International Journal of Chemical Studies

P-ISSN: 2349–8528 E-ISSN: 2321–4902 www.chemijournal.com IJCS 2021; 9(3): 339-345 © 2021 IJCS Received: 08-03-2021 Accepted: 13-04-2021

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Synthesis of La (III), Ce (III), Sm (III), and Nd (III) inner transition metal complexes of Thiazole ring as a ligand: Spectral, anti-bacterial and antifungal studies

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Abstract

The study of 16 new lanthanide (III) nitrate complexes with Schiff base ligands is discussed. Schiff bases were created by condensing 2-amino-4-(4-bromophenyl) thiazole with ortho hydroxy aldehyde and 3 different substituted ortho hydroxy aldehydes. Lanthanide (III) nitrates such lanthanum (III), cerium (III), neodymium (III), and samarium (III) nitrate were used to make new complexes. The complexes were characterised using physicochemical tests, including elemental analysis, spectrum analysis in the IR and UV-Vis, and fluorescence examinations. The lanthanide (III) nitrate complexes were also screened as part of the anti-bacterial and anti-fungal investigations.

Keywords: Schiff bases, Lanthanide (III) Nitrate complexes, anti-bacterial and anti-fungal studies and Spectral studies

Introduction

The most distinctive area of inorganic chemistry is coordination chemistry. It has improved understanding of test methodology, auxiliary examinations, coordination mix holding characteristics, and hypothetical administration. Since they were first recognised in the eighteenth century, coordination compounds have been a unique test for inorganic scientific experts. As more buildings were discovered, several theories were put forth to support their development and, moreover, their properties.

Metals are thought to have a wide variety of coordination properties that are significant in biology, industry, and medicine. Many coordination complexes with antibacterial properties are known at the moment.

Due to their intriguing physicochemical characteristics and strong biological activity, metal complexes of N and S chelating ligands have received a lot of attention. At the active sites of several metallobimolecules, the coordination of metals by the N and S atoms is crucial. O-vanillin-containing Schiff bases have antifungal and antibacterial effects ^[1], function as a weak tyrosinase inhibitor, and exhibit both antimutagenic and comutagenic capabilities in Eccherichia coli Andropogen nardus contains the natural aldehyde known as ovanillin. It is used in spice making as well as to relieve stomachaches ^[2, 3]. Several chemical compounds that are pharmacologically and physiologically active comprise heterocycles with thiazole rings.

Benzothiazole and sulphonamide derivative-containing compounds have been utilised therapeutically and as anti-inflammatory ^[6], antifungal ^[4, 5], antithyroid ^[11], anticancer ^[8], anticarbonic anhydrase ^[9], and antimalarial agents. Due to these chemicals' prominent biological actions,

Lanthanides, also known as lanthanons, make up the longest series in the periodic table. There are four in the transition series. Lanthanide (III) ions are the best ions for creating stable complexes with high coordination numbers because of their size and charge ^[12–17]. Lanthanide ions with coordination numbers 6 to 10 are said to exist in lanthanide coordination compounds. The contributions of Schiff base metal complexes have greatly advanced coordination chemistry ^[18–22]. In this article, we present lanthanide (III) nitrate compounds that contain several of the Schiff base ligands shown in Figure 1.

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Fig 1: Structure of Schiff base ligands L1 to L4.

Experimental

Materials and instrumentation

Fourier Transform Infrared (FT-IR) spectra were recorded on a Shimadzu model 8400s FT-IR spectrometer scanning at range between (4000-400 cm-1) via a KBr pellets. All solvents, organic chemicals, and inorganic salts those used in this work were all highly purified and directly used without any further purification. A Shimadzu UV-1650 UV-Visible spectrophotometer was used to measure UV-Visible spectra. Absolute ethanol was used as the measuring solvent during a double beam scan between 200 and 1100 nm. OMEGA Digital Melting Point Apparatus model MPS10-120 was used to measure the melting points in open capillary tubes.

Synthesis of ligand (L)

The following approach (Scheme 1) was described by Furniss BS, Hannaford AJ, Smith PWG, and Tatchell AR in Vogel's

Practical Organic Chemistry, Fifth Edition (Longman Scientific Technical, John Wiley and Sons), 1989. ^[23] 5-Substituted Ortho hydroxy aldehydes were dissolved in 25 ml of methanol and added to a 25 ml solution of 2-amino-4-(4-bromophenyl) thiazole in a 100 ml round bottom flask. The reaction mixture was then refluxed on a water bath for about 4 hours. After the reaction time was up, the reaction mixture was allowed to cool for a short period at room temperature, and as it did, a crystalline solid with a pale-yellow colour emerged. It was filtered, cleaned with methanol, recrystallized from DMF and DMSO, and dried at room temperature while under decreased pressure.

By using melting point, elemental analysis, and TLC, the purity of the ligand was examined. Mass, IR, 1H, 13C, UV, and fluorescence spectral analyses were also used to characterise it. The Yield was 72.01% as well. In Scheme 1, the process for making ligand (L) is shown.



Scheme 1: Synthesis of thiazole ring containing Schiff base ligands.

Physical properties of Schiff base ligands

Table No. 1 below contains analytical data as well as physical Ligand characteristics. Several Orth hydroxy aldehydes can combine to produce ligands that are coloured yellow or brown. All of the ligands are soluble in organic solvents such CHCI3, DMSO, and DMF but insoluble in water. The yield of all ligands is roughly equivalent to 72%, as shown in table

No. 1, and this is positive. All of the ligands' decomposition points are within a narrow range of 160°C-187°C, indicating strong thermal stability under typical circumstances. And ligands are allocated an empirical formula based on elemental analysis. Digital melting point equipment was used to record the melting point.

Table 1: Physical	parameters of Synthesized	l Schiff base ligands L1 To L4.
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Sr.	Molecular formula of	M.P.	% Yield	Colour And Solubility		Molecular	E	leme	ental	lana	lysis	in 9	%
No./ligand	Compound	in °C	in GM	Colour And Solubility		Weight.	С	Η	Ν	S	0	CI	Br
1/L1	C16H11N2OSBr L1	168-170	72.01	Yellow DMF, DMSO, CHCI3	0.66	359.24	53.49	3.08	7.79	8.92	4.45	-	22.24
2/L2	C16H10N2OSCIBr L2	170-174	67.68	Pale brown DMF,DMSO CHCI3	0.65	393.69	48.81	2.55	7.11	8.14	4.06	9.00	20.29
3/L3	C17H13N2OSBr L3	170	66.66	Whitish, Yellow DMF,DMSO CHCI3	0.64	373.27	54.70	3.50	7.50	8.58	4.28	-	21.40
4/L4	C17H13N2O2SBr L4	162-165	67.60	YELLOW CHCI3	0.62	389.27	52.45	3.36	7.19	8.23	8.21	-	20.52

Preparation of metal complexes

In order to prepare the metal complexes, 0.01 mol of cerium (III) nitrate is added to 0.02 mol of hot methanolic ligand solution with steady stirring at 7-8 pH for approximately 4 or less than 4 hours. After the action combination had reached room temperature, distilled water was added. The coloured

particles were separated and collected by filtering before being washed with hot ethanol, dried in a hoover over anhydrous calcium chloride, and then dried in a desiccator ^[24]. The pathway for the synthesis of Metal Complexes is presented in Scheme 2.



Scheme 2: Synthesis of Metal Complexes.

Results and Discussion

Complexes are created when the lanthanide (III) nitrates lanthanum (III) nitrate, cerium (III) nitrate, samarium (III) nitrate, and neodymium nitrate (III)—interact with Schiff base ligands. M = La, Ce, Sm, and Nd.

Table 2 below lists the physical characteristics of metal complexes, and Table 3 provides an elemental analysis of these complexes. Complexes that have just been created are generally stable and long-lasting. They are all non-hygroscopic and adequately soluble in ordinary organic solvents. In a PhNO2 media, these complexes exhibit almost low electrical conductivity, demonstrating their non-electrolytic nature.

This fact is also supported by the complexes' molecular weight information. Due to the existence of 4f-electrons, which are effectively protected by 5s2p2 electrons, lanthanum (III) nitrate complexes are basically diamagnetic in nature, whereas all other complexes are paramagnetic ^[25]. This demonstrates that 4f-electrons are not involved in the creation of the bond ^[26].

Physical parameters of the metal complexes

The metal complexes, which include La, III Ce, III Nd, and

Sm III, are crystalline with a high melting point and are soluble in organic solvents. They have a whitish and creamcolored appearance. They have a paramagnetic makeup.

Table 2: Physical parameters of the metal complexes

Sr. No.	Com p	M.P. in °C	% Yield in gm	Colour	solubility	Rf Value	Molecula r weight
1	C1	290 °C	88.88	Creamish	DMF,DMSO	0.59	857.39
2	C2	> 300 °C	76.41	Creamish	DMF,DMSO	0.57	926.28
3	C3	>295 °C	88.23	Whitish	DMF,DMSO	0.60	885.45
4	C4	> 300 °C	67.14	Creamish	DMF,DMSO	0.52	917.45
5	D1	> 300 °C	85.71	Creamish	DMF,DMSO	0.55	858.60
6	D2	>295 °C	88.67	Whitish	DMF,DMSO	0.58	927.49
7	D3	> 300 °C	72.50	Creamish	DMF,DMSO	0.59	886.66
8	D4	> 300 °C	65.07	Creamish	DMF,DMSO	0.53	918.66
9	E1	> 300 °C	61.53	Creamish	DMF,DMSO	0.56	862.73
10	E2	>295 °C	55.35	Whitish	DMF,DMSO	0.58	931.62
11	E3	>299 °C	58.00	Creamish	DMF,DMSO	0.58	890.78
12	E4	>300 °C	70.25	Creamish	DMF,DMSO	0.54	922.78
13	F1	> 300 °C	65.18	Creamish	DMF,DMSO	0.53	868.85
14	F2	> 300 °C	67.23	Creamish	DMF,DMSO	0.59	937.74
15	F3	$> 300 \ ^{\circ}\mathrm{C}$	80.74	Creamish	DMF,DMSO	0.55	896.90
16	F4	> 300 °C	85.57	Creamish	DMF,DMSO	0.56	928.90

Elemental analysis of the metal complexes

The below table shows the elemental analysis for all newly synthesized metal complexes

Table 3: Elemental analysis of the metal complexes

Sr. No.	Comp	La	С	Η	Ν	S	0	CI	Br
1	C1	16.20	44.82	2.58	6.53	7.47	3.73	-	18.63
2	C2	14.99	41.49	2.17	6.04	6.92	3.45	7.65	17.25
3	C3	15.68	46.12	2.95	6.32	7.24	3.61		18.04
4	C4	25.13	44.51	2.85	6.10	6.99	6.97		17.41
Sr.no	Comp	Ce	С	Н	Ν	S	0	CI	Br
1	D1	16.31	44.78	2.58	6.52	7.46	3.72		18.61
2	D2	15.10	41.43	2.17	6.03	6.91	3.44	7.64	17.22
3	D3	15.80	46.05	2.95	6.31	7.23	3.60	-	18.02
4	D4	15.25	44.45	2.85	6.09	6.98	6.96	_	17.39
Sr.no	Comp	Nd	С	Н	Ν	S	0	CI	Br
1	E1	16.71	44.55	2.56	6.49	7.43	3.70		18.52
2	E2	15.48	41.26	2.16	6.01	6.88	3.43	7.61	17.15
3	E3	16.19	45.84	2.94	6.28	7.19	3.59	-	17.93
4	E4	15.63	44.25	2.83	6.07	6.94	6.93		17.31
Sr.no	Comp	Sm	С	Н	Ν	S	0	CI	Br
1	F1	17.30	44.23	2.55	6.44	7.38	3.68		18.39
2	F2	16.03	40.98	2.40	5.97	6.83	3.41	7.56	17.04
3	F3	16.76	45.53	2.92	6.24	7.15	3.56	_	17.81
4	F4	16.18	43.96	2.82	6.03	6.19	6.88	_	17.20

IR spectra of the ligands and their complexes

Determine the site of coordination that may be involved in chelation by comparing the infrared spectra of the produced complexes with those of the free ligands (Table No. 4). The spectra of the ligands contain some guiding peaks that are helpful in reaching this objective. These peaks should play a role in chelation. The OH groups of these Schiff bases are implicated in intramolecular hydrogen bonding, as evidenced by the broad band at 3430-3281 cm1 of the stretching vibrations of phenolic OH groups in the IR spectra of the ligands ^[27-28]. On the other hand, the broadbands in the IR spectra of the complexes about 3425-3417 cm1 are caused by the presence of v(OH) water molecules; this conclusion is also supported by elemental and thermal analysis. The fact that this band shifts to a higher frequency in all of the complexes following deprotonation due to coordination suggests that the hydroxyl groups of the Schiff bases coordinate with Ln in the C-O-Ln link. Strong O-H stretching bands can be seen in the IR spectra of the Schiff bases L1, L2, L3, and L4 at and 3427, 2723, and 2725 cm-1, respectively ^[29], and the stretching vibration of the azomethine group can be seen in the IR spectra of the three Schiff bases at and 1628, 1630, and 1630 cm-1, respectively [30].

The >C=N double bond most frequently seen in Schiffs bases (azomethines) absorbs at the 1690–1640 cm–1 area, according to the IR data reported in the literature. It is typically a strong, fairly crisp band that is close to the >C=C stretching frequencies and slightly below the bands of carbonyl groups. The stretching frequency of >C=N- is discovered to be 1670 cm-1 in the absence of strain, steric hindrance, or other complicated circumstances and to diluted solutions generated from neutral solvent.

An indication that chelation occurs in these complexes

through the azomethine group ^[31] and deprotonated hydroxyl group at position seven of the benzene moiety is shown by a change in the vC=N (azomethine group) band in the IR spectra of the complexes towards higher or lower wave number (C3–OH). This is supported by the development of new, faint bands in the IR spectra of all complexes between 620 and 690 cm-1, which are possibly attributed to Ln-N ^[32]. The stretching vibration of Ln-O is characterised by the emergence of brand-new bands with varying intensities in the spectra of isolated solid complexes. The ligands' IR spectra don't contain these bands.

There are three ways a nitrate ligand can coordinate to a metal ion: as a tridentate ligand, a hexadentate ligand, or as an uncoordinated ion. The IR spectra of L1 and 2: 1 (M: L) complexes of L2 revealed the presence of nitrate as a counter ion. A band can be seen in these compounds at 1384–1387 cm1 ^[33, 34].

Table 4: IR Data for complex compounds in Cm⁻¹

Sr. no	Compound code	HC=N	C=C	C-S-C	Ln-N	Ln-O
1	C1	1650	1444	1077	540	675
2	C2	1624	1467	1038	571	651
3	C3	1584	1460	1080	560	647
4	C4	1640	1455	1079	529	669
5	D1	1624	1457	1080	551	692
6	D2	1625	1468	1037	570	651
7	D3	1632	1470	1066	567	629
8	D4	1631	1480	1086	-	650
9	E1	1632	1463	1069	557	657
10	E2	1635	1480	1071	511	647
11	E3	1633	1532	1069	499	667
12	E4	1642	1480	1071	510	670
13	F1	1639	1440	1089	530	658
14	F2	1633	1483	1072	520	648
15	F3	1587	1434	1069	512	673
16	F4	1587	1437	1080	519	670

Ultra-violet and fluorescence spectroscopy

The lanthanide (III) structures' UV electronic spectra reveal that the development is moving towards sequential frequencies, which confirm the coordination of the ligand to the metal ion [35-37]. The broadness of the band in the electronic otherworldly information of the two compounds is probably caused by the n-* advancement of the azomethine nitrogen in the thiazole structures. The organisation of some agent lanthanide (III), or 4f metal buildings, seen in lanthanide structures, is described using typical unearthly information Every other metric that displays favourable characteristics and suggests a covalent nature in the metal ligand band, particularly the estimation of B 12, suggests a similar link with the 4f orbital metal ligand band. When compared to those of the metal, the lanthanide contraction ^[38] moves the band location towards the lower wave number, causing the covalence to decrease from Pr (III) to Sm (III) edifices. There is no essential retention of lanthanum (III) at the obvious location. The band saw caused by the Ce (III), Nd (III), and Sm (III) structures might be attributed to the progression of the distinct 4f configuration from ground state to the electrified J-level ^[39].



Fig 4: Fluorescence spectra of compound-C4

Biological Activity: The ligand and its metal complexes were tested for their ability to combat several diseases, including bacteria and fungi. Against the microscopic organisms E. coli, B. subtilis, and growth A. niger, C. albicans, all the metal complexes were viewed as dynamic. All of the integrated

Fig 5: Fluorescence spectra of compound-E1

structures displayed notable performance, however the combined metal complexes showed improved antibacterial activity compared to the ligands from which they are incorporated.

 Table 5: Antibacterial activity of lanthanide complexes medium: Muller Hinton method: agar well method dose of compound: 1mg/ml cup size:

 10 mm

Sr. No.	Compound	E. Coli	B. Subtilis
1	A-B1-Ligand	1.6	1.5
2	A-B2-Ligand	1	1
3	A-B3-Ligand	1.8	1.5
4	A-B4-Ligand	1.6	1.8
5	C1-Complex	1.7	1.8
6	C2-Complex	1.8	2.1
7	C3-Complex	1.3	1.1
8	C4-Complex	2.1	1.2
9	D1-Complex	0.9	-
10	D2-Complex	0.9	0.9
11	D3-Complex	1.3	1.3
12	D4-Complex	-	0.8
13	E1-Complex	1.9	2.1
14	E2-Complex	1.4	1.8
15	E3-Complex	2.1	2.0
16	E4-Complex	1.6	1.9

17	F1-Complex	1.8	1.2
18	F2-Complex	1.7	1.4
19	F3-Complex	2.0	1.4
20	F4-Complex	1.9	1.9
21	Amphicillin	0.9	1.5
Medium - Muller	Hinton	Cup Size - 1	0 mm

Dose of compound - 1mg/ml Method - Agar well method

Zone of Inhibition - mm

 Table 6: Antifungal activity of Lanthanide complexes Medium: Potato Dextrose Agar Method: Agar well method Dose of Compound: 1mg/ml

 Cup Size: 10 mm

Sr. No.	Compound	Aspergillus niger	C. Albicans
1	A-B1-Ligand	1.2	1.3
2	A-B2-Ligand	0.9	1.9
3	A-B3-Ligand	1.4	0.8
4	A-B4-Ligand	1.5	1.1
5	C1-Complex	1.4	1.7
6	C2-Complex	1.5	1.6
7	C3-Complex	1.2	1.1
8	C4-Complex	1.2	1.3
9	D1-Complex	1.4	0.9
10	D2-Complex	1.2	1.3
11	D3-Complex	1.5	1.6
12	D4-Complex	1.8	1.2
13	E1-Complex	1.9	1.2
14	E2-Complex	0.5	1.5
15	E3-Complex	1.6	1.4
16	E4-Complex	1.4	1.1
17	F1-Complex	1.1	1.5
18	F2-Complex	1.4	1.7
19	F3-Complex	1.2	1.3
20	F4-Complex	1.7	1.5
21	Fluconyzol	0.7	-

Medium – Pottato Dextrose Agar

Dose of compound - 1mg/ml

Method - Agar well method

Cup Size - 10 mm Zone of Inhibition – mm

Table 7: Name and abbreviations of the metal complexes

Sr. No	Name of the Metal Complexes	Abbreviations of Metal Complexes
1	N-Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Lanthanum (III) Nitrate Complex	C1
2	5-chloro -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Lanthanum (III) Nitrate Complex	C2
3	5-methyl -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Lanthanum (III) Nitrate Complex	C3
4	5-methoxy -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Lanthanum (III) Nitrate Complex	C4
5	N-Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Cerium (III) Nitrate Complex	D1
6	5-chloro -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Cerium (III) Nitrate Complex	D2
7	5-methyl -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Cerium (III) Nitrate Complex	D3
8	5-methoxy -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Cerium (III) Nitrate Complex	D4
9	N-Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Neodymium (III) Nitrate Complex	E1
10	5-chloro -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Neodymium (III) Nitrate Complex	E2
11	5-methyl -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Neodymium (III) Nitrate Complex	E3
12	5-methoxy -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Neodymium (III) Nitrate Complex	E4
13	N-Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Samarium (III) Nitrate Complex	F1
14	5-chloro -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Samarium (III) Nitrate Complex	F2
15	5-methyl -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Samarium (III) Nitrate Complex	F3
16	5-methoxy -Salicylidene-4-(4-bromophenyl)-2-Amino thiazole Samarium (III) Nitrate Complex	F4

Conclusion

Environmentally friendly was described as the synthesis of Schiff bases utilising 2-amino-4-(4-bromophenyl) thiazole, Ortho hydroxy aldehyde, and 3 different substituted Ortho hydroxy aldehydes. The complex was created using the traditional condensation reaction method. The reactions of lanthanide nitrate salts [Nd+3, La+3, Ce+3, and Sm+3] with a Schiff base ligand resulted in a variety of metal complexes. Analytical tests, spectral measurements, thermal studies, and the characterization of the produced ligand with UV-Visible and infrared all served to corroborate the complexes' structural details. The outcomes demonstrated that these complexes have a consistency of 10, the elements are bound to the ligand via the two nitrogen atoms at C=N, and the metal: ligand bonding ratio is 1:2. We investigated the antibacterial and antifungal activity of numerous *in vitro* drugs against four pathogenic bacteria utilising agar disc-spreading. Aspergillus niger, E. coli, B. subtilis, and C. albicans.

Acknowledgment

The authors appreciate the assistance in spectrum characterisation provided by the director of IICT Hyderabad as well as the assistance in screening the compounds for biological activity provided by the director of the school of life sciences, S.R.T.M.U. Nanded. We also want to express our gratitude to the principal of Shivaji College in Udgir for providing the lab resources.

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