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# Studies of some rare earth metal complexes with Schiff base Ligand with the help of X-ray Diffraction

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**Abstract:**

Studies on the Powder X-ray diffraction parameter of some rare earth metal complexes such as Ce(III), Gd(III) and Tb(III) has been synthesized by a 2-[1-(4-methoxyphenylimino) ethyl-5-methoxyphenol] by using 2-Hydroxy-4-Methoxyacetophenone and 4-Methoxyaniline having equimolar ratio of 1:2 (Metal : Ligand). In the formation of different metal complexes. The complexes are different colours and different physic-chemical properties. The X-ray diffraction data suggest orthorhombic crystal system for Ce(III), Gd(III) and Tb(III) complex. The X-ray diffraction data were also be used for the determination of various parameter, unit cell volume and Miller indices values(hkl).

**Keywords:** X-ray diffraction studies, 2-Hydroxy-4-Methoxyacetophenone, 4-methoxyaniline

## Introduction:

Powder X-ray diffraction studies on the metal complexes of an instrumental technique that used to identify crystalline Material [1]. X-ray diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined [2]. But the systematic study on determination of Schiff base ligands and their metal complexes Ce(III), Gd(III) and Tb(III). A diffraction pattern plots intensity against the angle of the detector, 2θ. In a diffraction pattern, the peak position depends upon the wavelength. Absolute intensity (number of X-rays observed in a given peak) may vary by instrumental and experimental parameters. For these measurements, Rigaku MiniFlex X-ray Diffractometer with a radiation source of CuKα was used [3,4]. The objective of an X-Ray diffraction measurement is to determine the dimensions and shape of unit cell and to identify the detailed structure of the molecule. To achieve this objective, we must be able to mathematically express the nature of the measured interference pattern in terms of the position of the various atoms within the crystal [5].

**Experimental:**

All chemicals and solvents used were of analytical grade purchased from sigma Aldrich, Alfa alser Chemical Company ltd.. The powders were characterized by X-Ray diffraction (XRD) using Cu-K$\alpha_1$ radiation PAN analytical X-Ray diffract meter, at 40KVand 30mA in the range 20< 2θ >60 degree with step size of 0.02-degree 2θ and scanning speed of 0.5-degree 2θ per minutes.

**1) Synthesis of Schiff base Ligand:**

The Schiff base ligand were synthesized by total amount of solution prepared by 50 ml ethanol contain 0.01mol (0.166g) of 2-Hydroxy-4-Methoxyacetophenone and 0.01mol (0.123 g) of 4-methoxyaniline were reflux for 10 hrs. at 80 $^0$C temperature. Fine shining Gray solid crystal precipitate of the Schiff base ligand was filtered off, washed with ethanol and stored in vacuum desiccators over anhydrous calcium chloride. The purity of ligand was checked by using TLC by using silica gel plates. The product was purified and recrystallized with hot ethanol. Yield obtained were 70%.

**. 2) Synthesis of Metal complexes:**

For preparation of some lanthanide (III) complexes 0.02m of ligands in 25 ml alcohol was taken in 100 ml round bottom flask. then 25 ml alcoholic solution of metal nitrate 0.01m was added in it and heated for few minutes with constant stirring to ensure complete mixing of ligand. the content was stirred and reflux at 10 hrs. the resultant solid was digested for one hour. the precipitate was filtered, washed with ethanol, diethyl ether and dried in vacuum desiccators over anhydrous calcium chloride. the molar ratio 1:2 (metal: ligand)

**Results and Discussion:**

The study on powder X-ray diffraction of Ce (III), Gd(III) and Tb(III) complexes was Schiff base ligand scanned in the range of 5-75$^0$at wavelength 1.540598Å. The diffractogram and associated data depict the 2θ value each peak, relative intensity and inter-planar spacing (d-values).

**1) X-Ray Diffraction Studies of Ce (III) complex**

The Ce (III) complexes of ligands were selected for the study subjected to X-ray powder diffraction studies. The powder diffraction of these complexes is presented in (Fig. 01)**.** The X-ray powder data of all the main peaks have been indexed independently by trial-and-error method. The X-ray diffractogram of Ce (III) complex shows ten reflections with maxima at 2θ = 5.68° corresponding to d value 15.52 Å. The unit cell values of lattice constants are a = 20.231 Å, b = 15.478 Å, c = 11.236 Å and α = β = γ = 90° and unit cell volume V = 3518.389(Å$^3$).

The observed densities of Ce (III) complexes of ligands were 7.61 gcm$^{-3}$ respectively. While the calculated densities from Z values and cell volume for complexes of Ce (III) with were 7.60 gcm$^{-3}$ respectively. In concurrence with the cell parameters of the complexes. The Ce (III) complex the condition a ≠ b ≠ c and α = β = γ = 90° required for the compound to be orthorhombic was found to be satisfactory.

Damianos G. Paschalidis and Maria Gdaniec[6]. Studied the structure of Ce (III) complex of Hydrazone Schiff base ligand and found to monoclinic crystal system with space group P2$_1$/n with lattice parameters a = 11.32 Å, b = 17.39 Å, c = 17.15 Å and Z = 4. Conclusively The Ce (III) complex of crystallizes as orthorhombic crystal system.

**Table:1.Unit cell data and crystal lattice parameter of Ce (III) complex.**

| Parameter | Data | Parameter | Data |
|---|---|---|---|
| **a(Å)** | 20.231 | Volume (Å) | 3518.389 |
| **b(Å)** | 15.478 | Density(obs.) | 7.61 |
| **c(Å)** | 11.236 | Density (cal.) | 7.60 |
| **α(degree)** | 90 | Z | 2 |
| **β(degree)** | 90 | Space group | P |
| **γ(degree)** | 90 | Crystal system | Orthorhombic |

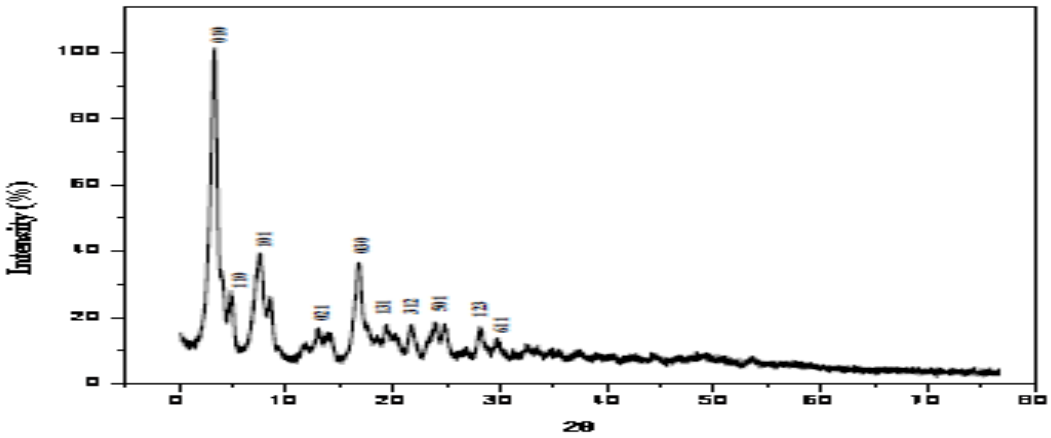


***Fig. 01. X-ray diffractogram of Ce (III) complex***

**2) X-Ray Diffraction Studies of Gd (III) complex**

The Gadolinium (III) complex of ligand was selected for the study of X-ray powder diffraction. Diffractogram is shown in Fig.02. The Gd (III) complex of showed six reflections with maxima at 2θ = 5.630 corresponding to d value 15.68Å. The unit cell values of lattice constants were a = 16.248Å, b = 12.456Å, c = 9.021 Å, α = β = γ = 90° and unit cell volume V = 1825.715 $(Å)^3$. The observed and calculated densities of Gd (III) complex were 7.89, 7.88 $gcm^{-3}$ respectively. In concurrence with these cell parameters of the complex, the condition such as a ≠ b ≠ c and α = β = γ = 90° required for the compound to be orthorhombic were tested and found to be satisfactory.

Single crystal of Gd (III) complex of bis (2-pyridine carboxaldehyde) propylene-1,3-diimine were synthesized by Michael[6]. The structural studies reported that the crystal has orthorhombic system with space group Pbca. The lattice parameters were a= 14.90Å, b = 16.38Å, c = 16.68 Å, and α = β = γ = 90° and the unit cell volume was 4073 $(Å)^3$ . The complex reported to show orthorhombic crystal system.

**Table:2. Unit cell data and crystal lattice parameter of Gd (III) complex.**

| Parameter | Data | Parameter | Data |
|---|---|---|---|
| **a(Å)** | 16.248 | Volume (Å) | 1825.715 |
| **b(Å)** | 12.456 | Density(obs.) | 7.89 |
| **c(Å)** | 9.021 | Density (cal.) | 7.88 |
| **α(degree)** | 90 | Z | 1 |

| | | | |
|---|---|---|---|
| **β(degree)** | 90 | Space group | P |
| **γ(degree)** | 90 | Crystal system | Orthorhombic |

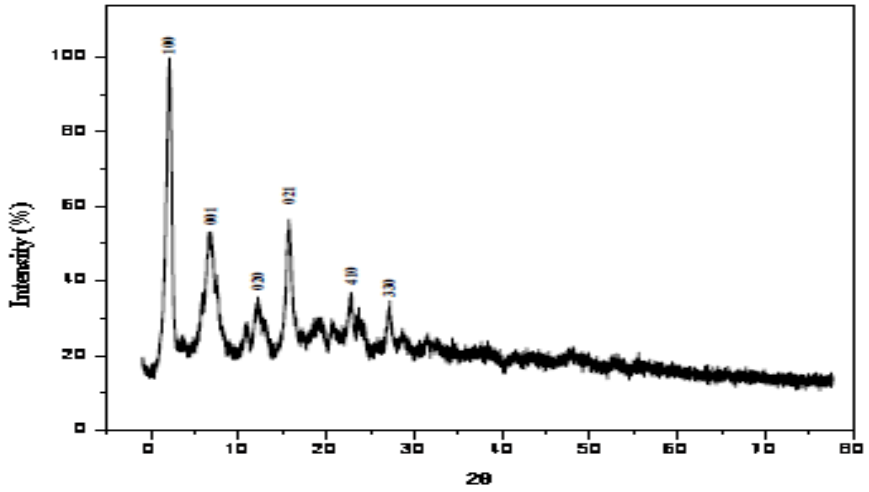


***Fig. 02. X-ray diffractogram of Gd (III) complex.***

**3) X-Ray Diffraction Studies of Tb (III) Complex**

The Terbium (III) complex of ligand was selected for the study of X-ray powder diffraction. Diffractogram is shown in Fig.03**.** The complex of showed thirty reflections with maxima at 2θ = 5.611 corresponding to d value 15.73Å. The unit cell values of lattice constants were a = 20.356Å, b = 15.325Å, c = 11.456 Å, α = 90°, β = 109°, γ = 103° and unit cell volume V = 3282.036 (Å)$^3$. The observed and calculated densities of Tb (III) complex were 7.80,779 gcm$^{-3}$ respectively. In concurrence with these cell parameters of the complex, the condition such as a ≠ b ≠ c and α = β = γ = 90° required for the compound to be orthorhombic were tested and found to be satisfactory.

Single crystal of Tb (III) complex of structural, magnetic and luminescent properties of Lanthanide complexes with N-Salicylideneglycin were synthesized by Jan Vanco[7]. The structural studies reported that the crystal has monoclinic system with space group C2/c. The lattice parameters were a = 38.25Å, b = 8.07Å, c = 14.24 Å, and α = β = γ = 90° and the unit cell volume was 4318.0 (Å)$^3$ .

**Table:3. Unit cell data and crystal lattice parameter of Tb (III) complex.**

| Parameter | Data | Parameter | Data |
|---|---|---|---|
| **a(Å)** | 20.356 | Volume (Å) | 3282.036 |
| **b(Å)** | 15.325 | Density(obs.) | 7.80 |
| **c(Å)** | 11.456 | Density (cal.) | 7.79 |
| **α(degree)** | 90 | Z | 1 |
| **β(degree)** | 109 | Space group | P |
| **γ(degree)** | 103 | Crystal system | Orthorhombic |

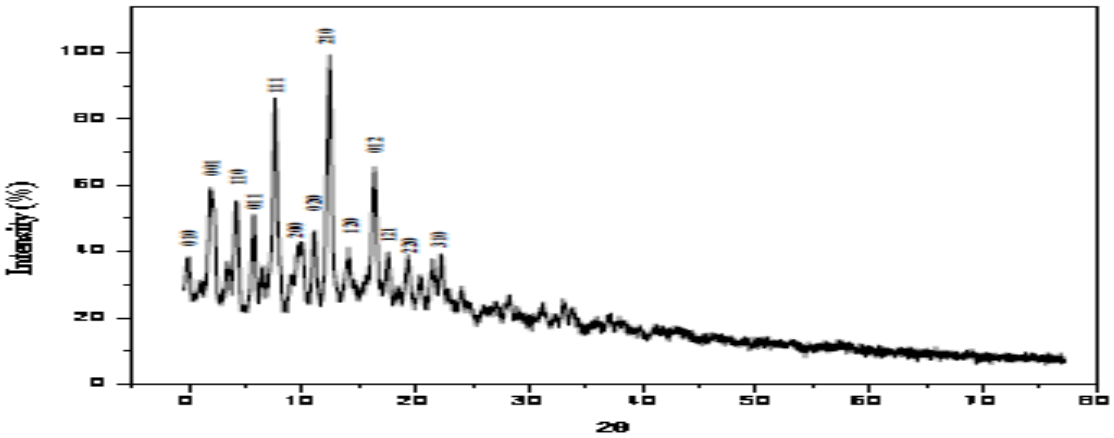


***Fig. 03. X-ray diffractogram of Tb (III) complex.***

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