Synthesis, spectroscopic and biological studies of some lanthanide (III) nitrate complexes with 1,1'bis-(ortho amino phenyl thio)-methane

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Abstract

The complexes of some lanthanide of trivalent (La, Nd, Gd and Dy) and tetravalent metal ion (Zr) with 1,1'bis-(ortho amino phenyl thio)-methane (L) have been synthesis and identified by elemental analysis (C.H.N.), FT-IR, UV-Vis spectra, conductivity measurements, atomic absorption and magnetic susceptibility. The complexes showed characteristic behaviour of octahedral geometry around the lanthanide ions with the (N, N, S) ligand coordinated in tridentate mode and the metal ion (Zr) with the (N, S). α, K_f, ε_{max} for complexes (Zr, La, Nd and Gd) were estimated. The study of biological activity of the ligand (L) and its complexes showed activity toward *Staphylococcus laureus*, *Streptoccus pyogenes*, *Pseudomonas aeruginosa* and *E. coli*.

Key Words: Synthesis, lanthanides, complexes, rare earths, biological study.

Introduction

Metal chelates of orthoamino phenyl thio benzyl derivatives have evoked great interest due to their versatile application in various fields[1-3]. Among of ortho amino phenyl thiol derivatives, the coordination chemistry of methylene dichloride has attracted much attention by virtue of their applicability as potential ligands for a large number of lanthanides[4-8]. Compared to transition metals, lanthanides have much higher coordination numbers and more flexible coordination geometry, which lead to the formation of unusual multidimensional architectures[9]. There have been large research activities on coordination compounds of the lanthanide ions with organic ligands[10].

In this paper the synthesis, characterization and biological activities of (Zr^{III}, La^{III}, Nd^{III}, Gd^{III} and Dy^{III}) with 1,1'bis-(ortho amino phenyl thio)-methane are reported.

Materials and methods

a- chemicals; All reagents used were analar or chemically pure grade by British Drug Houses (BDA), Merk and Fluka.

The chemical materials,1,1'bis-(orthoaminophynyl thio)-methane ($C_{13}H_{14}N_2S_2$), zircouium sulphatetetrahydrate ($Zr(SO_4)_2.4H_2O$), Lanthanuim nitratehexahydrate ($La(NO_3)_3.6H_2O$), Neodymuim nitrate hexahydrate ($Nd(NO_3)_3.6(H_2O)$ Gadolinium nitratehexahydrate ($Gd(NO_3)_3.6H_2O$) and Dysprosium nitrate hexahydrate ($Dy(NO_3)_3.6H_2O$), Ethanol (CH_3CH_2OH) 99%, Dimethyl formanid (DMF) 99.9%, Dimethyl sulphoxide (DMSO) 99.5%, tetrachloro carbon (CCl_4) 99.5% chloroform ($CHCl_3$) 99%.

b-Instruments:

- Elemental analysis for the ligad (L) and complexe were determined by calibration type: liner Regression Euro EA Elemental analysis were maed in Al- Kufa University.
- Melting point were determined by Gallen- Kamp apparatus.
- IR spectra were recorded as KBr discs in the rang (4000- 400) cm^{-1} using shimadzu- FTIR.
- UV- visible spectra were recorded by shimadzu- UV- Vis- 160A ultra violet spectra photometer at 25°C, using 1 cm quartz cell and etamind at the range of (200-1100) nm at 10^{-3} M in DMSO.
- An Applied Research- Laboratories model (3410 – minitorch sequential Inductivity coupled plasma spectrometer) I.C.P. in Ibn- Sina State company, was used for the atomic emission spectroscopy measurements.
- Molar conductivity of the complexes were measured on pw 9526 digital conductivity in DMSO at 10^{-3} M.
- Magnetic susceptibility were recorded by magnetic susceptibility Blance, model, Ms B– MKl.

Preparation

Synthesis of the lanthanides complexes

The ligand of 1,1'bis-(orthoaminophenylthio)-methane (L) was prepared from their[11].

An ethanolic solution of lanthanide ions (one mole) was added to one mole of the ligand (L) as: [$Zr(SO_4)_2.4H_2O$(0.35 gm. 1.00 mmol), $La(NO_3)_3.6H_2O$ (0.43 gm. 1.00 mmol), $Nd(NO_3)_3.6H_2O$ (0.43 gm. 1.00 mmol), $Gd(NO_3)_3.6H_2O$ (0.45 gm. 1.00 mmol), $Dy(NO_3)_3.6H_2O$ (0.45 gm. 1.00 mmol) were added to (0.26 gm. 1.00 mmol) of the ligand (L). the mixture was sitirred for 30 min. the product was filtered and washed with distilled water and dried under the vacuum. Color, melting point, yield,

elemental analysis and solubility of the ligand and its complexes are given in (Table 1).

Table (1): Color, melting point, yield, elemental analysis and solubility for the ligand (L) and its complexes.

NO	Compound	no. of mole and gm	Color	m.p. C° or (dec.)	Yield %	Elemental analysis Found (calculated)				Solubility
						C%	H %	N %	M %	
1	$C_{13}H_{14}N_2S_2$(L)	1.00 0.26	Yellow	90-92 C°	80	59.431 (59.541)	5.331 (5.343)	10.662 (10.687)	-	EtOH, $CHCl_3$, CH_2Cl_2, CCl_4, DMF,DMSO
2	L-Zr$(SO_4)_2$	1.00 0.35	Light yellow	120-122 (dec)	94	28.593 (28.608)	2.557 (2.567)	5.133 (5.134)	16.792 (16.743)	DMF, DMSO
3	L-La$(NO_3)_3$	1.00 0.43	Green	86-88 C°	85	26.573 (26.575)	2.372 (2.385)	11.923 (11.925)	23.669 (23.679)	DMF, DMSO
4	L-Nd$(NO_3)_3$	1.00 0.43	Dark yellow	102-104C°	86	26.340 (26.342)	2.353 (2.364)	11.800 (11.820)	24.338 (24.349)	DMF, DMSO
5	L-Gd$(NO_3)_3$	1.00 0.45	Yellow	84-86 C°	93	25.767 (25.776)	2.312 (2.313)	11.556 (11.566)	25.973 (25.974)	DMF, DMSO
6	L-Dy$(NO_3)_3$	1.00 0.45	Yellow	88-90 C°	77	25.542 (25.552)	2.284 (2.293)	11.465 (11.466)	26.614 (26.617)	DMF, DMSO

dec.=Decomposition

Results and Discussion

The infrared spectrum of the ligand in the solid state does not contain the

υ(S–H) and which is present in starting material orthoamino phenyl thiol at 2500-2600 cm^{-1} region. This indicate the displacement of SH hydrogen orthoamino phenyl thiol by means of $–CH_2–$. Further more new bands at 2931 cm^{-1} due to υ_{asym} and 2869 cm^{-1} due to υ_{sym}. Bands in the region 3375 cm^{-1}, 3294 cm^{-1} and 1608 cm^{-1} are diagnostic of the primary amine[12-14]. Fig. (1).

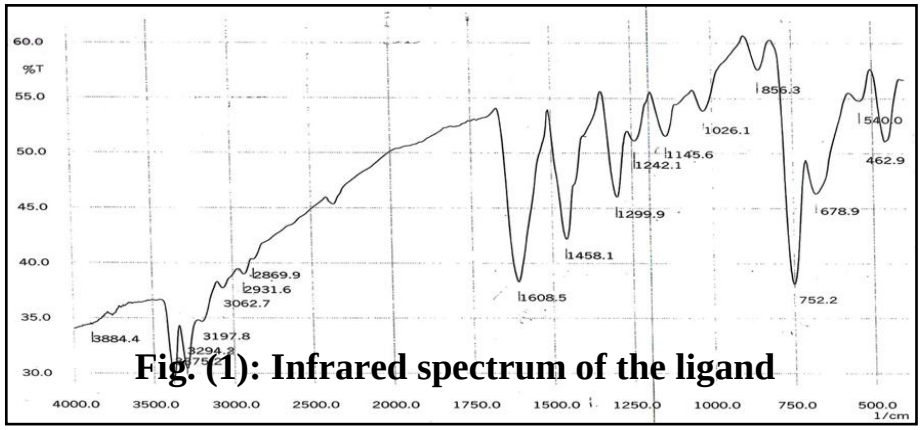


Fig. (1): Infrared spectrum of the ligand

The infrared spectra of all complexes showed, the multi bands in the range (3383-3298) cm^{-1} and split or broader band at (1610-1583) cm^{-1} and (1300- 1380) cm^{-1} with the formation of a new absorption bands for the

coordination bands (M–N) in the region (553-540) cm^{-1}, suggesting the coordination through nitrogen atom[15,16]. The free ligand (L) exhibits one S–CH_2 at 1299 cm^{-1}, 1026 cm^{-1} and 678 cm^{-1} in the crystalline state, where as all complexes exhibits change in shape and position of S–CH_2 rocking with M–S stretching vibration has been observed at Table (2)[17-19]. These observation can be related to coordinates S–CH_2 to all ions used.

The Zr^{+4} complex showed band at 1024 cm^{-1}, 972 cm^{-1}, and 694 cm^{-1}, are due to coordinate sulphat[20]. The La^{+3}, Nd^{+3}, Gd^{+3}, Dy^{+3} complexes spectrum exhibit bands at 920 cm^{-1}, 673 cm^{-1} due to coordinate nitrato ion[21-22]. Table (2) showed characteristic stretching vibration frequencies (cm^{-1}) located in the FT-IR of the ligand and its complexes. Fig. (2).

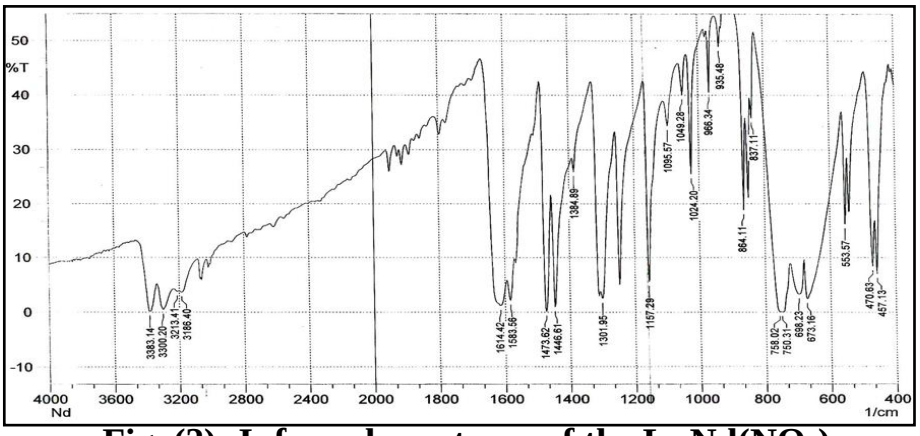


Fig. (2): Infrared spectrum of the L- $Nd(NO_3)_3$.

Table (2): characteristic stretching vibration frequencies (cm^{-1}) located in the FT-IR of the ligand and its complexes

No.	Compound	υ(N-H) cm^{-1}	υ(C-H) cm^{-1} Ar.	υ(C-H) cm^{-1} Ali.	υ(C-S) cm^{-1}	υ(Ln-N) cm^{-1}	υ(Ln-S) cm^{-1}	υSO_4^{-2} υNO_3^- cm^{-1}
1	$C_{13}H_{14}N_2S_2$(L)	3375(asym)m.br 3294(sym)m.br 1608(S.S) 1300(sh)	3197(m.s) 3062(m.s)	2931(asym)m.s 2869(sym)m.s 1458(S.S)	1299(s.s) 1026(m.s) 678(m.s)	–	–	–
2	L-$Zr(SO_4)_2$	3379(m.br) 3300(m.br) 3200(w.s) 1301,1311(s.s)	319(w.s) 3062(w.s)	2980(w.s) 2810(w.s) 1473(s.s) 1446(s.s)	1247(s.s) 1030(s.s) 694(s.s) 669(s.s)	550(m.s) 542(m.s)	470(s.s) 457(s.s)	1024(m.s) 972(m.s) 674(m.s)
3	L-$La(NO_3)_3$	3377(m.br) 3300(m.br) 1620(s.s) 1583(m.s) 1384,1340(m.s) 1311(m.s)	3160(m.s) 3020(w.s)	2980(m.s) 2880(m.s) 1473(s.s) 1446(s.s)	1246(s.s) 1024,1043(s.s) 694(s.s)	553(m.s) 540(m.s)	470(s.s) 457(s.s)	920(m.s) 671(m.s)
4	L-$Nd(NO_3)_3$	3383(m.br) 3300(m.br) 3213(m.br) 1301,1310(s.s)	3186(w.s) 3060(m.s)	2990(m.s) 2850(w.s) 1473(s.s) 1446(s.s)	1246(s.s) 1029(s.s) 1024(s.s) 698(s.s)	553(m.s) 540(m.s)	470(s.s) 457(s.s)	935(m.s) 673(m.s)
5	L-$Gd(NO_3)_3$	3377(m.br) 3298(s.s) 1610(s.s)	3182(m.s) 3062(w.s)	2950(m.s) 2890(m.s) 1473(s.s)	1246(s.s) 1025(s.s) 1093(m.s)	554(m.s) 542(m.s)	462(s.s) 457(s.s)	933(m.s) 671(m.s)

		1583(s.s) 1320,1300(s.s)		1446(s.s)	696(s.s)			
6	L-Dy$(NO_3)_3$	3379(s.s) 3298(s.s) 1612(s.s) 1583(s.s) 1301,1311(s.s)	3180(m.s) 3062(m.s)	2980(s.s) 2877(m.s) 1473(s.s) 1446(s.s)	1247(s.s) 1024(s.s) 1095(s.s) 671(s.s)	553(s.s) 540(s.s)	470(s.s) 457(s.s)	933(s.s) 671(s.s)

Ar = aromatic, asym = asymmetric, sym = symmetric, sh=shoulder br=broad, m=medium, s=strong, w=weak

The Uv – Visible spectra of the ligand (L) and complexes

The UV-visible spectra of the ligand (L) in DMSO solution exhibited strong absorption bands at (266 nm, 37593 cm^{-1}) and (356 nm, 28089 cm^{-1}), (348 nm, 28735 cm^{-1}). This may attributed to the π–π* and n–π* transition.

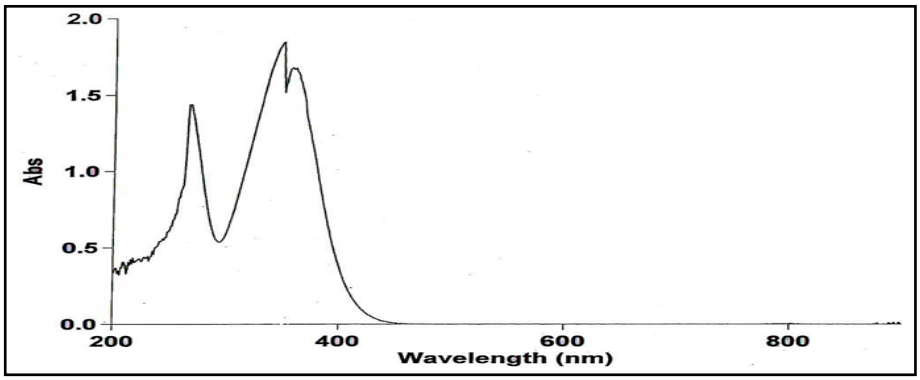


Fig. (3): UV- Visible spectrum of the ligand (L)

The UV-visible spectrum for Zr^{+4} complex showed one band in the region (415 nm, 24096 cm^{-1}) is due to the charge transfer UV-visible spectrum for La^{+3} complex showed one band in the region (500 nm, 20000 cm^{-1}), Nd^{+3} complex showed one band in the region (429 nm, 23310 cm^{-1}), Gd^{+3} complex showed two bands at (510 nm, 19607 cm^{-1}), (416 nm, 24038 cm^{-1}) and Dy^{+3} complex appeared one band at (560 nm, 17857 cm^{-1}) this due to F–F transition[23,24].

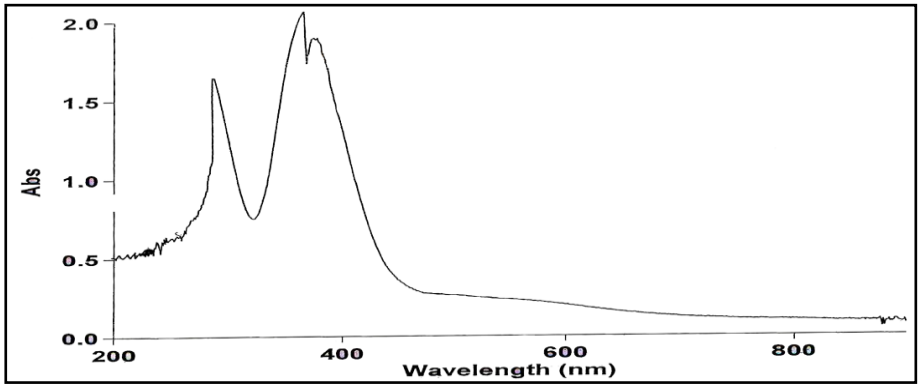


Fig. (4): UV- Visible spectrum of L- Gd$(NO_3)_3$

The moler conductance of all complexes in DMSO was found to be low which suggested coordination of anion to the metal[25].

The magnetic moment values of the complexes are presented in Table (3). Lanthanum (III) complex was diamagnetic while all other complexes were paramagnetic. Paramagnetic behavior of the complexes was consistent with the presence of unpaired electrons.

The observed magnetic moment values of the complexes were in good agreement with the calculated values of the corresponding lanthanide (III) ions. This indicates the minor participation of 4 electrons in band formation. Electronic spectra, conductance in (DMSO), magnetic moment (B.M) of the ligand and its complexes given in Table (3).

Table (3): Electronic spectra, conductance in (DMSO), magnetic moment (B.M) of the ligand (L) and its complexes

No.	Compound	$\lambda_{(nm)}$ cm^{-1}	Λ s. cm^{-1} DMSO (10^{-3} M)	μ_{eff} (B.M)
1	$C_{13}H_{14}N_2S_2$(L)	37593(266) 28089(356) 28735(348)	–	–
2	L-Zr$(SO_4)_2$	24096(415)	11.2	3.25
3	L-La$(NO_3)_3$	20000(500)	13.67	D*
4	L-Nd$(NO_3)_3$	23310(429)	12.57	3.65
5	L-Gd$(NO_3)_3$	24038(416) 19607(510)	11.35	7.86
6	L-Dy$(NO_3)_3$	17857(560)	9.62	6.73

D* Diamagnetic, B.M = Bohr magneton

Study of Zr^{+4}, La^{+3}, Nd^{+3} and Gd^{+3}

The complexes of the ligand (L) with selected metal ions (Zr^{+4}, La^{+3}, Nd^{+3} and Gd^{+3}) were studied in solution using ethanol as solvent, in order to determine (M:L) ratio in the prepared complexes, following molar ratio method[26]. A series of solutions were prepared having a constant (C) 10^{-3} M of the hydrated metal salts and the ligand (L). the (M:L) ratio was determined from the relation ship between the absorption of the observed light and the mole ratio (M:L) found to be (1:1) Fig. 10. The results of complexes formation in solution are given in Table (4, 5).

V_M= volume of metal in ml
V_L= volume of ligand in ml

Table (4): Continuous variation slop for Zr^{+4} ion λ (415 nm)		
L-$Zr(SO_4)_2$		
V_M	V_L	Abs
1 ml	0.25	0.23
1	0.50	0.43
1	0.75	0.69
1	1	0.90
1	1.25	0.91
1	1.50	0.89
1	1.75	0.92
1	2	0.91
1	2.25	0.93
1	2.50	0.89
1	2.75	0.91
1	3	0.89
1	3.25	0.92
1	3.50	0.92
1	3.75	0.89
1	4	0.91

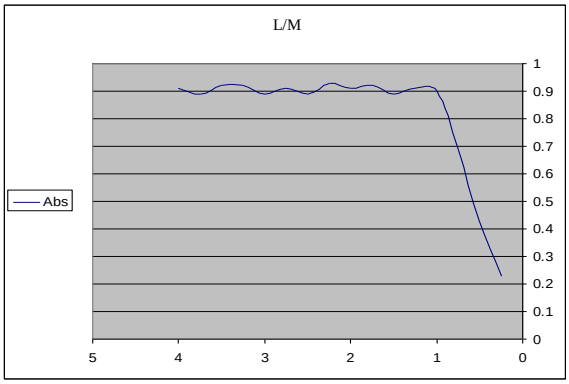


Fig. (5): Continuous variation slop for Zr^{+4} ion λ (415 nm)

Table (5): Continuous variation slop for La^{+3} ion λ (339 nm)		
L-$La(NO_3)_2$		
V_M	V_L	Abs
1 ml	0.25	0.22
1	0.50	0.41
1	0.75	0.45
1	1	0.50
1	1.25	0.51
1	1.50	0.51
1	1.75	0.49
1	2	0.48
1	2.25	0.52
1	2.50	0.51
1	2.75	0.49
1	3	0.52
1	3.25	0.51
1	3.50	0.49
1	3.75	0.51
1	4	0.49

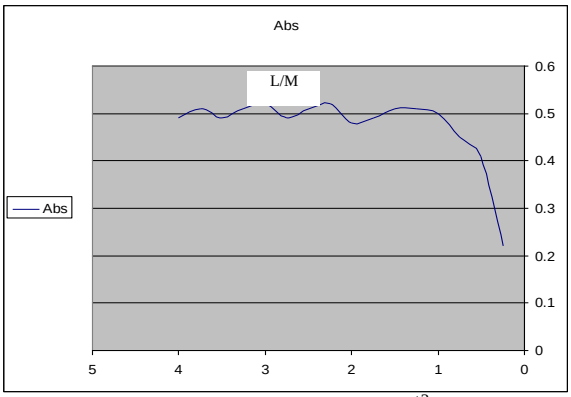


Fig. (6): Continuous variation slop for La^{+3} ion λ (339 nm)

Stability constant (Kf) of (1:1) (M:L) complex was evaluated using the following equations (1), (2)

$Kf=1-\alpha/\alpha^2C$……(1)

$\alpha=A_m-As/A_m$……(2)

(α) is the degree of the dissociation, (C) is the concentration values of the solution were measured at (λ_{max}) of the maximum absorption. The molar absorptivity (ε_{max}) (eq.3) has been calculated using equation;

$A= \varepsilon_{max}.b.c$…..(3)

(A) is: the average of three measurement of the absorption containing the same amount of metal ion and three fold excess of ligand, (b) is the depth of the quartz cell usually equal cm.

Table (6): As, Am, K_f, ε_{max} and λ_{max} of the Zr^{+4}, La^{+3} Nd^{+3} and Gd^{+3} complexes

No.	Compound	As	Am	α	Formation constant (Kf)	Molar absorpitivity ε_{max} $L.mol^{-1}.cm^{-1}$	λ_{max} (nm)
1	Zr-complex	0.90	0.93	0.051	9.462×10^5	4321	415
2	La-complex	0.50	0.52	0.013	5.840×10^6	1983	339
3	Nd-complex	0.60	0.63	0.047	4.314×10^5	4702	350
4	Gd-cmplex	1.50	1.52	0.013	5.840×10^6	1058	348

As= The absorbance value of the solution when the VM=VL =1
Am= High the absorbance value of the solution.

The atomic absorption analysis was used to confirm our molar ratio calculation of [metal: ligand] (M:L) for synthesis complex as well. The results showed a ratio (M:L) (1:1) for all complexes Table (1).

Biological activity study:

The biological activity of the prepared ligand and its complexes were studied against selected types of micro organisms which include grame positive bacteria like *streptococcus pyogenes*, *staphylococcus* and gram negative bacteria like *E. coli*, *pseudomonas aeruginosa* in agar diffusion method, which is used (DMSO) as a solvent, and we are used these antibiotics disc which include Amoxicillin and cephalosporin as control. Agar diffusion method involves the exposure of the zone of inhibition toward the diffusion of micro organisms on agar plate. The plates were incubated for [24] hrs. at (37C°), the zone of inhibition of bacterial growth around the disc was observed. Table (8), (9).

Table (8): Effect of ligend and its complexes on gram positive bacteria.

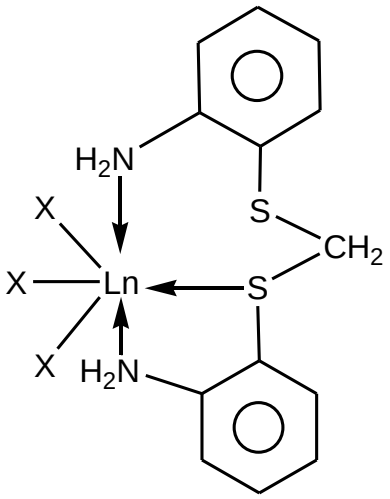
Ligand or complexes	Diameter of inhibition zone (mm) at concentration pyogenes 1 mg/ml		Diameter of inhibition zone (mm) at concentration 5mg/ml	
	Streptococcus pyogenes	Staphylococcus aureus	Streptococcus pyogenes	Staphylococcus aureus
Amoxicillin	28.9	27.2	29.3	28.5
$C_{13}H_{14}N_2S_2$(L)	16	16.2	16.4	16.2
L-$Zr(SO_4)_2$	16.8	16.5	18.2	17.8
L-$La(No_3)_3$	11.2	11.5	12.2	12.6
L- $Nd(NO_3)_3$	17.4	17	18.4	17
L- $Gd(NO_3)_3$	15	14.8	15.2	16
L-$Dy(NO_3)_3$	14.8	14	15.6	14.2

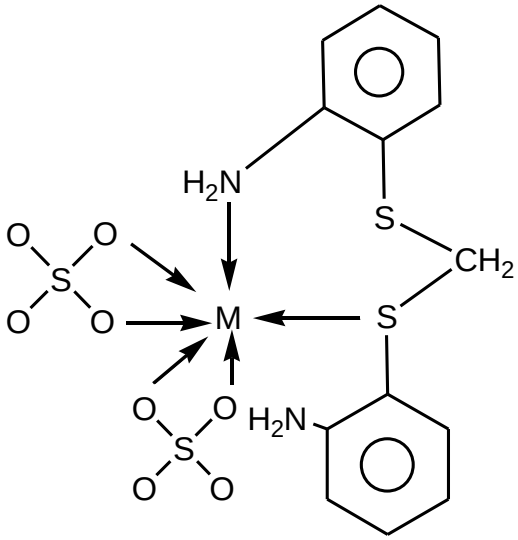
Table (9) Effect of ligand and its complexes on gram negative bacteria.

Ligand or complexes	Diameter of inhibition zone (mm) at concentration 1 mg/ml		Diameter of inhibition zone (mm) at concentration 5mg/ml	
	E. Coli	Pseudomonas aeruginosa	E. Coli	Pseudomonas aeruginosa
Cephalosporin	25.4	26.8	28.6	27.2
$C_{13}H_{14}N_2S_2$(L)	16.6	16.4	17.2	14.2
L-$Zr(SO_4)_2$	12.6	12	13.4	14.2
L-$La(NO_3)_3$	15	14.8	15.2	16
L-$Nd(NO_3)_3$	14.8	14.6	15.6	15
L-$Gd(NO_3)_3$	10.2	10	9.6	10.2
L-$Dy(NO_3)_3$	9.2	9	10.2	10.4

Conclusion

A series of complexes of Zr^{+4}, La^{+3}, Nd^{+3}, Gd^{+3}, Dy^{+3} with 1,1'bis-ortho aminophenyl thio) methane (L) have been prepared and characterized. The tridentate ligand (L) (N,N,S) is binding lanthanide ions forming octahedral structure and the bidentatet ligand (L) (N,S) is binding metal ions formaing octahedral structure as follow:





Ln^{+3} = La, Nd, Gd, Dy
$X = NO_3^{-1}$

M^{+4} = Zr

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تحضير، دراسة طيفية وبايولوجية لبعض

نترات اللانثانات (مع 1،1 ثنائي-(اورثوامينو فنيل ثايو) ميثان

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الخلاصة:

تم تحضير معقدات بعض اللانثانات ثلاثية التكافؤ (اللانثانيوم، النيودميوم، كادولينيوم والديسبروسيوم) (الزركونيوم) مع 1،1¯- ثنائي- (اورثو امينو فنيل ثايو)- ميثان وتم تشخيصها بتحليل العناصر (كاربون، هيدروجين، نتروجين) واطياف الأشعة تحت الحمراء والبنفسجية- المرئية، قياسات التوصيلية الكهربائية، الامتصاص الذري اللهبي والخواص المغناطيسية واستنتج من التحاليل ان المعقدات لها شكل ثماني السطوح حول ايونات اللانثانات من تناسق اللكاند من خلال (S, N, N) وايون ثنائي التكافؤ وحول ايون الفلز (Zr) من تناسق اللكاند من خلال (S, N).

وقد تم حساب قيم α، K_f، ε_{max}، لمعقدات الزركونيوم، اللانثانيوم، النيودميوم، وكادولينيوم، كما تم دراسة الفعالية البايولوجية لليكاند ومعقداته وقد أظهرت النتائج امتلاكها فعالية اتجاه أنواع البكتريا *Streptococcus py ogenes*, *staphylococcus laureus*, *pseudomonas aeruginosa* و *E. Coli*.