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NOTE

Synthesis and Biological Studies of Salicylhydrazone and their Complexes with Lanthanum

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The present study comprises the synthesis of La(III) complexes of salicylhydrazone. All the complexes were characterized by elemental analysis and FT-infrared spectral studies. The biological applications of these compexes are also reported.

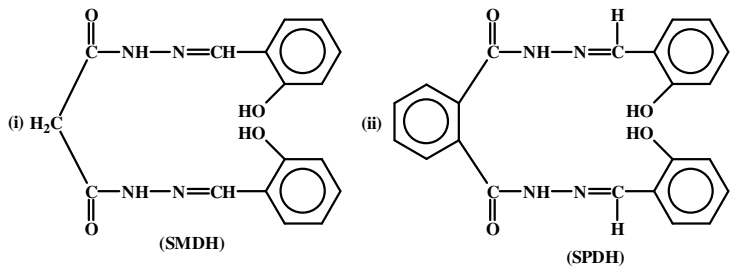
Key Words: Lanthanum(III) complexes, Salicylhydrazone.

The hydrazides and hydrazones metal complexes have an important role in the field of co-ordination chemistry. Various hydrazides and their derivative possess an antimicrobial activity against various micro-organism in addition they also possess an antifungal[1,2], insecticidal[3] and antiinflamatary[4] properties.

The present work deals with synthesis of some chemotherapeutically important salicylhydrazone complexes of lanthanum(III). The synthesized complexes are characterized and their biological activities have also been evaluated.

Synthesis of salicylaldehyde malonyl dihydrazone (SMDH): Malonyl dihydrazide (4.9 g, 2 mol) was dissolved in 25 mL methanol and 54 g of salicylaldehyde was added. The resultant mixture was refluxed for *ca.* 3 h on a water bath. A grayish white solid material was obtained which was filtered washed with ethanol and dried. The crude product was recrystallized from ethanol and dried over calcium chloride in vacuum dessicator under reduced pressure.

Synthesis of salicylaldehyde phthanoyl dehydrazone (SPDH): Phthalic dihydrazide (1.94 g, 2 mol) was dissolved in 20 mL ethanol and salicylaldehyde (1.06 g, 3 mol) was added. The resultant mixture was refluxed for *ca.* 4 h on a water bath. The yellow solid obtained which is filtered washed with ethanol and dried water reduced pressure.



Structure of ligand

Synthesis of lanthanum(III) complexes

Synthesis of salicylaldehyde malonyl dihydrazone lanthanum(III) complex: Salicylaldehyde malonyl dihydrazone (1.02 g, 3 mol) was dissolved in 10 mL dimethyl formamide solution and lanthanum(III) nitrate (0.866 g, 2 mol) was dissolved in 20 mL methanol. Both these solutions were mixed and the reaction mixture was refluxed for *ca.* 12 h on a water bath. The above contents were cooled and kept overnight, on concentration the complex was separated out in the form of white crystals. The crystals were filtered, washed with water, alcohol and finally with ether and done dried *in vacuo*.

Synthesis of salicylaldehyde phthanoyl dihydrazone lanthanum(III) complex: Salicylaldehyde phthanoyl dihydrazone (1.208 g, 3 mol) was dissolved in 20 mL methanol and added to 20 mL ethanolic solution of lanthanum(III) nitrate (0.86 g, 2 mol). Both the solutions were mixed and the reaction mixture was refluxed on a water bath for *ca.* 18 h. A yellow crystalline product was obtained which was filtered, washed with DMF, ethanol and ether. The crystals were finally dried *in vacuo*.

The physico-chemical properties of the synthesized ligands and La(III) complexes are given in Table-1.

TABLE-1
PHYSICO-CHEMICAL PROPERTIES OF LIGANDS AND ITS METAL COMPLEXES

	m.w./m.p. (ºC)	Elemental analysis (%): Calcd. (Found)		
		C	H	N
SMDH ($C_{17}H_{14}O_4N_4$)	340/289	58.97 (60.00)	4.49 (4.71)	16.04 (16.47)
SPDH ($C_{20}H_{18}O_4N_4$)	402/302	58.54 (59.54)	4.68 (4.47)	13.00 (13.73)
$La_2(C_{17}H_{14}O_4N_4)_3$	1292/338[d]	46.20 (47.36)	3.42 (3.25)	12.84 (13.02)
$La_2(C_{20}H_{18}O_4N_4)_3$	1478/368[d]	48.87 (48.38)	3.10 (3.26)	10.90 (11.36)

[d]Decomposition temperature.

The band between 3520-3060 cm^{-1} and a strong band near 1630[5,6] indicates the formation of hydrazone due to candensation of acid hydrazide with aldehyde and ketone. The N-H deformation occurs[7,8] between 1300-1260 cm^{-1} and the bands of amide group in the region of 1630-1600 cm^{-1} in due to C=N stretching vibration in the ligands[9,10] in the hydrazones on chelation with metal the normal frequency of amide I bond in shifted to lower frequency region by 30-10 cm^{-1} while in the amide II the frequency increases[11,12] by 10-15 cm^{-1}. The co-ordination of amide oxygen to the metal ion is also supported by the above changes in the amide group vibration.

The lowering in the frequency by 30-10 cm^{-1} or more in C=N stretching vibration (1630-1610 cm^{-1}) with a decreases in intensity of bond also suggested that nitrogen of azomethene group is co-ordinated to the metal ion[13,14]. Some new bands in the far IR region (620-510 cm^{-1}) and (470-440 cm^{-1}) indicates the formation of some new (La-O) (La-N) and other bonds in the metal chelates.

For biocidal studies the minimum concentration of the compound preventing the detectable growth is taken as a measure of biocidal activity. It is given in Table-2.

TABLE-2
BIOLOGICAL ACTIVITIES OF SYNTHESIZED LIGAND AND ITS COMPLEXES

Name of ligand/ complex	Zone of inhibition (MIC in μg/mL)			
	S. aureus	*E. coli*	*A. niger*	*C. Albicans*
SMDH	100	100	100	100
SPDH	100	100	100	75
$La_2(C_{17}H_{14}O_4N_4)_3$	50	12.5	25	25
$La_2(C_{20}H_{18}O_4N_4)_3$	25	12.5	25	50

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