



## Synthetic and Spectral studies of complexing behaviour of perchlorate complexes of 4[N-acetophenone amino] Antipyrine (C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O)

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### ABSTRACT

Pyrazolone and their substituted compounds have been reported to possess strong pharmaceutical properties, therefore, the metal complexes of these compounds should be of pharmaceutical interest. Some substituted pyrazolones have been found to show antifungal activities and complexation. It is expected that trivalent lanthanides form stable complexes with ligands having N donor atom because they behave as hard acid. Complexes were prepared by adding a methanolic solution of Lanthanide(III) perchlorate in a hot methanolic solution of ligands in (1:3) molar ratio. Characterization of newly synthesized complexes were done by using molecular weight, magnetic susceptibility, molar conductance, infrared and electronic spectroscopy.

**Keywords-** Lanthanide perchlorate complexes, pyrazolones, magnetic susceptibility, I.R and electronic spectroscopy.

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### INTRODUCTION

The coordination chemistry of pyrazole and 5-pyrazolone derived ligands has received much attention in recent years, primarily because of their biological importance [1, 2, 3]. It is well known that pyrazoles, pyrazolin-5-ones display pronounced antioxidant [4, 5, 6] antineoplastic activity [7, 8]. Some substituted pyrazolones have been found to show antifungal activities and complexation [9, 10, 11]. The coordination numbers shown by the tripositive lanthanide ion generally vary from six to ten. The geometry of various lanthanides(III) complexes involving different heteroatom were discussed by Koppikar et al.

The compounds having N, S and O-donor atoms are important because of their significant anticancer, antibacterial and antifungal activity [12]. It is well known that some drugs have increased activity when administered as metal complexes than as free organic compounds [13, 14].

Two conditions are necessary for a metal ion to form high coordination number, such as effective size of the metal ion and a high positive formal charge, which are full filled with Lanthanide ions and form six to ten coordinated complexes. According to HSAB theory Lanthanides belong to class-A metal (hard acid) group, in which the coordinating ability of ligands decreases along the series N>P>As; O>S>Se and F>Cl>Br hence the ligands containing N and O donor atoms are the best chelating agents.

The main object of the present work has therefore been to synthesis of some new Schiff base derived from 4-aminoantipyrine i.e. 4[(N-acetophenone) amino] antipyrine, and Isolation of solid complexes of Ln (ClO<sub>4</sub>)<sub>3</sub>; Ln=La, Pr, Nd, Sm, Gd, Tb and Dy) with these Schiff bases. Characterisation of the newly synthesized complexes by molar conductance, molecular weight magnetic susceptibility, infrared and electronic spectroscopy.

### MATERIAL AND METHODS

The lanthanide perchlorates were obtained by boiling the corresponding Lanthanide oxides with perchloric acid (A.R.) and evaporating off the excess of acid to get the coloured precipitate. The Schiff base was prepared by refluxing of 1 mole of aromatic carbonyl compound with 1 mole of 4-aminoantipyrine in 30 ml methanol for three hours on a water bath. After cooling yellow crystalline product was filtered and washed with methanol and ether and finally crystallised from ethanol and dried in vacuum desiccator.

**Preparation and isolation of [Ln (ClO<sub>4</sub>)<sub>3</sub>. 4(C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O)]**(Ln=La, Pr, Nd, Sm, Gd, Tb and Dy) (AAAP- C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O)

A methanolic solution of the lanthanide (III) perchlorate was treated with a hot methanolic solution of the ligand in (1:3) molar ratio. After refluxing the mixture on water bath for 3 hours. The solvent was partially distilled off. The contents cooled to room temperature solid precipitates form were suction filtered, washed with water, methanol and collected over anhydrous calcium chloride under vacuum desiccator.

**RESULT AND DISCUSSION****Characterization, Identification and confirmation of structure of complexes**

The chemical analysis of newly isolated compound are given in table1.

**Table 1 : Analytical, conductivity and molecular weight data of lanthanide (III) perchlorato complexes of 4[N(acetophenone)amino] antipyrine (C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O)**

S. N.	Complex	Found (calc.%)					$\Omega$ M (ohm <sup>-1</sup> cm <sup>2</sup> mole <sup>-1</sup> )	Electrolytic Nature	Average Mol. Wt.	Formula Weight
		Metal	C	N	Anion	H				
1	La(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	(8.38) 8.20	(55.03) 54.90	(10.14) 10.0	(18.01) 17.93	(4.59) 4.40	76.77	1:3 Electrolyte	412	1657.4
2	Pr(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	(8.49) 8.30	(54.96) 54.80	(10.12) 10.0	(17.99) 17.82	(4.58) 4.40	77.80	1:3 Electrolyte	413	1659.4
3	Nd(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	(8.67) 8.50	(54.85) 54.70	(10.10) 10.0	(17.95) 17.80	(4.57) 4.40	70.70	1:3 Electrolyte	416	1662.7
4	Sm(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	(9.01) 8.95	(54.65) 54.50	(10.07) 9.90	(17.89) 17.70	(4.55) 4.35	80.10	1:3 Electrolyte	415	1668.9
5	Gd(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	(9.39) 9.20	(54.42) 54.30	(10.03) 9.90	(17.81) 17.70	(4.53) 4.35	82.20	1:3 Electrolyte	417	1675.8
6	Tb(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	(9.47) 9.32	(54.37) 54.22	(10.02) 9.90	(17.80) 17.70	(4.53) 4.40	83.30	1:3 Electrolyte	418	1677.4
7	Dy(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	(9.67) 9.50	(54.25) 54.10	(9.99) 9.82	(17.76) 17.60	(4.52) 4.40	78.80	1:3 Electrolyte	419	1681.0

The isolated complexes are stable at room temperature and can be kept for many days without any decomposition. Prepared complexes do not have or lattice water molecule because no loss in weight was observed when complexes were heated up to 120-130°C. All the isolated complexes are soluble in organic solvent like acetone alcohol nitrobenzene as well as in dimethyl sulphoxide and dimethyl formaldehyde.

**Magnetic Measurement**

Gouy's method was used for the measurement of magnetic susceptibility using an electromagnetic field of  $8.5 \times 10^3$  gauss and a semi-micro mettler balance[15]. These measurement are recorded at room temperature. Lanthanides are widely differ from transition metal ion in their magnetic behaviour. The observed value of magnetic moment of these complexes are given in table 2.

**Table 2 : Permanent magnetic moments (B.M.) of Lanthanide ions and their complexes with 4-[(N-acetophenone) amino] antipyrine(C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O) at room temperature**

S. No.	Ln <sup>3+</sup>	Theoretical			Measured
		Hund	Van Vleck	R <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .8H <sub>2</sub> O	ClO <sub>4</sub> <sup>-</sup>
1	La	0.00	0.00	0.00	0.34
2	Pr	3.60	3.62	3.48	3.62
3	Nd	3.62	3.68	3.52	3.60
4	Sm	0.84	1.55	1.53	1.62
5	Gd	7.94	7.94	7.81	7.93
6	Tb	9.70	9.70	9.40	9.32
7	Dy	10.60	10.60	10.30	10.48

(I) Ln (ClO<sub>4</sub>)<sub>3</sub>.(C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O)<sub>4</sub>

Ln = La, Pr, Nd, Sm, Gd, Tb, Dy

The diamagnetic nature of lanthanum complexes are reveal in table 2 which was expected from their electronic configuration however Ln<sup>3+</sup> ions are paramagnetic because of 4f electrons .These 4f electrons

are effectively shielded by  $5s^2 5p^6$  electrons. These complexes obey simple curic equation but shows a little deviation Van Bleck values because of the crystal field splitting of orbitals.

### IR Spectra of the isolated complexes

The IR absorption frequencies of isolated complexes have been made by comparing their spectra with allied compounds such as antipyrine, 4 -amino antipyrine substituted 4- amino antipyrine[16,17] and mono substituted benzene ring. The infrared absorption frequencies are shown in table 3.

S. No.	Assignment	(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	La(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	Pr(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	Nd(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	Sm(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	Gd(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	Tb(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	Dy(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)
1	N-CH <sub>3</sub> (asym. And sym)	3295 m	3290 m	3295 m	3305 m	3305 m	3295 m	3290 m	3295 m
		3205 sh	3205 w	3295 w	3205 sh	3205 sh	3207 w	3204 w	3200 sh
		3190 m	3195 m	3195 m	3190 m	3190 m	3195 w	3190 m	3190 m
2	(C-H), (C=C), (C-N) Stretchings	3120 sh	3125 sh	3125 vw	3135 w	3135 w	3135 w	3135 w	3130 w
		3080 vw	3085 vw	3085 vw	3055 w	3055 w	3055 w	3056 w	3050 w
		3030 w	3035 w	3035 w	3025 sh	3025 w	3025sh	3025 sh	3024 sh
		3000 sh	3010 sh	3005 sh	2980 w	2985 w	2985 w	2985 w	2980 w
		2980 vw	2935 w	2930 w	2930 w	2935 w	2945 w	2940 w	2940 w
3	(C=N) Stretching(Azomethine)	1590 vs	1555 s	1565 s	1565 s	1565 s	1565 s	1560 m	1555 m
			1535 m	1535 m	1535 m	1540 w	1545 w	1530 sh	1540 w
4	C=C, C=N, Stretching and ring Vibration	1500	1495 s	1490 s	1495 s	1490 s	1490 s	1490 s	1500 s
		1450 sh	1455 m	1457 m	1450 m	1450 m	1435 m	1430 m	1440 m
		1460 sh	1435 m	1435 w	1435 w	1435 w	1410 sh	1410 sh	1410 sh
		1425 sh	1410 sh	1410 w	1405 w	1400 w	1406 w	1400 sh	1400 w
		1410 s							
5	Ring deformation	1365 s	1375 s	1370 s	1385 s	1375 s	1360 s	1370 s	1360 s
6	C-H in plane deformation	1300 vs	1305 sh	1305 m	1310 m	1295 m	1290 m	1290 m	1290 m
		1250 vs	1270 s	1250 s	1255 s	1255 s	1260 w	1250 s	1250 m
		1220 sh	1190 s	1195 m	1195 m	1130 m	1130 m	1130 m	1135 m
		1180 m	1110 s	1135 m	1130 m	1090 s	1110 s	1105 s	1100 s
		1165 w		1110 s <sup>a</sup>	1095 s <sup>a</sup>				
7	Ring breathing	1080 vw	1085 w	1085 vw	1085 vw	1080 vw	1080 vw	1080 vw	1080 vw
		1055 m	1050 m	1060 m	1060 m	1065 m	1060 m	1065 m	1060 m
		1020s	1020m	1030 m	1025 m	1020 m	1020 m	1025 m	1025 m
		1000 m		1010 m	1005 m	1005 m	1000 w	1000 w	1000 w
8	C-H out - of - plane deformation	955 m	970 w	980 w	970 w	970 w	975 w	975 w	985 w
		925 m	925 w	925 w	925 w	925 w	925 w	920 w	925 w
		840 m	870 w	880 m	880 m	880 m	875 m	875 m	875 m
		820 s	735 m	715 w	710 m	700 w	700 w	700 w	710 w
		770 w							
9	Out-of-plane ring deformation	695 m	690 m	700 m	700 m	705 m	700 m	700 w	700 w
		680 s	625 s <sup>a</sup>	625 s <sup>a</sup>	630 s <sup>a</sup>	635 s <sup>a</sup>	625 s <sup>a</sup>	625 s <sup>a</sup>	625 s <sup>a</sup>
10	ν (Ln-O) / ν (Ln-N) metal- ligand Vibrations	-	430 m	435 m	435 m	435 m	440 m	430 m	420 m
			385 w	375 w	380 w	380 w	380 w	385 w	365 w

### a - coupled with perchlorato absorptions

A regular tetrahedral ClO<sub>4</sub> ion has four fundamental vibrations, viz,  $\nu_1$  the  $a_1$ -type ClO stretch,  $\nu_2$  the e-type OClO bend (Both Raman @ active only)  $\nu_3$  the  $t_2$  type ClO stretch and  $\nu_4$  and  $t_2$  type OClO (both infrared and Raman active). Isolated ClO<sub>4</sub> ions show only two absorptions in the IR spectra due to  $\nu_3$  (at ca.

1100 cm<sup>-1</sup> and  $\nu_4$  at ca. 620 cm<sup>-1</sup>). When the T<sub>d</sub> symmetry is lowered to C<sub>3v</sub>, the triple degeneracy is removed and the  $\nu_1$  (at ca. 930 cm<sup>-1</sup>) and  $\nu_2$  (at ca. 460 cm<sup>-1</sup>) modes become infrared active.

The  $\nu_3$  and  $\nu_4$  bands in perchlorate complexes are summarized in Table 3. In all the complexes only two strong  $\nu_3$  and  $\nu_4$  bands are observed at 1110-1080 cm<sup>-1</sup> region and 630-620 cm<sup>-1</sup> region respectively, for perchlorate ion indicating that tetrahedral symmetry has not been disturbed on complexation, and the perchlorate ions are not bonded to metal ion [18]. This conclusion is also supported by conductance and molecular weight data.

S. NO.	Complex	$\nu_3$	$\nu_4$
	1	2	3
1.	La(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	1110 S	625 S
2.	Pr(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	1105 S	620 S
3.	Nd(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	1085 S	625 S
4.	Sm(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	1085 S	620 S
5.	Gd(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	1115 S	625
6.	Tb(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	1105 S	625
7.	Dy(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	1100 S	610 S

#### Electronic spectra of isolated complexes

The electronic absorption spectra of the isolated complexes have been recorded in aquas and non aquas solution as well as in solids. The weak narrow line-like bands in the UV-visible and near infrared region of the spectrum, arise due to Laporte forbidden internal transition within the 4f-levels. The crystal field splitting for the trivalent lanthanides are so small that the spectra of the compounds closely resemble with those of the free ion [19]. Due to external crystal field, the degeneracy in 4f orbitals is removed. The spectra reveal a shift of band towards lower energy as compared to aqua ion owing to nephelauxetic effect. The covalent parameter ( $\beta$ ) and bonding parameter ( $b_{1/2}$ ) are less than 1 while positive value of nephelauxetic parameter ( $\delta$ ) indicates a moderate covalent characteristic of a bond between metal and ligands.

$$\bar{\beta} = \frac{\nu(\text{complex})}{\nu(\text{aquo})}; b_{\frac{1}{2}} = \left[ \frac{1}{2} (1 - \bar{\beta}) \right]^{\frac{1}{2}}$$

$$\delta = \frac{100(1 - \bar{\beta})}{\beta}$$

The hyper sensitive band of complexes are similar to that of nine and eight coordinated complexes observed by Karkar. This suggests the coordination number of nine and eight around the metal ion in the complexes [20,21]. The electronic spectra data are shown in table 5.

**Table 5 : Electronic spectral data (cm<sup>-1</sup>) and related bonding parameters of lanthanide(III) perchlorate complexes of 4[N (acetophenone) amino] antipyrine (C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O)**

S. No.	Complexes	Ln(ClO <sub>4</sub> ) <sub>3</sub> Electronic Spectral Bands	Complex Electronic Spectral Bands	Energy levels	(1- $\beta$ )	$\beta$	$b_{1/2}$	$\delta\%$
	1	2	3	4	5	6	7	8
1	Nd(ClO <sub>4</sub> ) <sub>3</sub> (C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O) <sub>4</sub>	19600	19458	I <sub>9/2</sub> → <sup>2</sup> G <sub>9/2</sub>	0.00724	0.99276	0.06019	0.72927
		17380	17238	→ <sup>4</sup> G <sub>5/2</sub>	0.00817	0.99182	0.06392	0.82373
		13680	13620	→ <sup>2</sup> S <sub>3/2</sub> <sup>4</sup> F <sub>7/2</sub>	0.00439	0.99561	0.04683	0.44093
		12470	12410	→ <sup>4</sup> F <sub>5/2</sub> <sup>4</sup> H <sub>9/2</sub>	0.00481	0.99519	0.04905	0.48332
2	Sm(ClO <sub>4</sub> ) <sub>3</sub> (C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O) <sub>4</sub>	24870	24730	<sup>4</sup> H <sub>5/2</sub> → <sup>4</sup> F <sub>9/2</sub>	0.00563	0.99437	0.05305	0.56618
		24000	23680	→ <sup>6</sup> P <sub>5/2</sub>	0.01333	0.98666	0.08165	1.35101
		21550	21500	→ <sup>4</sup> I <sub>13/2</sub>	0.00232	0.99768	0.03406	0.232253

### Thermal study of isolated complexes

The thermal behaviour of perchlorate complexes however loss in mass correspond to two organic molecule noticed in the temperature range 290-350°C, and complete removal of the organic molecule occur at (400 – 480°C), finally consistent weight occur in the formation of metallic oxide. (~820°C).

**Table6:Thermoanalytical results of some lanthanide (III) perchlorate complexes of 4[(N-acetophenone) amino] antipyrine(C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O)**

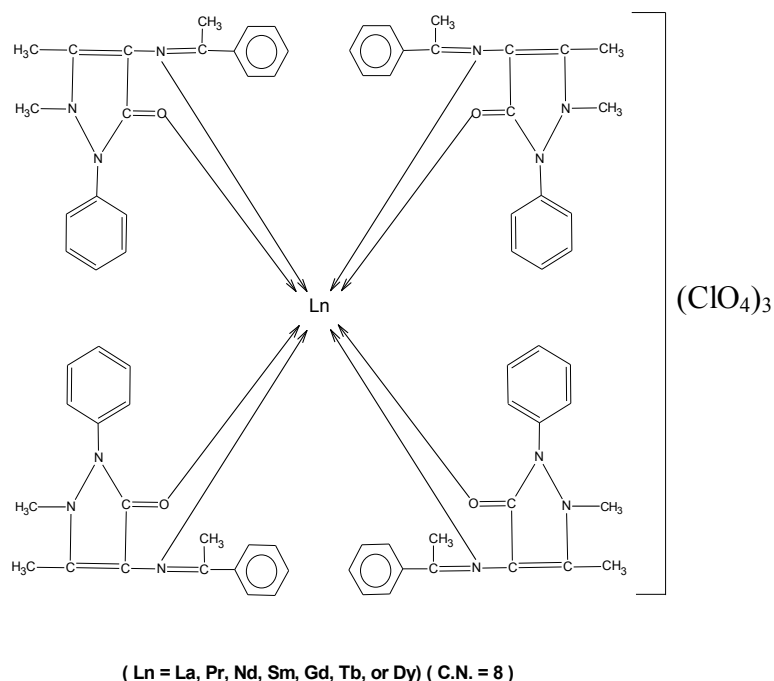
S. No.	Complex	Sample Weight (mg)	Residual Mass (mg)	Ligand mass loss %				Residual %	
				250 – 320°C		380 – 450°C		Ca. 800°C	
				Theor. <sup>a</sup>	Exp.	Theor. <sup>b</sup>	Exp.	Theor. <sup>c</sup>	Exp.
1	La(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	18	1.93	46.31	46.87	73.61	74.19	11.83	12.23
2	Nd(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	16	1.75	46.16	46.76	73.37	73.92	12.15	12.65
3	Gd(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	21	2.46	45.8	46.5	72.8	73.5	12.99	13.44
4	Dy(ClO <sub>4</sub> ) <sub>3</sub> .4(C <sub>19</sub> H <sub>19</sub> N <sub>3</sub> O)	14	1.68	45.66	46.31	72.58	73.08	13.35	13.85

a - Calculated for loss of 2.25 moles of ligand

b - Calculated for Total loss of ligand

c - Calculated as lanthanideoxide, La<sub>2</sub>O<sub>3</sub>,Nd<sub>2</sub>O<sub>3</sub>,Gd<sub>2</sub>O<sub>3</sub>, Dy<sub>2</sub>O<sub>3</sub>

After conclusion of all above result the following structure are suggested for the isolated complexes.



**Figure 1 : Structure of lanthanide (III) perchlorato complexes of 4[(N-acetophenone) amino] antipyrine (C<sub>19</sub>H<sub>19</sub>N<sub>3</sub>O)**

### CONFLICTS OF INTEREST

There are no conflicts of interest.

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