



SYNTHESIS AND CHARACTERIZATION OF ND(III), DY(III), SM(III) AND PR(III) COMPLEXES WITH SUBSTITUTED DIACETYLMONOOXIMETHIOCARBOHYDRAZIDE-P-CHLOROBENZALDEHYDE

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Abstract

Ligand resulted from reaction between diacetylmonooximethiocarbohydrazide and p-chlorobenzaldehyde have been synthesised and investigated for its coordinating properties for tripositive metal ions such as Nd(III), Dy(III), Sm(III), and Pr(III). All complexes prepared were of 1:1 electrolyte bichelated compounds and are characterized as seven coordinated geometries based on electronic spectrum and magnetic moment data. In FTIR spectra, through the nitrogen atom of azomethine and oximino groups, the prepared HDMTpCB ligand interacts with the central metal ions and functions as a bidentate ligand. For the Ln(III) complexes, the magnetic moments and electronic spectral data support seven coordinated geometry for these complexes. Additionally, using the X-ray diffraction method, the structure of the Ln(III) complexes has been established.

Keywords: Diacetylmonooximethiocarbohydrazide, p-chlorobenzaldehyde, FT(IR) spectra

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Introduction:

Studies on the development, synthesis, and characterization of transition and inner transition metal complexes had been exhaustively explored owing to their antibacterial properties among varied applications studied¹⁻⁵. The inner transition metals have been used to develop model metallic species of amazing power throughout the past few decades⁶⁻⁹.

The diacetylmonooximethiocarbohydrazide halo-benzaldehyde derivatives and corresponding transition and inner transition metal complexes were previously reported¹⁰⁻¹¹. In view of this, we describe the development and characterization of Nd(III), Dy(III), Sm(III), and Pr(III) complexes of diacetylmonooximethiocarbohydrazide-*p*-chlorobenzaldehyde. IUPAC name of the prepared ligand is *N''*-(*E*)-(4-chlorophenyl) methylidene]-*N'''* -[(2*E*,3*E*)-3-(hydroxyimino) butan-2-ylidene] thio-carbohy drazide. To characterize the synthesized HDMTpCB ligand and its transition metal complexes, various techniques such as physicochemical studies, elemental analysis, spectroscopic studies (UV-Vis, FT-IR, PMR, ESR and X-ray powder diffraction), magnetic and conductance measurements were used.

Experimental:

All of the chemicals and reagents used were of AR grade and have they been purified before use. Solvents of the Spectro grade were used for spectral and conductance studies. The result of the elemental analysis (C, H, N, and O) was performed using an analyzer from Thermo Finning's FLASH-1112 series. Volumetric analysis was used to determine the metal content of the produced complexes. Measurements of the molar conductance of metal complexes in nitrobenzene solutions were made using an ELICO-C1-180

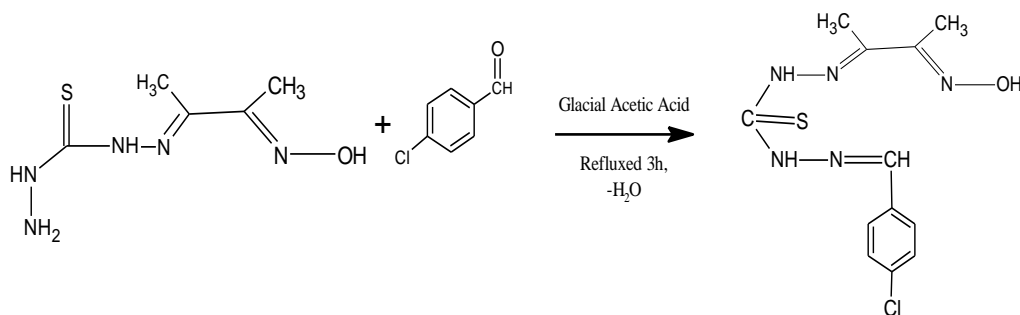
conductivity meter and recorded on a Gouy balance.

On a JASCO V650 UV-visible spectrophotometer, the electronic absorption spectra of trivalent metal complexes in chloroform solution were recorded (double beam). FT-IR spectra of trivalent metal complexes were recorded on solid KBr pellets using a Bruker spectrophotometer. Ln(III) complex was subjected to an X-ray diffraction study using a Nonius KappaCCD diffractometer (MoK α radiation, $\lambda = 0.71069 \text{ \AA}$) at room temperature. With the aid of the software SHELXS-86 and SIR-97, the structures of complexes were solved using the direct technique, and for atoms other than hydrogen, least squares were used to refine the results (CRYSTALS). Hydrogen atoms were isotopically purified.

All hydrogen atoms in complexes were refined in places determined geometrically (except for water molecules containing no hydrogen atoms). The lengths of the C-H and N-H bonds ranged between 0.93-0.98 and 0.86-0.89 \AA . The U_{eq} values of the carbon and nitrogen atoms were assumed to be 1.2–1.5 times higher than the thermal factors UH.

Synthesis of the HDMTpCB:

The diacetylmonooximethiocarbohydrazide (HDM TCH) was synthesized using the published method in the literature¹². Diacetylmonooximethiocarbohydrazide (0.01mol) was combined with *p*-chlorobenzaldehyde (30ml) in a hot ethanolic solution (0.01 l mol). After cooling and three hours of refluxing the reaction mixture, the solid chemicals were filtered out and collected. The mixture was then vacuum-dried after being rinsed with hot distilled water.



Preparation metal complexes:

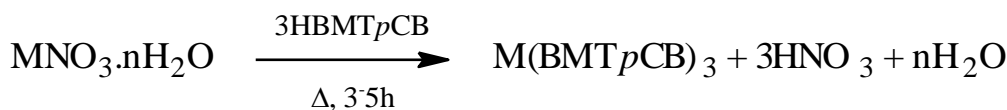
Aqueous solution of metal (III) nitrates (1mmol) was added in 15 mL ethanol solvent and pH of solution was adjusted to value of 7. Subsequently with stirring consistently, 25mL ethanolic solution

of the HDMTpCB ligand (3mmol) was added to the metal (III) nitrate solution and was permitted to reflux for 3-5 hours. After completion of reaction, colored solid complexes were separated, collected, and dried under vacuum after cooling.

Results and Discussion:

Numerous fresh, solid-colored, air-stable trivalent metal complexes of the HBMTpCB ligand have

been synthesized (Table-1). The following are the sequential synthesis routes:



All of these prepared Ln(III) complexes were 1:1 electrolytic in nature ($\Lambda_m = 25.66\text{--}28.98\Omega^{-1}\text{cm}^2\text{mol}^{-1}$ and consistently paramagnetic, which

indicated that the complexes had a high spin octahedral arrangement. Other trivalent metal complexes described by earlier researchers lend confirmation to these conclusions¹³⁻¹⁴.

Table-1:Data on the ligand's physical properties and interactions with its lanthanide (III) metal complexes

Compound	Color	Yield %	M.P. point °C	Elemental Analysis							Magnetic Moments (B.M.)	Electrical Conductance $10^{-3}\text{M}(\text{in NB})$ mhos
				% M Found (Calcd)	% C Found (Calcd)	% H Found (Calcd)	% N Found (Calcd)	% O Found (Calcd)	% S Found (Calcd)	% Cl Found (Calcd)		
HDMPBB	Yellow	84.67	203	-	46.23 (46.19)	4.50 (4.53)	22.40 (22.46)	5.09 (5.13)	10.12 (10.20)	11.33 (11.37)	-	-
[Nd(BMTpCB) ₃]NO ₃	Yellow	79.66	231	17.40 (17.46)	52.06 (52.29)	4.71 (4.72)	27.00 (27.11)	11.49 (11.60)	11.52 (11.60)	4.27 (4.30)	3.84	26.88
[Dy(BMTpCB) ₃]NO ₃	Brown	78.88	241	19.14 (19.24)	51.09 (51.15)	4.59 (4.62)	26.52 (26.52)	11.32 (11.40)	11.39 (11.40)	4.18 (4.20)	10.55	28.98
[Pr(BMTpCB) ₃]NO ₃	Yellow	81.11	242	16.84 (16.93)	51.89 (51.90)	4.67 (4.69)	26.82 (26.91)	11.44 (11.50)	11.42 (11.50)	4.21 (4.26)	3.63	28.85
[Sm(BMTpCB) ₃]NO ₃	Yellow	80.99	239	16.89 (16.93)	51.86 (51.90)	4.66 (4.69)	26.70 (26.91)	11.47 (11.50)	11.46 (11.50)	4.25 (4.26)	3.58	26.01

The electronic absorption spectrum of [Nd(BMTpCB)₃]NO₃ complex exhibited five absorption bands at 512, 582, 679, 741 and 801 nm which is assigned to be transitions $^4\text{I}_{9/2} \rightarrow ^4\text{G}_{9/2}$, $^4\text{I}_{9/2} \rightarrow ^5\text{G}_{5/2}$, $^4\text{I}_{9/2} \rightarrow ^4\text{F}_{9/2}$, $^4\text{I}_{9/2} \rightarrow ^2\text{S}_{3/2}$, $^4\text{F}_{7/2}$ and $^4\text{I}_{9/2} \rightarrow ^4\text{F}_{5/2}$ respectively for seven coordinated geometry, confirmed by its magnetic moment found to be 3.84 BM¹⁵⁻¹⁶. The [Dy(BMTpCB)₃]NO₃ complex had produced six bands at 909, 806, 755, 474, 454 and 420nm due to the $^4\text{H}_{15/2} \rightarrow ^4\text{F}_{7/2}$, $^4\text{H}_{15/2} \rightarrow ^4\text{F}_{5/2}$, $^4\text{H}_{15/2} \rightarrow ^4\text{F}_{3/2}$, $^4\text{H}_{15/2} \rightarrow ^4\text{F}_{9/2}$, $^4\text{H}_{15/2} \rightarrow ^4\text{F}_{15/2}$ and $^4\text{H}_{15/2} \rightarrow ^4\text{G}_{11/2}$ transition respectively of seven coordinated

supported by observed magnetic moment at 10.55 B.M. for this Dy(III) complex¹⁷⁻¹⁹.

The [Sm(BMTpCB)₃]NO₃ complex had exhibited five transitions $^6\text{H}_{5/2} \rightarrow ^4\text{F}_{3/2}$, $^6\text{H}_{5/2} \rightarrow ^4\text{G}_{3/2}$, $^6\text{H}_{5/2} \rightarrow ^4\text{I}_{9/2}$, $^6\text{H}_{5/2} \rightarrow ^4\text{F}_{13/2}$ and $^6\text{H}_{5/2} \rightarrow ^4\text{F}_{5/2}$ at 524, 500, 476, 468 and 455nm respectively²⁰. The electronic absorption spectrum of Pr(III) complex had traced bands at 590, 480, 468 and 444 nm assigned to transitions $^3\text{H}_4 \rightarrow ^1\text{D}_2$, $^3\text{H}_4 \rightarrow ^3\text{P}_1$, $^3\text{H}_4 \rightarrow ^1\text{F}_6$, and $^3\text{H}_4 \rightarrow ^3\text{P}_2$, respectively confirmed by its magnetic moment found to be 3.63B.M.²¹⁻²².

Table-2: HDMPpCB ligand electronic absorption spectral data and its Ln(III) metal complexes

Compound	λ_{nm}	$\epsilon (\text{dm}^3/\text{mol}/\text{cm})$	Transition
HDMHpCB	371	3458	$\eta \rightarrow \pi^*$
	243	9789	$\pi \rightarrow \pi^*$
	231	10555	$\pi \rightarrow \pi^*$
[Nd(DMTpCB) ₃]NO ₃	801	699	$^4\text{I}_{9/2} \rightarrow ^4\text{F}_{5/2}$
	741	2478	$^4\text{I}_{9/2} \rightarrow ^2\text{S}_{3/2}$, $^4\text{F}_{7/2}$
	679	2978	$^4\text{I}_{9/2} \rightarrow ^4\text{F}_{9/2}$
	582	6871	$^4\text{I}_{9/2} \rightarrow ^5\text{G}_{5/2}$
			$\beta_{\text{ave}} = 0.9547$, $\eta = 0.0247$, $b^{1/2} = 0.1163$, $\delta\% = 5.090$

	512	7314	$^4I_{9/2} \rightarrow ^4G_{9/2}$	
[Dy(DMTpCB) ₃]NO ₃	909	36	$^4H_{15/2} \rightarrow ^4F_{7/2}$	$\beta_{ave} = 0.9889,$ $\eta = 0.0056,$ $b^{1/2} = 0.0618,$ $\delta\% = 1.1352$
	806	105	$^4H_{15/2} \rightarrow ^4F_{5/2}$	
	755	347	$^4H_{15/2} \rightarrow ^4F_{3/2}$	
	474	1047	$^4H_{15/2} \rightarrow ^4F_{9/2}$	
	454	2145	$^4H_{15/2} \rightarrow ^4F_{15/2}$	
	420	3479	$^4H_{15/2} \rightarrow ^4G_{11/2}$	
[Sm(DMTpCB) ₃]NO ₃	524	2572	$^6H_{5/2} \rightarrow ^4F_{3/2}$	$\beta_{ave} = 0.9976,$ $\eta = 0.0012,$ $b^{1/2} = 0.0328,$ $\delta\% = 0.2393$
	500	3145	$^6H_{5/2} \rightarrow ^4G_{3/2}$	
	476	7589	$^6H_{5/2} \rightarrow ^4I_{9/2}$	
	468	7892	$^6H_{5/2} \rightarrow ^4F_{13/2}$	
	455	10475	$^6H_{5/2} \rightarrow ^4F_{5/2}$	
[Pr(DMTpCB) ₃]NO ₃	590	997	$^3H_4 \rightarrow ^1D_2$	$\beta_{ave} = 0.9976,$ $\eta = 0.00122,$ $b^{1/2} = 0.0244, \delta\% =$ 0.2452
	480	2978	$^3H_4 \rightarrow ^3P_1$	
	468	5789	$^3H_4 \rightarrow ^1F_6$	
	444	10578	$^3H_4 \rightarrow ^3P_2$	

Table-3 shows the FT-IR spectra of Ln(III) complexes. The HBMTpCB ligand's broad band at 3381 cm⁻¹ in its spectrum, which was ascribed to the oximino group's (-OH) after complexation, showed that the oximino group was taking part in coordination by deprotonation²³⁻²⁴. In the HBMTpCB ligand, the oximino (>C=NOH) and azomethine (>C=NN-) groups displayed intense stretching vibrations at 1528 and 1588cm⁻¹,

respectively. In the Ln(III) metal complexes, these two vibrations are displaced to lower frequencies²⁵⁻²⁷. This suggested that both oximino and aromethine groups were involved in coordination process through nitrogen atoms²⁸⁻²⁹. The non-ligand bands in the areas 514-522 and 543-604cm⁻¹ of the Ln(III) metal complexes were attributed to the (M-N) and (MN) vibrations, respectively³⁰⁻³¹.

Table-3: The ligand's (HDMTpCB) and its metal complexes' (cm⁻¹) FT(IR) spectral bands

Assignments	-OH	N-H	-CH ₃	-CH=	vC=NN	vC=NO	vC=S	vN→O	vPh-Cl	vM-N	vM→N
HDMTpBB	3381	3187	3066	2796	1588	1528	1176	1009	860	-	-
[Nd(BMTpCB) ₃]NO ₃	-	3199	3058	2840	1580	1480	1175	1075	859	556	515
[Dy(BMTpCB) ₃]NO ₃	-	3194	3109	2833	1571	1473	1211	1051	844	585	522
[Pr(BMTpCB) ₃]NO ₃	-	3202	3060	2833	1583	1489	1176	1090	859	543	515
[Sm(BMTpCB) ₃]NO ₃	-	3197	3055	2840	1573	1474	1175	1075	881	604	520

PMR spectra:

The sharp acidic peak at 11.60 ppm in the PMR spectrum of the HDMTpCB ligand was generated by the (OH) group. The multiplet signal of the HDMTpCB ligand in the PMR spectrum is formed by the characteristics of aromatic protons. A sharp singlet associated with the methine (-CH=) group was detected at 9.10 ppm for HDMTpCB.

X-Ray Diffraction:

X-ray diffraction of homo-binuclear metal complexes was scanned in the 2θ range of 10–80° range at 1.5406. The related diffractogram data showed the relative intensity, interplanar spacing, and each peak's 2θ value (d-values). Scherer's formula was used to calculate the average crystallite size (dxrd) of the metal complexes. Sharp crystalline peaks were visible in the XRD patterns, indicating the crystalline phase of metal

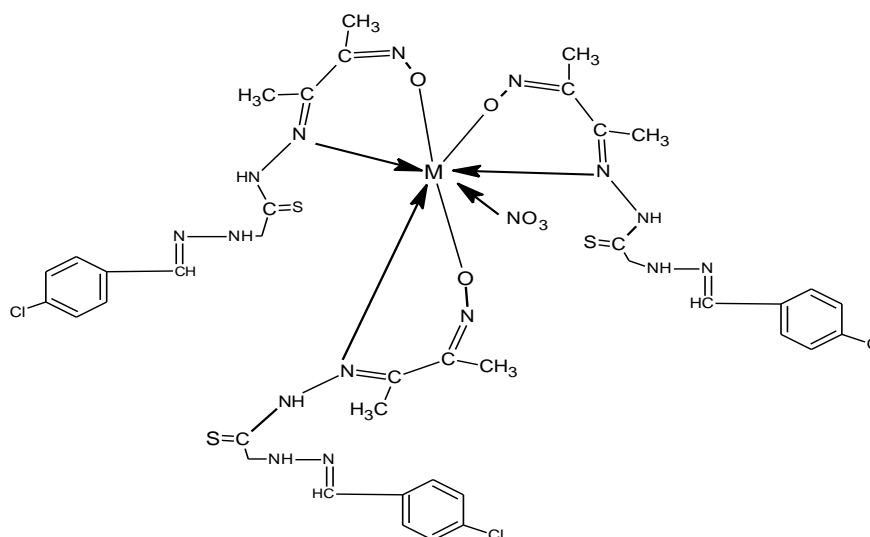
complexes³². The average crystallite size of the inner transition metal complexes is 2, 22, 46, and 42 nm, respectively. The found indices values for (hkl) were (222) for Nd(III), Pr(III), Sm(III) and Dy(III) complexes. For metal complexes, the computed lattice parameters are a=b=c=7.5, 7.04, 6.59, and 3.18. The reported values and found for the metal complexes may be associated with seven coordinated systems

Conclusion:

The HDMTpCB ligand and the lanthanide (III) ions are associated with the nitrogen, oxygen, and sulphur atoms of the higher carbohydrazide group, as evidenced by their geometry, physico-chemical properties, and spectrum data. In trinuclear lanthanide (III) complexes of the HDMTpCB ligand, the metal:ligand ratio is 1:3. The seven-coordinated geometry of all the HBMTpCB ligand

complexes that have been synthesised is in good agreement with the structure that has been

suggested for the effective lanthanide (III) complexes.



Where: Ln = Nd(III), Pr(III), Sm(III) and Dy(III)

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