 ppm in the case of Cu (II) complex indicating the presence of water molecules coordinated to the metal atom. The multiplet observed in the region 6.91-7.75 ppm due to aromatic protons for the ligand showed a shift to 6.32-7.74 ppm for Cu complex may be due to the drifting of ring of electrons towards the metal ion.

In the ^{1}H NMR spectrum of the OHBAPTH-La complex as shown Fig.6., a signal appeared due to azomethine proton has been shifted to 2.82 ppm compared to 2.42 ppm in the case of ligand[26]. This down field shift indicates the deshielding of azomethine proton on coordination through nitrogen atom of azomethine group. The signal observed at 0.92 ppm due to the methyl protons in the ligand is shifted to 0.94 ppm for the La complex. The singlet appeared at 11.39 ppm due to phenolic hydroxyl proton is absent in the NMR spectrum of La complex indicating the deprotonation of hydroxyl group and the involvement of that oxygen in coordination[27]. A new signal is observed as a singlet at 4.39 ppm in the case of La (III) complex indicating the presence of water molecules coordinated to the metal atom. The multiplet observed in the region 6.91-7.75

ppm due to aromatic protons for the ligand showed a shift to 6.26-7.78 ppm for La complex may be due to the drifting of ring of electrons towards the metal ion.

Study of Copper and Lanthanam complexes through UV Spectrophotometer:

Cu (II) and La(III) complexes of Schiff base compounds formed between p-ToluicHydrazide with different ketones and aldehydes like OHBAPTH was prepared and characterized by employing several physico-chemical spectral methods. It was observed that these metal complexes were stable in room temperature like any other Schiff base complexes. All these metal chelates are easily soluble in DMF, and are sparingly soluble in alkaline medium.

Analysis of OHBAPTH ligand and its metal complexes:

The electronic spectra of the aqueous solutions of $Cu^{+2}$and La^{+3}individual ions are compared with the corresponding ligand nature. The data is given in Table.4.and Fig.(8-10). The data indicates that the energy of the d-d transitions in the complexes is slightly less when compared to the corresponding aqua ions either because of slight covalent interaction of the 3d vacant orbitals with ligands, leading to some delocalization with consequent reduction in inter electronic repulsion, or by increased nuclear shielding of the orbitals due to slight covalent ligand-metal electron drift. The transition for the ligand occurred at 295 nm. But on complexation with the different metal ions like Copper and Lanthanum new bands appeared at 320 nm and 336 nm respectively corresponding to the transitional charge transfer from the ligand to the different metal ions[28]. Bands occurred in the region of 320-336 nm for all complexes are assigned to charge transfer transition (L→M). Based on the results octahedral structure is proposed for Cu^{+2} and La^{+3}omplexes.

ESR Spectral studies of OHBAPTH-Cu complex:

ESR spectra of Cu metal complexes give useful information regarding the stereochemistry and nature of metal–ligand bonding. Only one broad signal is exhibited in the ESR spectra of the complexes in polycrystalline state, which is attributed to dipolar broadening and enhanced spin lattice relaxation. Anisotropic spectra obtained for all complexes in DMF at LNT and representative ESR spectra of OHBAPTH-Cu(II) ion complex is presented in Fig.11. In this low temperature spectrum, four peaks of small intensity have been identified which are considered to originate from $g_{\|}$component.

The spin Hamiltonian, orbital reduction and bonding parameters of OHBAPTH-Cu complex are presented in Table.5.

The $g_{\|}$and $g_{\perp}$are computed from the spectrum using DPPH free radical as g marker. Kivelson& Neiman have reported that$g_{\|}$value is less than 0.08 for covalent character and is greater than 2.3

for ionic character of the metal – ligand bond in complexes. Applying this criterion, the covalent bond character can be predicted to exist between the metal and the ligand complexes[29]. The trend $g_{\|} > g_{ave} > g_{\perp} > 2.0023$ observed for the complex suggests that the unpaired electron is localized in $d_{x^2-y^2}$ or d_{z^2} orbital of the Cu (II) ions for the complexes. The G value for Copper complex is given in table.5. It is observed that the G value of present complex is greater than four and suggest that there are no interactions between Copper-Copper[30] centers in DMF medium.

The ESR parameters $g_{\|}$, $g_{\perp}$, $A_{\|}^{*}$ and $A_{\perp}^{*}$ of the complexes and the energies of d-d transitions are used to evaluate the orbital reduction parameters ($K_{\|}$,$K_{\perp}$), the bonding parameters (α^2), the dipolar interaction (P). The observed $K_{\|} < K_{\perp}$indicates the presence of out of plane π bonding. The α^2 value for the present chelate is 0.552. It indicates that the complex have covalent character. This shows an appreciable covalence in the inplane 'σ' bonding. Giordano and Bereman suggested the identification of bonding groups from the values of dipolar term P. The reduction of P values from the free ion value ($0.036cm^{-1}$) might be attributable to the strong covalent bonding. The value of P obtained for the present complex is 0.0152 cm^{-1} and remains consistent with bonding of Copper ions to oxygen and nitrogen donor atoms respectively[31]. The shape of ESR lines, ESR data together with the electronic spectral data suggest an octahedral geometry for OHBAPTH-Cu complex.

ESR Spectral studies of OHBAPTH-La complex:

ESR spectra of La metal complexes give useful information regarding the stereochemistry and nature of metal–ligand bonding. Only one broad signal is exhibited in the ESR spectra of the complexes in polycrystalline state, which is attributed to dipolar broadening and enhanced spin lattice relaxation. Anisotropic spectra obtained for all complexes in DMF at LNT and representative ESR spectra of OHBAPTH-La(III) ion complex is presented in Fig.12. In this low temperature spectrum, four peaks of small intensity have been identified which are considered to originate from $g_{\|}$ component.

The spin Hamiltonian, orbital reduction and bonding parameters of OHBAPTH-La complex are presented in table.5.

The $g_{\|}$and $g_{\perp}$are computed from the spectrum using DPPH free radical as g marker. Kivelson& Neimanhave reported that $g_{\|}$value is less than 0.08 for covalent character and is greater than 2.3 for ionic character of the metal – ligand bond in complexes. Applying this criterion, the covalent bond character can be predicted to exist between the metal and the ligand complexes[29]. The trend $g_{\|} > g_{ave} > g_{\perp} > 2.0023$ observed for the complex suggests that the unpaired electron is localized in $d_{x^2-y^2}$ or d_{z^2} orbital of the La (III) ions for the complexes. The G value for Lanthanum complex is

given in table V.7. It is observed that the G value of present complex is greater than four and suggest that there are no interactions between Lanthanum-Lanthanum[30]centers in DMF medium.

The ESR parameters $g_{\|}$, $g_{\perp}$, $A_{\|}^{*}$ and $A_{\perp}^{*}$ of the complexes and the energies of d-d transitions are used to evaluate the orbital reduction parameters ($K_{\|}$,$K_{\perp}$), the bonding parameters (α^2), the dipolar interaction (P). The observed $K_{\|} < K_{\perp}$indicates the presence of out of plane π bonding. The α^2 value for the present chelate is 0.622. It indicates that the complex have covalent character. This shows an appreciable covalence in the inplane 'σ' bonding. Giordano and Bereman suggested the identification of bonding groups from the values of dipolar term P. The reduction of P values from the free ion value ($0.036cm^{-1}$) might be attributable to the strong covalent bonding. The value of P obtained for the present complex is 0.0167 cm^{-1} and remain consistent with bonding of Lanthanum ions to oxygen and nitrogen donor atoms respectively. The shape of ESR lines, ESR data together with the electronic spectral data suggest an octahedral geometry for OHBAPTH-La complex[31].

Magnetic behavior of ligand and its metal complexes:

The magnetic properties along with spectroscopic properties acquire greater significance in the characterization of metal complexes.

Magnetic Susceptibility Measurements of Copper and Lanthanum complexes:

Cu (II) and La(III) complexes of Schiff base compounds formed p-ToluicHydrazide with 2-Hydroxy benzaldehyde like OHBAPTH was prepared and characterized by employing several physico-chemical spectral methods.

The magnetic susceptibility values are given in table: 6. The Copper complex at room temperature was observed the magnetic momentis 1.85 B.M. This magnetic momentum value indicates the presence of unpaired electrons as expected for Cu (II) complex. The magnetic moment value also revealed that the complex is monomeric in nature and metal-metal interaction along the axial positions is absent. It was observed that there was considerable orbital contribution and effective magnetic moments for octahedral complexes at room temperature around the range 5.0-5.2 B.M for high spin octahedral complexes[32], the magnetic moment was observed 1.85 B.M for Cu (II) complexes. Thermal analysis showed that the Copper complexes involved the loss of two water molecules at about 80-240^{0}C. This suggests that two water molecules coordinated with the central metal ion, which is further confirmed by their characteristic IR spectrum.

The magnetic susceptibility values are given in table .6. The Lanthanum complex at room temperature was observed the magnetic moment is 5.49 B.M. This magnetic momentum value indicates the presence of unpaired electrons as expected for La (III) complex. This magnetic

momentum range suggests an octahedral geometry. Thermal analysis showed that the Lanthanum complex involved the lose of two water molecules at about 110-270^{0}C. This suggests that two water molecules coordinated with the central metal ion, which is further confirmed by their characteristic IR spectrum.

Thermal behavior of Cu (II) and La(III) Metal complexes of OHBAPTH:

The Thermo gravimetric studies of all the complexes were carried out in air at a heating rate of 10^o C per minute. The thermal analysis curves of the complexes are given in Fig.(13-14). The thermo analytical data is summarized in Table.7. The thermal decomposition of the complexes proceeds in three stages. The Cu (II) and La(III) complexes are thermally stable up to 80 and 110^{o}c respectively. The first stage of decomposition corresponding to endothermic dehydration of complexes by the loss of two water molecules occur[33] in the temperature range 70-240^{o}cand 110-270^{o}C respectively. The intermediates formed are stable up to 310^{o}c and 280^{o}c. The second decomposition with exothermic peak by the loss of ligand moiety occursin the temperature range 310-420^{o}cand 280-390^{o}C. The solid residues above 420 and 410 ^{0}C were identified as Cu (II) and La(III) metal oxides respectively. In all the complexes, the final products are metal oxides.

Conductivity Measurements of OHBAPTH metal complexes:

The molar conductance of complexes in DMF (~10^{-3} M) was determined at 27+2^{o}C using systronic 303 reading conductivity bridge Cu (II) and La(III) complexes of azomethine compound formed due to the condensation of 2-Hydroxy Benzaldehyde with P-ToluicHydrazide ligand is prepared. The complex of OHBAPTH ligand is highly soluble in dimethyl formamide (DMF). Therefore these metal chelates are dissolved in DMF to perform conductivity measurements. A known amount of solid complex was transferred into 25 ml standard flask and dissolved in DMF. The contents were made up to the mark with DMF. The complex solution is transferred into a clean and dry 100 ml beaker. The molar conductance values of these metal complexes which are residual are given in Table 8. These values suggest non-electrolytic nature of the present complexes.

Powder XRD study of OHBAPTH-Cu and OHBAPTH-La complexes:

The powder X-ray diffraction data obtained for OHBAPTH-Cu and OHBAPTH-La complexes with difractograms using DROL-2 powder diffractometer. Radiation filled by metal foil. The difractograms (13 diffractions) reflects Fig.15., between 3-50 (2**θ**) values for Cu complex and (15 diffractions) reflects Fig.16., between 3-50 (2**θ**) values for La complex. Where θ is Brages angle all the main peaks are indicted and calculated values of Miller indices (h k l) along with observed d-specified and reveled intensities are specified in the Fig: 15 and 16. All the peaks have been

indexed 2θ values compared in graph. Comparison values revels that there is good agreement between values of 2θ and d-values. The powder x-ray diffraction data showed identical features with very poor crystalinity. The patterns are qualitative and dispersive in intensity for Cu and La complexes. XRD patterns are used to explain qualitatively the degree of crystalinity. X-ray Diffraction data of OHBAPTH-Cu and OHBAPTH-La complexes are presented in table.9 and 10.

Biological activity:

The author in this present investigation attempted to find out antibacterial activity of ligand and their metal complexes against Escherichia coli, Bacillus *subtills*and Enterococcus faecails choosing serial paper disc method Table 11.The results of the biological activity of the metal complexes indicated the following facts. A comparative study of the ligand and their complexes indicates that the metal chelates exhibited higher antibacterial activity than that of the free ligand. The increase in the antibacterial activity of metal chelates was found due to the effect of metal ion on the metal chelates which could be explained on the basis of overtones concept and chelation theory. On chelation the polarity of the metal ion reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of positive charges of metal ion with donor groups. It was further noted that the delocalization of electrons over the whole chelate ring enhanced the lipophillicity of the complexes. This increased lipophillicityenhanced the penetration of the complexes into lipid membrane and blocking the metal sites on enzymes of microorganism**.** The zones of inhibition of the ligand metal complexes were in the Table.11**.**

CONCLUSION

The author again the above information, it can be concluded that Schiff base of 2-Hydroxy Benzaldehyde with alkalamine namely p-ToulicHydrazide acts as a very good complexing agent towards many transition metal ions. By using above spectral studies these behave bidentate during complexation. All the metal complexes carry no charge and are thermally stable. As such no single technique is independent of predicting final structures of the complexes. Hence the entire information available from all the studies is clubbed together and appropriate structures of the complexes under investigation can be formulated as follows.

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Table-1: Analytical data of the ligand and their metal complexes.

			Complex		
			OHBAPTH	**$Cu(OHBAPTH)_2X_2$**	**$La(OHBAPTH)_2X_2$**
Molecular weight			330.40	760.346	835.70
Co lour			Pale yellow	Green	Pale yellow
Yield			89	78	81
M.P			190-192	306-308	246-248
Elemental Analysis	C %	Calculated	70.78	59.19	52.67
		Found	70.69	59.11	52.60
	H%	Calculated	5.50	5.26	4.68
		Found	5.26	5.21	4.57
	N %	Calculated	11.01	9.20	8.19
		Found	11.09	9.09	8.08
	O%	Calculated	14.15	15.78	14.04
		Found	14.06	15.67	14.09
	M%	Calculated	-	10.45	20.32
		Found	-	10.39	20.22

Table.2. Important IR Spectral bands of OHBAPTH ligand and its metal complexes

Compound	□OH Water	□OH Phenolic	□C=N	□N-H	□C=O	□M-O	□M-N
OHBAPTH	-	3620	1647	3330	1710	-	-
OHBAPTH-Cu	3439	-	1629	3325	1708	470	690
OHBAPTH-La	3434	-	1624	3308	1680	490	681

Table.3.^{1}H NMR spectral data of OHBAPTH ligand and its metal complexes

Compound	H-C=N	Ar-H	$-CH_3$	N-H	Ar-OH	H_2O-OH
OHBAPTH	2.42	6.91-7.75	0.92	8.72	11.39	-
OHBAPTH-Cu	2.52	6.32-7.74	0.91	8.35	-	4.23
OHBAPTH-La	2.82	6.26-7.78	0.94	8.32	-	4.39

Table-4. Electronic Spectral data of OHBAPTH ligand and its metal complexes

Compound	$\lambda_{max}$of compound
OHBAPTH	295
OHBAPTH-Cu	320
OHBAPTH-La	336

Table:5. Spin Hamiltonian and orbital reduction parameters of OHBAPTH-Cu complex and OHBAPTH-La in DMF solution

Parameters	OHBAPTH-Cu	OHBAPTH-La
$g_{\parallel}$	2.1765	2.2998
$g_{\perp}$	2.0372	2.0746
g_{ave}	2.0836	2.1493
G	4.7446	4.0187
$A_{\parallel}^{*}$	0.01880	0.01902
$A_{\perp}^{*}$	0.00524	0.00666
A^{*}_{ave}	0.0072	0.01067
d-d	16420	17520
$K_{\parallel}$	0.6160	0.615
$K_{\perp}$	0.757	0.729
P*	0.0152	0.0167
α^2	0.552	0.622

* Values are given as cm^{-1} units.

Table 6.Magnetic moments of Copper and Lanthanum complexes

Complex	Cupper in (B.M)	Lanthanum in (B.M)
$(OHBAPTH)_2$	1.85	5.49

Table 7. Thermal Analytical Data of the OHBAPTH Ligand metal complexes

Complex X=H_2O	Molecular weight in gm	Weight of the complex taken in mg	Temperature range in ^{o}C	Probable assignment	Mass loss (%)	Total mass loss (%)
[Cu L_2.2X] L=$C_{15}H_{14}O_2N_2$	608.146	12.20	80 to 240 310 to 420 Above 420	Loss of $2H_2O$ molecules Loss of 2L molecules Corresponds to CuO	5.9196 81.6580 13.080	70.43
[La L_2.2X] L=$C_{15}H_{14}O_2N_2$	683.50	10.07	110 to 270 280 to 390 Above 410	Loss of $2H_2O$ molecules Loss of 2L molecules Corresponds to LaO	5.267 72.6554 22.6627	92.453

Table:8. Molar conductivity of Cu and La complexes

Metal complexes	Molar conductance(ohm^{-1} cm^2mol^{-1})
OHBAPTH-Cu	6.97
OHBAPTH-La	8.12

Table:9. X-ray Diffraction data of OHBAPTH-Cu complex

S.No.	d expt	d Calc	2θ expt	2θCalc	h k l
1.	18.1562	18.15592	4.863	4.86298	1 1 0
2.	4.4006	4.40012	20.162	20.16197	2 21
3.	3.5446	3.54434	24.959	24.95769	2 32
4.	3.4618	3.46099	25.713	25.71198	2 5 1
5.	3.3475	3.34700	26.607	26.60591	3 1 0
6.	3.0611	3.06099	29.149	29.14569	4 4 1
7.	2.5349	2.53472	35.381	35.37981	6 2 1
8.	2.1410	2.14098	42.174	42.17128	943
9.	2.0188	2.018791	44.861	44.85912	9 6 1
10.	1.9396	1.93975	46.798	46.79799	9 8 7
11.	1.8300	1.82997	49.785	49.78345	10 2 1

Table:10. X-ray Diffraction data of OHBAPTH-La complex

S.No.	d expt	d Calc	2θ expt	2θCalc	h k l
1.	15.055	15.05482	5.866	5.86594	321
2.	10.934	10.93371	8.080	8.08122	421
3.	7.517	7.51639	11.764	11.76398	532
4.	5.483	5.48202	16.155	16.15479	621
5.	4.760	4.75951	18.628	18.62791	630
6.	4.182	4.18164	21.230	21.22941	743
7.	3.827	3.82652	22.326	23.32569	841
8.	3.636	3.63562	24.464	24.46398	943
9.	3.391	3.38957	26.271	26.27019	9 3 1
10.	3.092	3.09101	28.864	28.86395	947
11.	2.884	2.88332	30.990	30.98792	985
12.	2.572	2.57102	34.870	34.86871	10 1 0
13.	1.967	1.96685	46.112	46.11126	10 2 1

Table:11. Antibacterial activities of ligands and their transition metal complexes (Zone formation in mm)

Compound	Escherichia *coli*	Bacillus *subtills*	Enterococcus *faecails*
OHBAPTH	**8**	**11**	**14**
$(OHBAPTH)_2$ Cu	**10**	**9**	**11**
$(OHBAPTH)_2$La	**12**	**14**	**10**

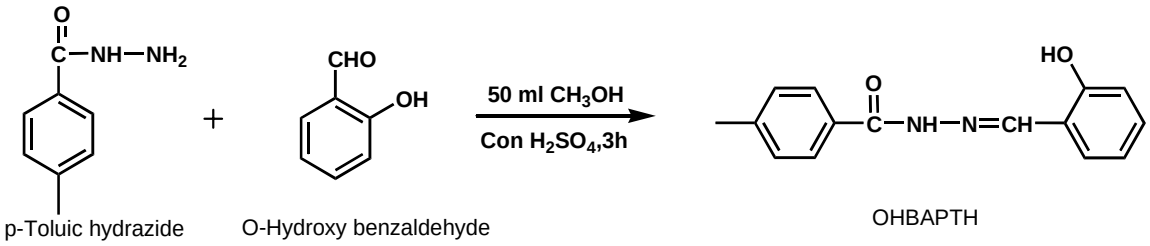


Fig.1.Prepartion of OHBAPTH Ligand

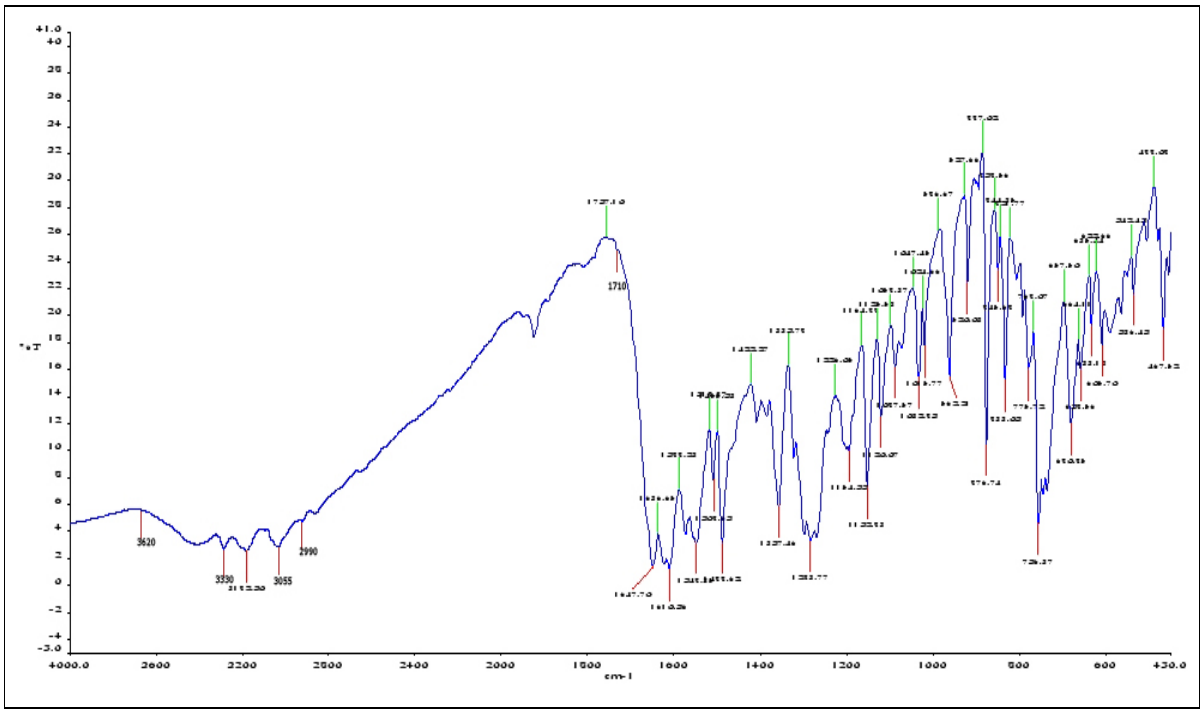


Fig.2.:IR Spectra of OHBAPTH Ligand

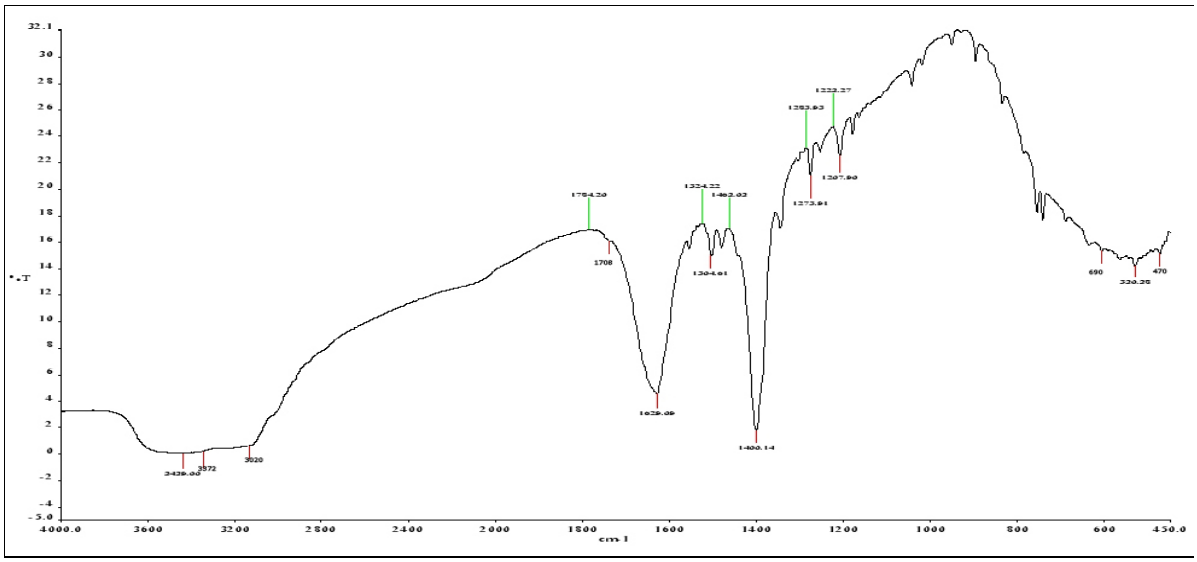


Fig .3: IR Spectra of OHBAPTH-Cu Complex

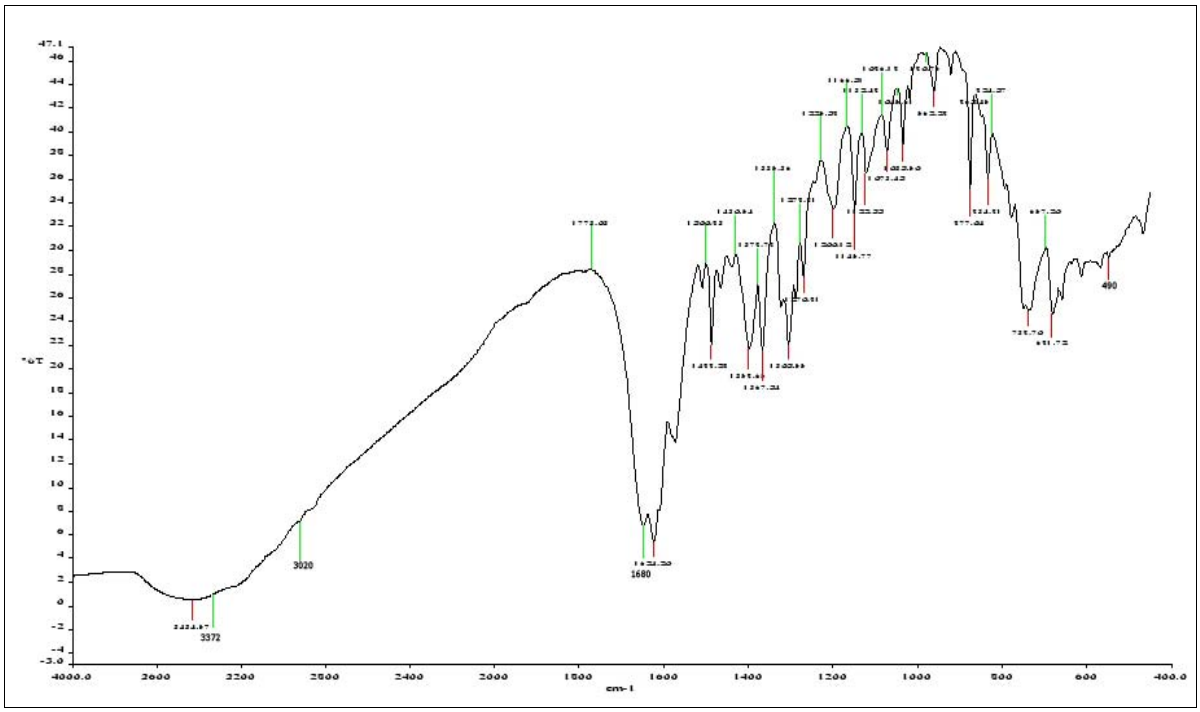


Fig.4: IR Spectra of OHBAPTH-La Complex

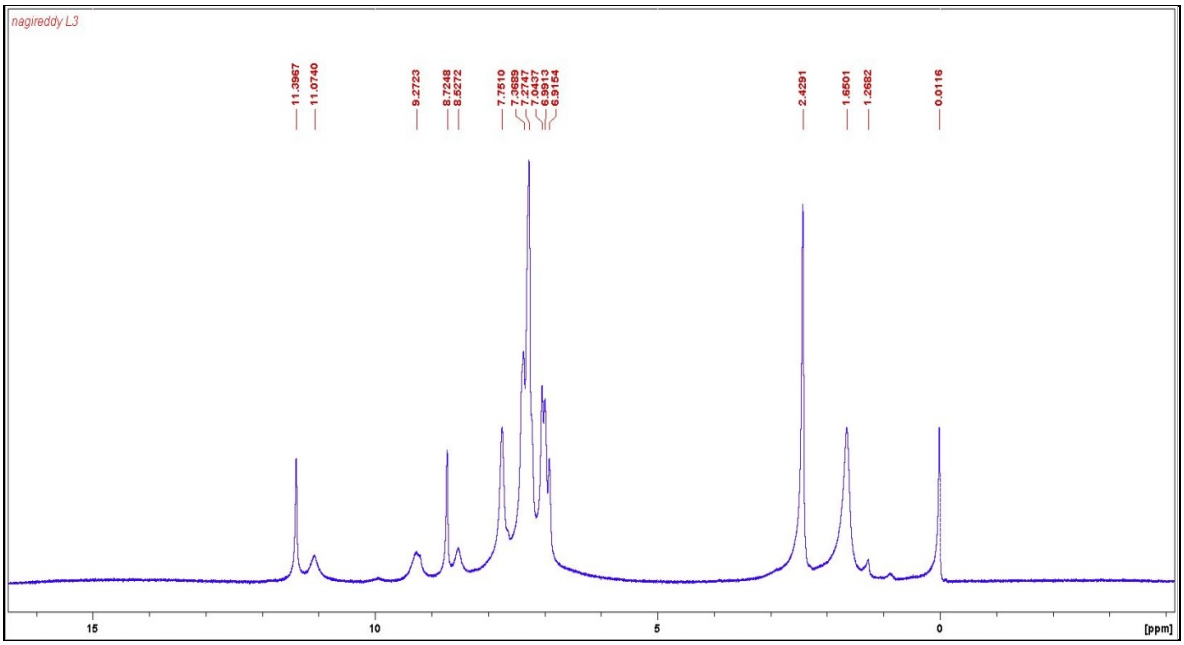


Fig.5. NMR Spectra of OHBAPTH Ligand

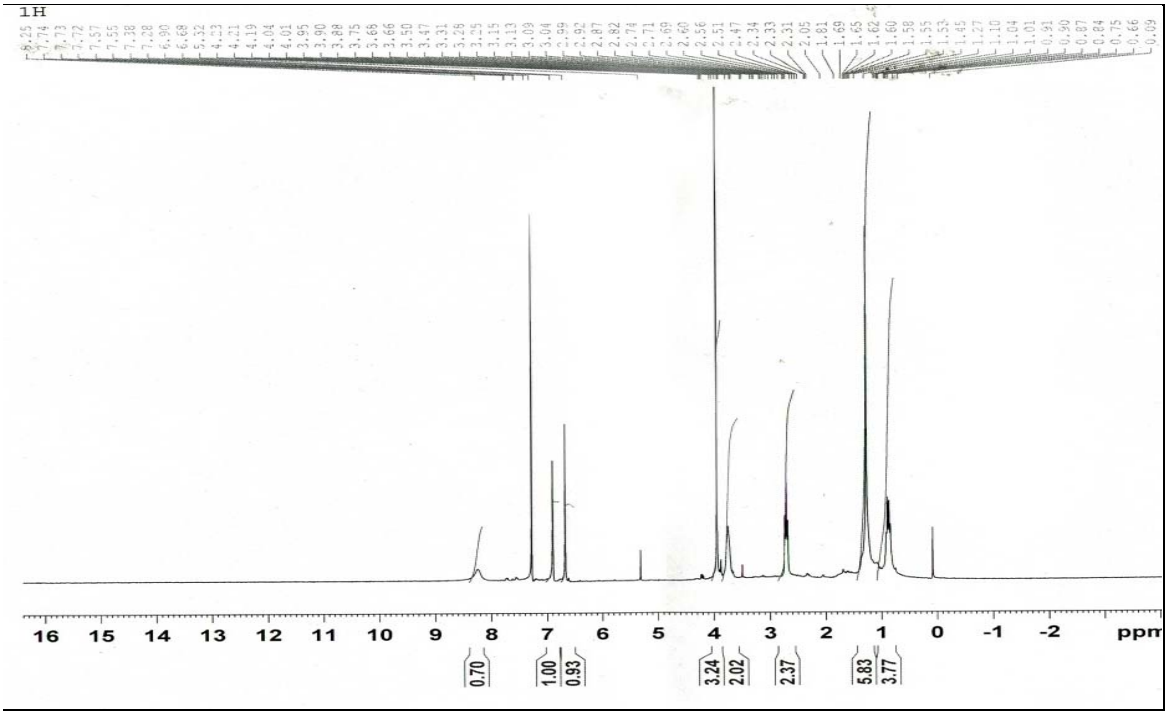


Fig.6. NMR Spectra of OHBAPTH-CuComplex

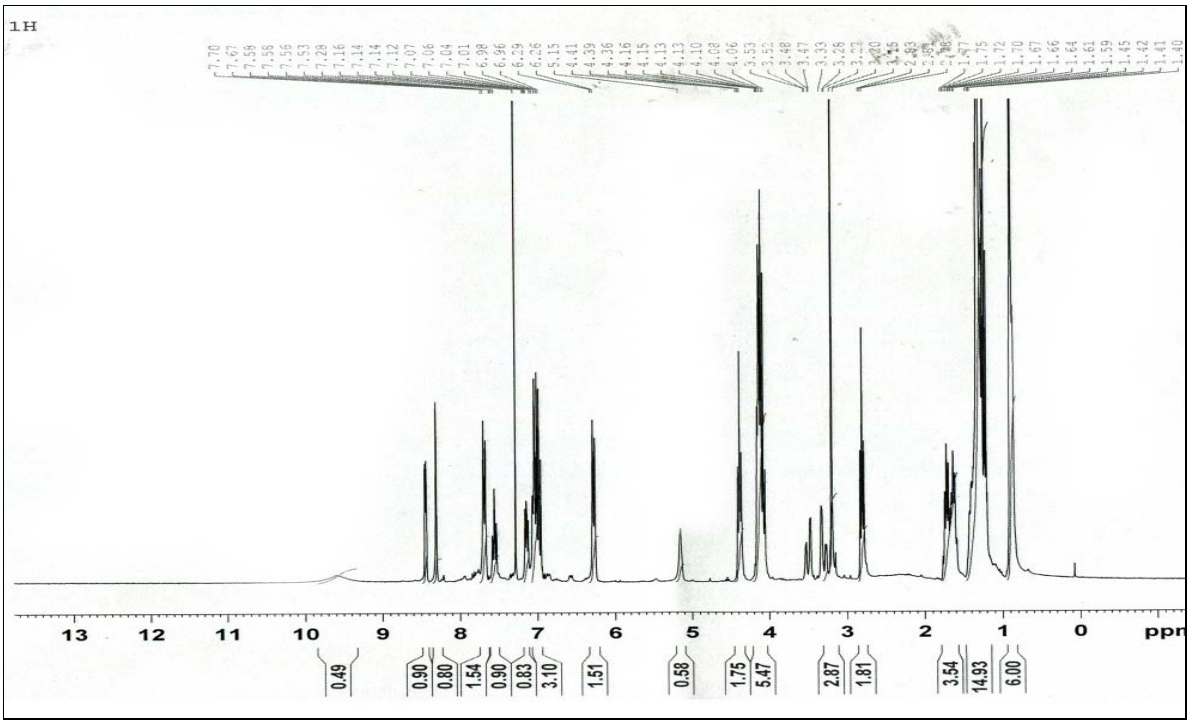


Fig.7. NMR Spectra of OHBAPTH-La Complex

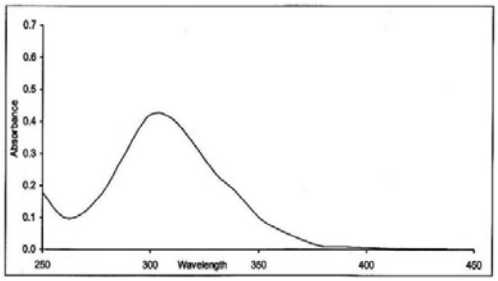


Fig.8 : UV spectraof OHBAPTH Ligand

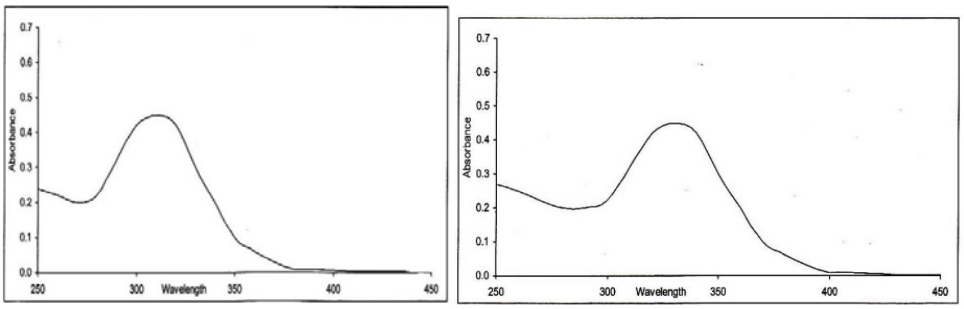


Fig.9&10 : UV Spectraof OHBAPTH-Cu & OHBAPTH-La complexes

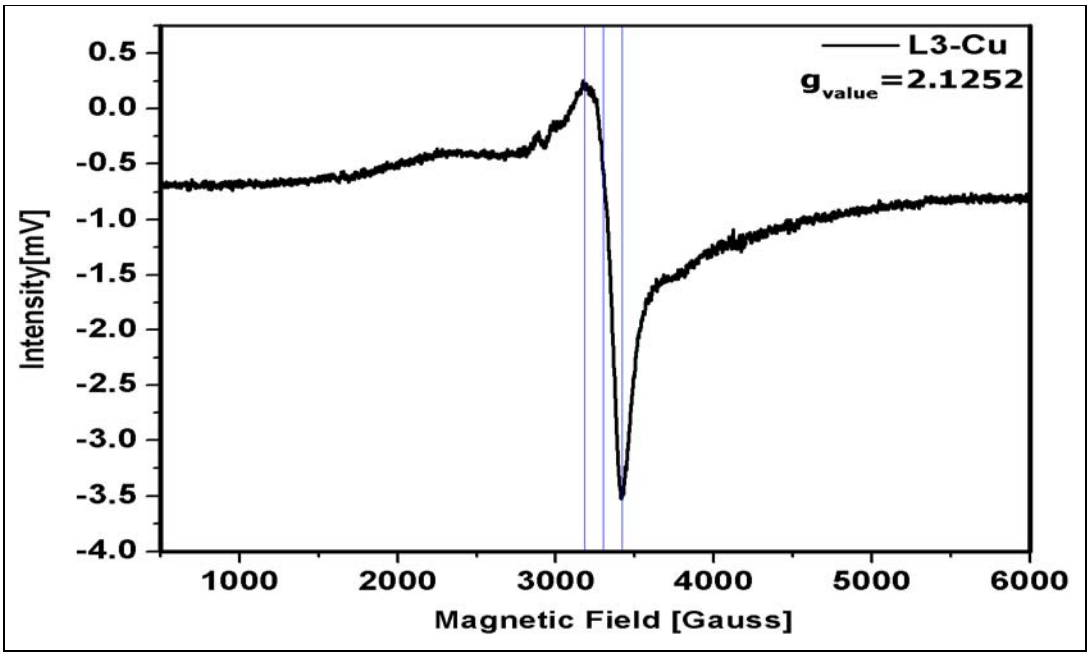


Fig.11: ESR Spectra of OHBAPTH-Cu complex

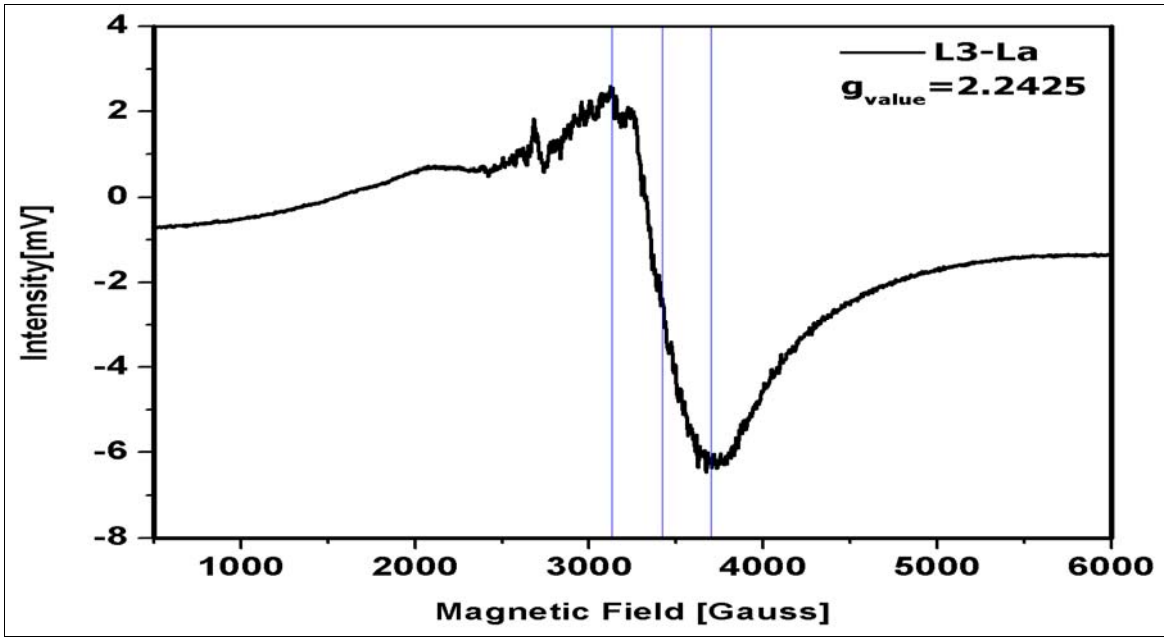


Fig.12: ESR Spectra of OHBAPTH-La complex

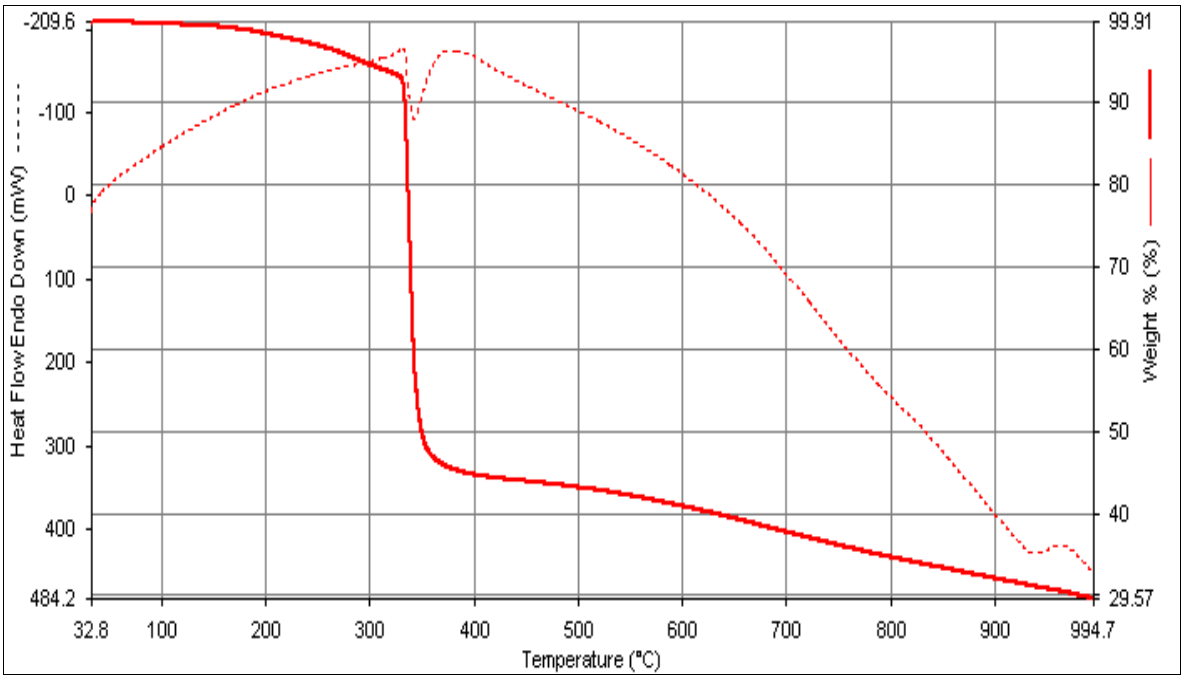


Fig.13: TG/ DTA of OHBAPTH-Cu complex

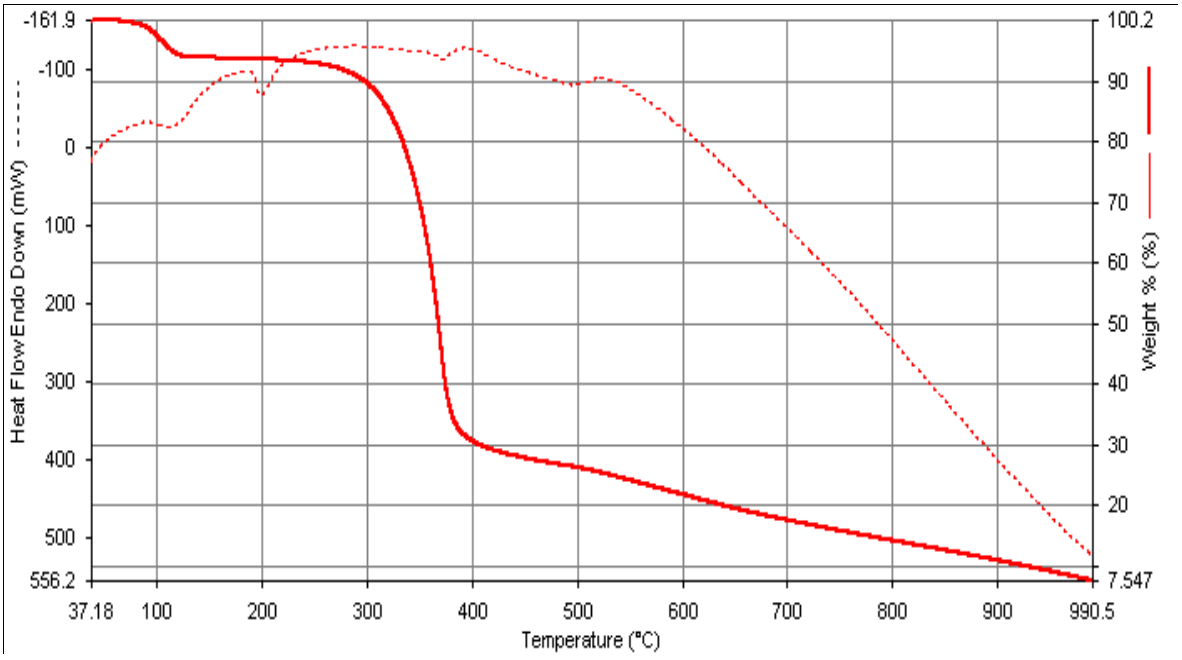


Fig.14: TG/ DTA ofOHBAPTH-La complex

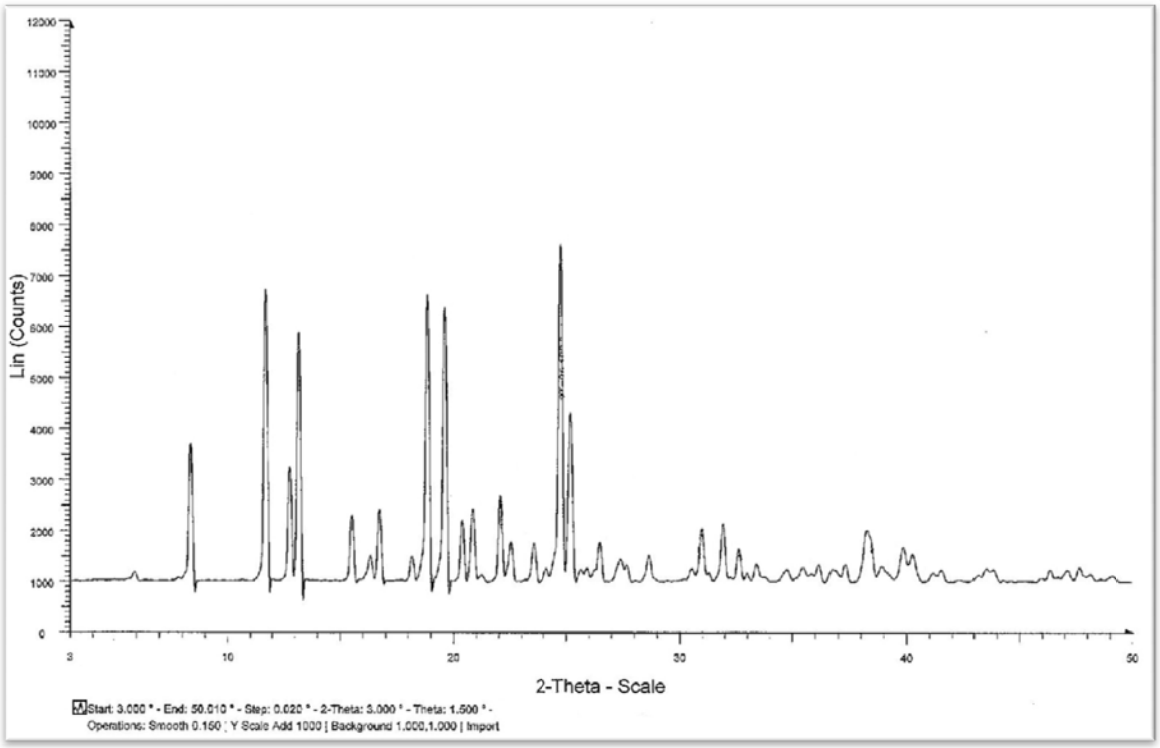


Fig. 15. Powder XRD of OHBAPTH-Cu complex

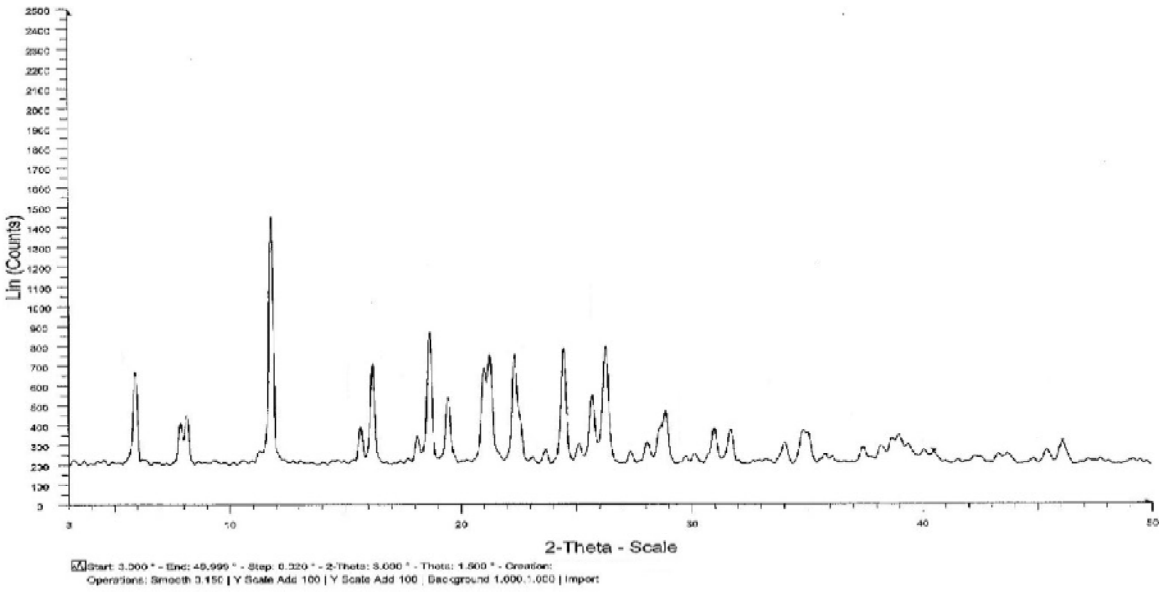
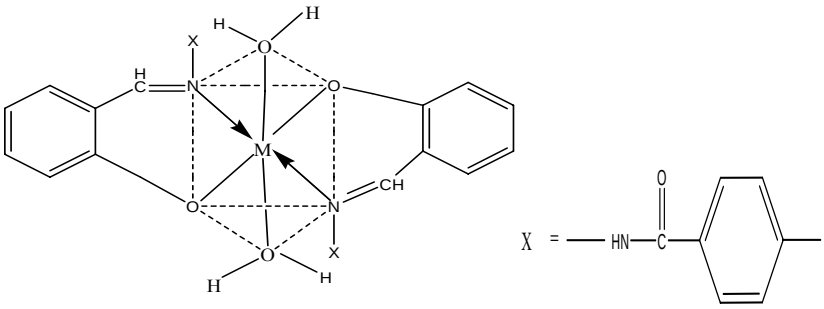


Fig. 16. Powder XRD of OHBAPTH-La complex



M= Cu(II) & La(III)

Fig.17.OHBAPTHMetal complexes

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