

# Preparation and Analysis of Mixed Ligand Lanthanum Complexes involving determination of their Physicochemical, Spectral, Thermal properties and Antibacterial Investigation.

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## KEYWORDS

*Amino acids,  
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## Abstract

The mixed ligand lanthanum (III) metal complexes are prepared from ortho hydroxybenzaldehyde oxime ( OHBO) and chemical compounds L-alanine, L-isoleucine, L-proline, L-phenylalanine. All complexes are off white in colour and stable solids. The non-electrolyte nature of all the new synthesized complexes confirmed by conductometric measurement. The infrared study of lanthanum complexes reveals the strong metal ligand bonding by the oxygen and nitrogen atoms in the complexes. magnetic susceptibility also shows that the mixed ligand complexes are paramagnetic in nature. The differential thermal analysis and thermo gravimetric measurement indicates the presence of coordinated water molecules. The high decomposition temperature of the complexes shows the strong metal ligand bonding within complex molecules. UV-Visible spectroscopy clears the intra ligand and LCMT transitions in the complexes. complexes are formed in 1:2:1 ratio indicating by Elemental analysis. Agar cup method and Tube dilution method are employed for antibacterial detection of lanthanum (III) metal complexes which indicates that complexes exhibit antibacterial activity against selected strains of bacterial

## Introduction

In the recent years, synthesis of complexes of transition and inner transition elements from two or more different ligands is the topic of interest for many researchers as the mixed ligand complexes have more stability. It is known that in the biological systems, these complexes play the crucial role.[1-2] There are numerous mixed ligand complexes which are studied as they are showing various biological activities like anticancer, antifungal,

antibacterial [3-7] Large number of ligands containing different donor atoms like O,N are used for the synthesis of mixed ligand complexes to find their antidiabetic and antituberculosis activities[8-9] Mixed ligand complexes of lanthanides mainly cerium metal with amino acids are prepared and characterized and studied for their various biological activities like antibacterial, antimicrobial activities etc.[10-13] The amino

acids containing N,O,S donor atoms with the active groups like  $-NH_2$ ,  $-SH$  and  $-COOH$  have ability to form metal complexes, received lots of interest showing vital biological activities are also prepared and characterized.[14-17] The mixed ligand complexes also employed in various field like acts as catalyst in many reactions such as hydroformylation, reduction, oxidation, oxidative cleavage, decomposition of hydrogen peroxide etc. and thus get their share of attention. Preparation of cerium metal complexes, their characterization and antibacterial investigation have been also reported by many researchers.[18-20] Keeping these various facts in mind emerging through the literature survey, we reported here the synthesis of mixed ligand complexes of Cerium metal by using Salicylaldoxime as primary ligand and various amino acids L-Valine, L-Glycine, L-Hydroxy proline, L-Threonine as secondary ligands. All the complexes were characterized by Spectral and Physico-chemical methods. Determination of molar conductance,  $p^H$ , magnetic susceptibility of the complexes as well as Elemental analysis, UV-Visible, FTIR spectral, Thermal analysis were carried out. Antibacterial investigation of the complexes also performed.

## Methodology

### 2.1 Materials :

The compounds required , Lanthanum III)chloride heptahydrate, ortho hydroxylbenzaldehyde oxime ,L-alanine, L-isoleucine, L-proline, L-phenylalanine were purchased from S.D. Fine Chemicals, Mumbai, and taken for the synthesis of the mixed ligand complexes. The analytical grade solvents dimethyl sulphoxide, dimethyl formamide, acetone, ethanol were used and purified by using standard protocol.[21-22]

### 2.2 Instrumentation :

Electric conductance of all lanthanum complexes were measured by Equiptronics Autoranging Conductivity Meter Model No. EQ-667 by preparing 0.001 M solution of complexes in DMSO solvent. C, H and N content were measured on Elementar Unicube at Spring Testing Solutions Laboratory, Mumbai . Thermal behavior of the Lanthanum complexes were recorded in controlled nitrogen atmosphere on a TGA 5500 by water at Spring Testing Solutions Laboratory, Mumbai at the heating rate of  $10^\circ C$  per minute on increasing temperature up to  $900^\circ C$ . UV spectra of the all lanthanum complexes were recorded in  $10^{-3}$  M solution in DMSO on Shimadzu UV/VIS-1900 spectrophotometer in the ultraviolet and visible region of 200 nm to 800 nm range. Shimadzu FT-IR spectrophotometer of IRAffinity<sup>-1</sup> was utilized to report the Infrared spectra of all the complexes in KBr disc in the region 4000-400

cm<sup>-1</sup> on Aligent Microlab Carry 630 IR instrument. Both Electronic absorption spectra and FTIR were measured at Spring Testing Solutions Laboratory, Mumbai .

### 2.3 Synthesis of mixed ligand Complexes :

The alcoholic solution (10 ml) of lanthanum (III) chloride heptahydrate (371 mg. 1mmol) was prepared. The alcoholic solution (10 ml) of ortho hydroxylbenzaldehyde oxime (OHBO) (274 mg, 2mmol) was mixed with constant stirring by using magnetic stirrer. Then all the reaction mixture was kept in boiling water bath for 20-30 minutes with stirring. Then to this hot reaction mixture , the aqueous solution of secondary ligand compound was added dropwise with constant stirring. The whole reaction mass was then heated for 10-15 minutes in a water bath at 50 °C. The lanthanum complex was then precipitated by raising the pH of reaction mixture.. The whole reaction mixture was cooled and solid complex was filtered then it was washed by water followed by using ethanol. All the complexes were air dried for 24 hours and utilized for further study

### 2.4 Antibacterial Screening :

#### 2.4.1 Tube Dilution Method

This method was employed to determine the minimum inhibitory concentration (MIC) of complexes. mixed ligand Lanthanum (III) complexes were subjected to *in vitro*

screening against the selected strains of bacteria *S. aureus*, *C. diphtheriae*, *S. typhi* and *P. aeruginosa* using Muller Hinton broth as the culture medium. Stock solution of concentration 1000 µg/cm<sup>3</sup> was obtained by dissolving the test compound of 10 mg in 10 cm<sup>3</sup> of DMSO solvent. After successfully preparing the stock solution, the various aliquots of 5, 10, 15, 20 to ...., 250 µg/cm<sup>3</sup> were obtained in test broth. Preparation of Bacterial inoculums in sterilized Muller Hinton broth and their incubation in refrigerator for 24 hrs. at 37<sup>0</sup>C was performed. Aliquots were then dispensed in borosilicate test tube of diameter 150 x 20 mm and the bacterial inoculums 0.1 cm<sup>3</sup> of the desired bacterial strain (*S. aureus*, *C. diphtheriae*, *S. typhi* and *P. aeruginosa*) containing 10<sup>6</sup> bacteria/cm<sup>3</sup> was inoculated in the tube. The tubes were kept on a rotary shaker for 24 h at 37<sup>0</sup>C for incubation. On the next day, the presence or absence of the growth of the test organisms was observed to find the MIC of complexes. Tetracycline was used as standard control and MIC of tetracycline against selected strain of bacteria was compared with test compound.

#### 2.4.2 Agar Cup Method

Lanthanum (III) metal were subjected to check their antibacterial activity against the selected

strains of bacteria, *S. aureus*, *C. diphtheriae*, the gram positive and *S. typhi* and *P. aeruginosa* gram negative bacteria using Agar Cup method. In this method, the petriplate filled with sterile nutrient agar containing desired strain of bacteria was poured to a height of about 5mm and then kept the plate as it to solidify the nutrient agar. After this, with help of sterile cork borer, a single cup having 8 mm diameter was cut from middle part of the plate and it was filled with the sample solution of 1000 µg/cm<sup>3</sup> concentration. This test solution was allowed to spread in surrounding agar by keeping 10 minutes in refrigerator and the incubated for 24 h at 37 °C.

**Table No. 1 :**

| Complex                                         | Empirical Formula                                               | Molecular Weight | Colour    |
|-------------------------------------------------|-----------------------------------------------------------------|------------------|-----------|
| [La(OHBO) <sub>2</sub> (Ala).2H <sub>2</sub> O] | LaC <sub>17</sub> H <sub>22</sub> O <sub>8</sub> N <sub>3</sub> | 535              | off white |
| [La(OHBO) <sub>2</sub> (Pro).2H <sub>2</sub> O] | LaC <sub>19</sub> H <sub>24</sub> O <sub>8</sub> N <sub>3</sub> | 561              | off white |
| [La(OHBO) <sub>2</sub> (Iso).2H <sub>2</sub> O] | LaC <sub>20</sub> H <sub>28</sub> O <sub>8</sub> N <sub>3</sub> | 577              | off white |
| [La(OHBO) <sub>2</sub> (Phe).2H <sub>2</sub> O] | LaC <sub>23</sub> H <sub>26</sub> O <sub>8</sub> N <sub>3</sub> | 611              | off white |

Table No.2 indicates the temperature of decomposition of all lanthanum complexes which are in the range of 245-260 which shows that there is strong Metal-ligands bonding within the complexes.as well as they are also

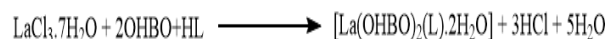
**Table No-2:**

| Complex                                         | Decomposition Temp. (°C) | pH   |
|-------------------------------------------------|--------------------------|------|
| [La(OHBO) <sub>2</sub> (Ala).2H <sub>2</sub> O] | 255                      | 6.95 |
| [La(OHBO) <sub>2</sub> (Pro).2H <sub>2</sub> O] | 260                      | 7.03 |
| [La(OHBO) <sub>2</sub> (Iso).2H <sub>2</sub> O] | 250                      | 6.98 |
| [La(OHBO) <sub>2</sub> (Phe).2H <sub>2</sub> O] | 245                      | 7.06 |

## Results and discussion

### 3.1 Preparation of complexes

The lanthanum complexes synthesized as follows



Where, OHBO : ortho hydroxylbenzaldehyde oxime

HL : secondary ligand

### 3.2 Physico-Chemical Properties of complexes

The synthesized lanthanum metal mixed ligand complexes are off white in colour (Table No.1) and are non-hygroscopic in nature.

insoluble in common organic solvents like ethanol , acetone, methanol etc. but in the solvents like DMSO and DMF, complexes are partially miscible.

Table No-3 explains the molar conductance of lanthanum complexes and room temperature magnetic susceptibility of all complexes. The molar conductance values are in the range of 0.0024 to 0.0017 Mhos  $\text{cm}^2 \text{mol}^{-1}$  showing that the all lanthanum complexes are nonelectrolyte

in nature.[23] The elemental analysis data of complexes given in the Table No-3 which confirms that the general formula of lanthanum complexes is 1:2:1 [24] The room temperature effective magnetic moment of all the lanthanum (III) complexes shows that all complexes are Diamagnetic in nature.[25]

**Table No-3 :**

| Complex                                         | Elemental Analysis Found (Calculated) |                |                |                  | Molar Conductance (Mhos $\text{cm}^2 \text{mol}^{-1}$ ) | Magnetic Nature |
|-------------------------------------------------|---------------------------------------|----------------|----------------|------------------|---------------------------------------------------------|-----------------|
|                                                 | % C                                   | % H            | % N            | % M              |                                                         |                 |
| [La(OHBO) <sub>2</sub> (Ala).2H <sub>2</sub> O] | 38.08<br>(38.13)                      | 4.10<br>(4.11) | 7.83<br>(7.85) | 25.96<br>(25.98) | 0.0024                                                  | Diamagnetic     |
| [La(OHBO) <sub>2</sub> (Pro).2H <sub>2</sub> O] | 40.62<br>(40.64)                      | 4.25<br>(4.27) | 7.43<br>(7.48) | 24.73<br>(24.77) | 0.0028                                                  | Diamagnetic     |
| [La(OHBO) <sub>2</sub> (Iso).2H <sub>2</sub> O] | 41.56<br>(41.59)                      | 4.82<br>(4.85) | 7.25<br>(7.27) | 24.06<br>(24.09) | 0.0021                                                  | Diamagnetic     |
| [La(OHBO) <sub>2</sub> (Phe).2H <sub>2</sub> O] | 45.15<br>(45.18)                      | 4.24<br>(4.25) | 6.86<br>(6.87) | 22.71<br>(22.75) | 0.0017                                                  | Diamagnetic     |

### Electronic absorption spectra of the complexes :

The electronic spectra of prepared complexes were measured in DMSO solvent.

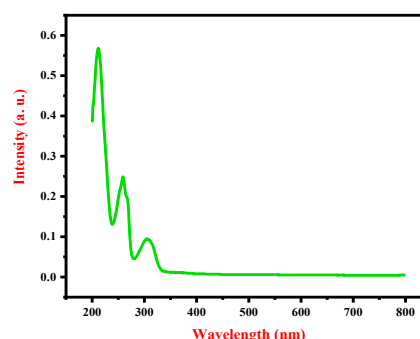
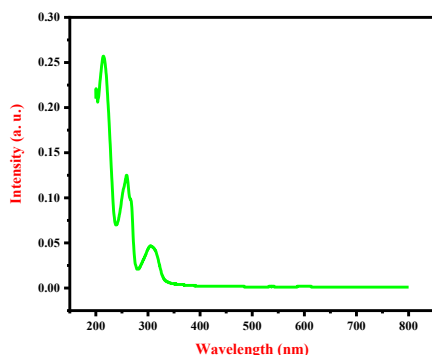
In the UV-visible spectra, many amino acids shows peak below 200 nm transitions.[26].

These bands are appear to higher wavelength region in the all the complexes showing  $\pi \rightarrow \pi^*$  transitions and charge transfer transitions in the complexes from ligand to metal ion and vice versa.

**Table No. 4 : Electronic Absorption Spectra of lanthanum complexes**

| Complex                                         | $\lambda$ (nm) | $\nu$ (cm <sup>-1</sup> ) | Proposed Assignments    |
|-------------------------------------------------|----------------|---------------------------|-------------------------|
| [La(OHBO) <sub>2</sub> (Ala).2H <sub>2</sub> O] | 210            | 47619                     | $\pi \rightarrow \pi^*$ |
|                                                 | 252            | 39682                     | $n \rightarrow \pi^*$   |
|                                                 | 315            | 31746                     | Charge-transfer         |
| [La(OHBO) <sub>2</sub> (Pro).2H <sub>2</sub> O] | 220            | 45454                     | $\pi \rightarrow \pi^*$ |
|                                                 | 255            | 39215                     | $n \rightarrow \pi^*$   |
|                                                 | 312            | 32051                     | Charge-transfer         |

|                                                 |     |       |                         |
|-------------------------------------------------|-----|-------|-------------------------|
| [La(OHBO) <sub>2</sub> (Iso).2H <sub>2</sub> O] | 215 | 46512 | $\pi \rightarrow \pi^*$ |
|                                                 | 259 | 38610 | $n \rightarrow \pi^*$   |
|                                                 | 306 | 32680 | Charge-transfer         |
| [La(OHBO) <sub>2</sub> (Phe).2H <sub>2</sub> O] | 211 | 47393 | $\pi \rightarrow \pi^*$ |
|                                                 | 259 | 38610 | $n \rightarrow \pi^*$   |
|                                                 | 308 | 32467 | Charge-transfer         |



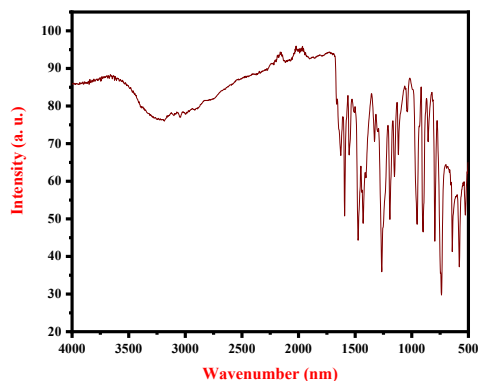
### IR spectra of Lanthanum Complexes :

The primary ligand ortho hydroxylbenzaldehyde oxime shows band at  $3550\text{ cm}^{-1}$  which is due to the phenolic -OH group. For free oxime -OH group, the band obtained in the range of  $3225$  to  $3360\text{ cm}^{-1}$ . The absence of band at  $3550\text{ cm}^{-1}$  in the all the synthesized lanthanum complexes confirms the deprotonation of OH group and coordination of phenolic oxygen with lanthanum metal in the synthesized complexes. The band for free oxime group has been present in the all the lanthanum complexes and not involved in the bond formation. The shift of frequency of C=N stretching vibrations to lower range of  $1590$ - $1593\text{ cm}^{-1}$  confirms that the oxime group of primary ligand bonded through nitrogen to central metal lanthanum in all the complexes. The strong band obtained in

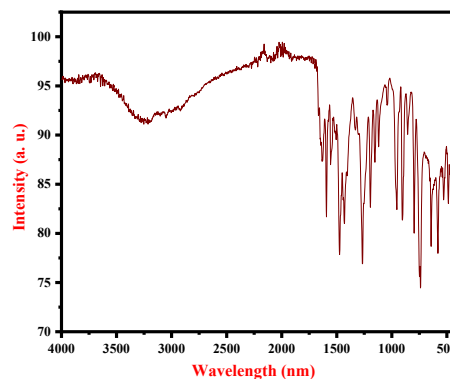
the region between  $952$ - $956\text{ cm}^{-1}$  in all lanthanum complexes is due to C-O stretching vibrations. However the strong bands observed in the range of wavelength  $1265$  -  $1267\text{ cm}^{-1}$  and weak bands ranging from  $1474\text{ cm}^{-1}$  -  $1472\text{ cm}^{-1}$  are ascribed for COO - asymmetric vibration modes and symmetric vibration modes respectively in the all complexes. It confirms that metal and acid group of secondary ligand coordinated through oxygen atom. The band in the range of  $1593$ - $1591\text{ cm}^{-1}$  and  $1628$ - $1615\text{ cm}^{-1}$  are due to N-H stretching vibrations in all the lanthanum complexes which also signify that the secondary ligand amino acids also bonded to lanthanum metal in the all complexes. The medium bands observed in the range of  $579$ - $585\text{ cm}^{-1}$  and  $643$ - $644\text{ cm}^{-1}$  are mainly assigned to M-N and M-O stretching vibrations. This

provides bonding with oxygen and nitrogen of

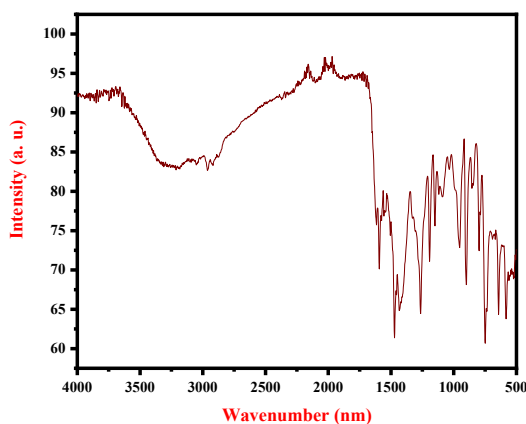
the both ligands.[27-28]



[La(OHBO)<sub>2</sub>(Ala).2H<sub>2</sub>O]



[La(OHBO)<sub>2</sub>(Pro).2H<sub>2</sub>O]



[La(OHBO)<sub>2</sub>(Iso).2H<sub>2</sub>O]

### Antibacterial studies:

The diameter of the zone inhibition (mm) was used to compare the antibacterial behavior of the complexes. It confirms that all complexes exhibit biological activity by inhibiting the growth of microorganisms. The lanthanum Complexes were more sensitive against bacterial strain *S. aureus* and *S. typhi* as compared to *C. diphtheria* and *P. aeruginosa*. It was found that lanthanum metal complexes

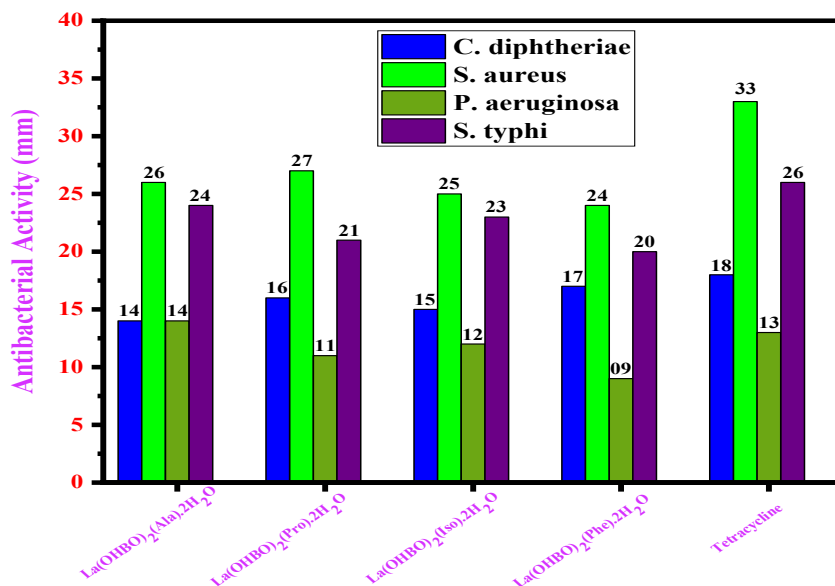
showing more activity to stop the growth of bacteria than ligands.[29] The MIC) of the ligand, metal salt and cerium complexes was lie in the range of 100-200 µg/mL, 75-200 µg/mL and 10-35 µg/mL respectively. Due to chelation, metal complexes inhibit the growth of bacteria as it lowers the polarity of metal ions in the complexes and increases hydrophobic property and helps to permeate through the lipid layers of microorganism.

**Table No.- 5 : Antibacterial results of synthesized Lanthanum (III) Complexes by Agar Cup Method**

| Sr. No. | Complex                                         | Antibacterial Activity (mm) with |                  |                      |                 |
|---------|-------------------------------------------------|----------------------------------|------------------|----------------------|-----------------|
|         |                                                 | <i>C. diphtheriae</i>            | <i>S. aureus</i> | <i>P. aeruginosa</i> | <i>S. typhi</i> |
| 1.      | [La(OHBO) <sub>2</sub> (Ala).2H <sub>2</sub> O] | 14                               | 26               | 14                   | 24              |
| 3.      | [La(OHBO) <sub>2</sub> (Pro).2H <sub>2</sub> O] | 16                               | 27               | 11                   | 21              |
| 4.      | [La(OHBO) <sub>2</sub> (Iso).2H <sub>2</sub> O] | 15                               | 25               | 12                   | 23              |
| 5.      | [La(OHBO) <sub>2</sub> (Phe).2H <sub>2</sub> O] | 17                               | 24               | 09                   | 20              |
| 6.      | Tetracycline                                    | 18                               | 33               | 13                   | 26              |

**Table 07: MIC (µg/mL) data of prepared Lanthanum (III) Complexes**

| Complex                                         | MIC (µg/mL)           |                  |                      |                 |
|-------------------------------------------------|-----------------------|------------------|----------------------|-----------------|
|                                                 | <i>C. diphtheriae</i> | <i>S. aureus</i> | <i>P. aeruginosa</i> | <i>S. typhi</i> |
| [La(OHBO) <sub>2</sub> (Ala).2H <sub>2</sub> O] | 30                    | 15               | 35                   | 15              |
| [La(OHBO) <sub>2</sub> (Pro).2H <sub>2</sub> O] | 35                    | 10               | 25                   | 20              |
| [La(OHBO) <sub>2</sub> (Iso).2H <sub>2</sub> O] | 30                    | 15               | 30                   | 10              |
| [La(OHBO) <sub>2</sub> (Phe).2H <sub>2</sub> O] | 35                    | 15               | 25                   | 15              |
| LaCl <sub>3</sub> ·7H <sub>2</sub> O            | 160                   | 80               | 200                  | 75              |
| OHBO ligand                                     | 200                   | 100              | 180                  | 130             |
| Tetracycline                                    | 2.0                   | 1.5              | 8.0                  | 1.5             |



### Thermal Study of the complexes :

The thermal study of the synthesized

complexes was carried by using DTA and TG

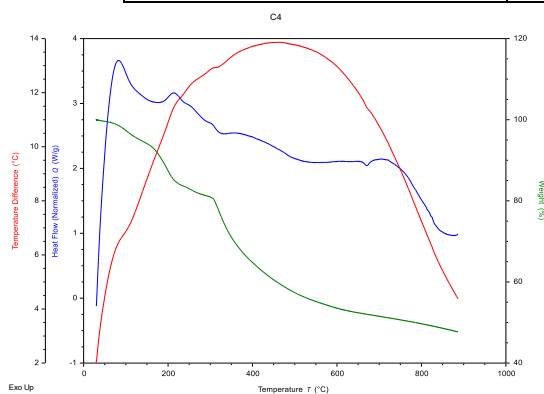
analysis in the controlled atmosphere of

nitrogen gas with the constant heating rate of 10 °C / minute. All complexes were heated upto 900°C and the thermogram curve was got to exhibit three decomposition curves indicating that there was gradual weight loss in the complex as the temperature has been raised. All the lanthanum complexes showing similar behavior in the TG, DTA study. First loss has been observed in the range of 10-240°C which due to the coordinated water molecules. Second

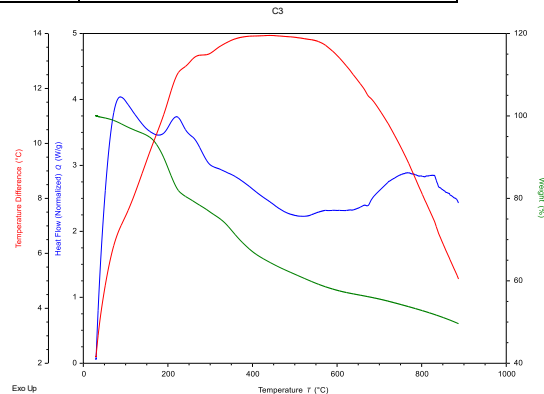
loss in the weight of complex seen in the range of 250-390°C due to loss of secondary ligand. The final loss in the weight observed in the range of 400-750°C due to simultaneous loss of two hydroxylbenzaldehyde oxime ligand. After this, TG-DTA curve shows straight line confirming the end of decomposition of mixed ligand lanthanum complexes.

**Table No.8 : Data of Thermal analysis of complexes :**

| Complex                                         | Temp. Range (°C) | Loss in Weight due to |
|-------------------------------------------------|------------------|-----------------------|
| [La(OHBO) <sub>2</sub> (Ala).2H <sub>2</sub> O] | 15-230           | Two water molecules   |
|                                                 | 255-395          | Amino acid            |
|                                                 | 400-755          | Two OHBO molecules    |
| [La(OHBO) <sub>2</sub> (Pro).2H <sub>2</sub> O] | 10-240           | Two water molecules   |
|                                                 | 260-390          | Amino acid            |
|                                                 | 400-750          | Two OHBO molecules    |
| [La(OHBO) <sub>2</sub> (Iso).2H <sub>2</sub> O] | 28-230           | Two water molecules   |
|                                                 | 250-400          | Amino acid            |
|                                                 | 410-740          | Two OHBO molecules    |
| [La(OHBO) <sub>2</sub> (Phe).2H <sub>2</sub> O] | 20-220           | Two water molecules   |
|                                                 | 245-405          | Amino acid            |
|                                                 | 405-760          | Two OHBO molecules    |

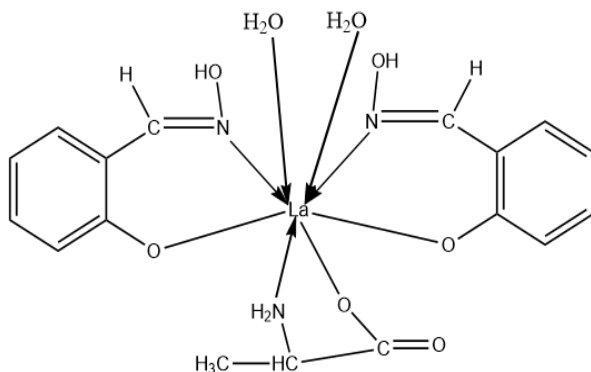


**[La(OHBO)<sub>2</sub>(Iso).2H<sub>2</sub>O]**

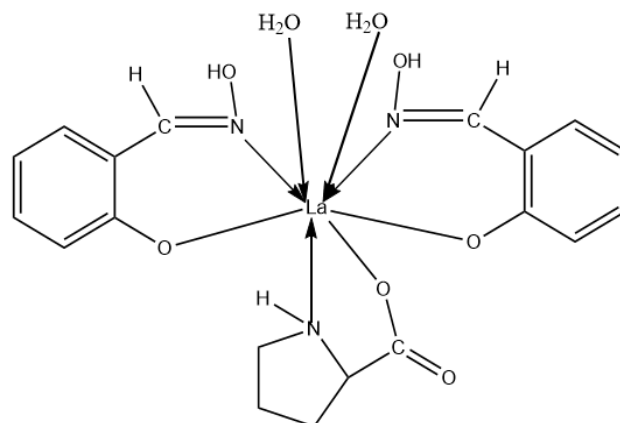


**[La(OHBO)<sub>2</sub>(Phe).2H<sub>2</sub>O]**

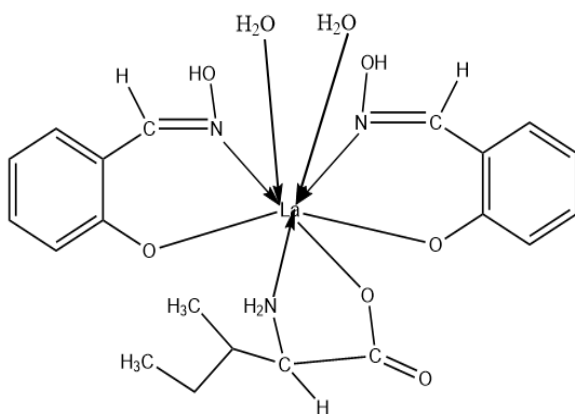
Hence the proposed structures of La(III) metal mixed ligand complexes are as following-



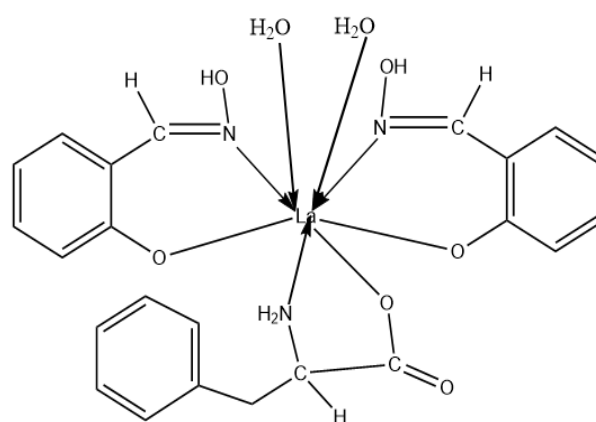
[La(OHBO)<sub>2</sub>(Ala).2H<sub>2</sub>O]



[La(OHBO)<sub>2</sub>(Pro).2H<sub>2</sub>O]



[La(OHBO)<sub>2</sub>(Iso).2H<sub>2</sub>O]



[La(OHBO)<sub>2</sub>(Phe).2H<sub>2</sub>O]

## Conclusion

We have successfully synthesized mixed ligand complexes of Lanthanum (III) metal of type [La(OHBO)<sub>2</sub>(L).2H<sub>2</sub>O] using Ligands

containing N.O donor atoms. Complexes were off white in color and stable at room temperature. Molar conductance of all complexes was very low indicating that the

complexes are non-electrolyte in nature. The detail IR study of lanthanum complexes clears that lanthanum metal bonding taking place through the N-and O-donor atoms of the primary and secondary ligands. The ultra-violet study of all the complexes signify that there is intra ligand and charge transfer transitions in the synthesized complexes. All the lanthanum metal complexes has high decomposition temperature values which confirms that it has strong metal ligand bonding. The Differential thermal analysis and thermo gravimetric analysis which indicates gradual weight loss in the lanthanum complexes confirming the presence of water molecules and ligands in the complexes. Magnetic study of new synthesized complexes shown that all complexes are diamagnetic in nature. Hence lanthanum complexes has proposed coordination number eight. The antimicrobial study of complexes shows that complexes were more active against the strain of bacterial *S. typhi* and *S. aureus* as compared to bacterial strain *C. diphtheria* and *P. aeruginosa*.

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#### COMPETING INTERESTS

Authors have declared that they have no conflict of interest.

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