

Structural Elucidation of the Complexes of Fe(III) with Ligand N-(5-Hydroxynaphthyl)-2-Mercaptopropanamide (HMP-H) and N-(5-Chloronaphthyl)-2-Mercaptopropanamide (CMP-H)

Parashuram Ram

Assistant Professor, Department of Chemistry, B.S.College, Danapur, Patna 800012

Email id- dr.p.prasad.chem@gmail.com

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Abstract : Fe(III) have been combined to the N-(5-hydroxynaphthyl)-2-mercaptopropanamide and N-(5-chloronaphthyl)-2-mercaptopropanamide having general chemical formula of complexes $[ML_3].nH_2O$ where $n=0,1,2,3...$ and L is the given ligand. These compounds have been characterized on the basis of usual method such as elemental analysis, measurement of magnetic moment, I.R, UV studies. On characterization, all the complexes have been found to octahedral geometry. All the complexes show a good antibacterial activity.

(Keywords: N-(5-chloronaphthyl)-2-mercaptopropanamide, N-(5-chloronaphthyl)-2-mercaptopropanamide, Trivalent Iron complexes, bacterial activity)

Introduction

Iron is the most abundant element found in the surrounding environment and play crucial role for survival of terrestrial organism. Iron also participates in biochemical process like ribonucleic reduction, oxygen transport, energy production, photosynthesis, and nitrogen reduction (1). The iron complexes of ligand having O and S as a potential donor center have received considerable interest. Several Fe (III) complexes have been known as contrast for magnetic resonance imaging (MRI) (2). Some Fe (III) complexes have been known for catalytic, antitumor and antimicrobial activity (3-7). The formation of structure of complexes thioglycolic acid, o-thiosalicylic acid related ligands have been investigated (8-18). The Iron (III) complexes of the ligands reported have not been communicated earlier.

Experimental

Material : All the chemicals used were of analytical grade. 1-amino-5-hydroxynaphthalene, 1-amino-5-chloronaphthalene, 2-mercaptopropanoic acid, $FeCl_3$, ethanol, KOH, were purchased from the E Merck (India) Limited, Mumbai. Ciprofloxacin hydrochloride was purchased from SRL, India.

Preparation

(a) Preparation of N-(5-hydroxynaphthyl)-2-mercaptopropanamide. This ligand was prepared in a manner analogous to the preparation of the thioglycolanilide. For this equimolar proportion of 1-amino-5-hydroxynaphthalene and 2-mercaptopropanoic acid were mixed in 500ml conical flask and heated in a glycerene bath at $110^\circ C - 120^\circ C$ for about 2-3 hours in slow current of CO_2 . When liquid was poured into a beaker of water, a solid mass is obtained. It is filtered, dried and crushed. It was purified by repeated recrystallization from dilute alcohol. Finally ligand obtained is dried in a vacuum desiccator. The ligand obtained shows melting point $143^\circ C - 144^\circ C$.

(b) Preparation of N-(5-chloronaphthyl)-2-mercaptopropanamide. It was prepared as ligand N-(5-hydroxynaphthyl)-2-mercaptopropanamide except that 1-amino-5-chloronaphthalene was used in place of 1-amino-5-hydroxynaphthalene. The ligand obtained has melting point $133^\circ C - 135^\circ C$.

(C) Preparation of Fe(III) complexes

Each the Fe(III) complexes prepared by adding an aqueous ethanolic solution of ligand (0.035 mole in 40ml) and an aqueous ethanolic solution of $FeCl_3$ (0.30mole in 15ml) the content

was digested well on water bath the orange yellow precipitate of Fe(III) complexes . It was filtered, washed with ethanolic water and dried in desiccator over KOH.

Synthesis Scheme

A general preparative method for Fe(III) complexes

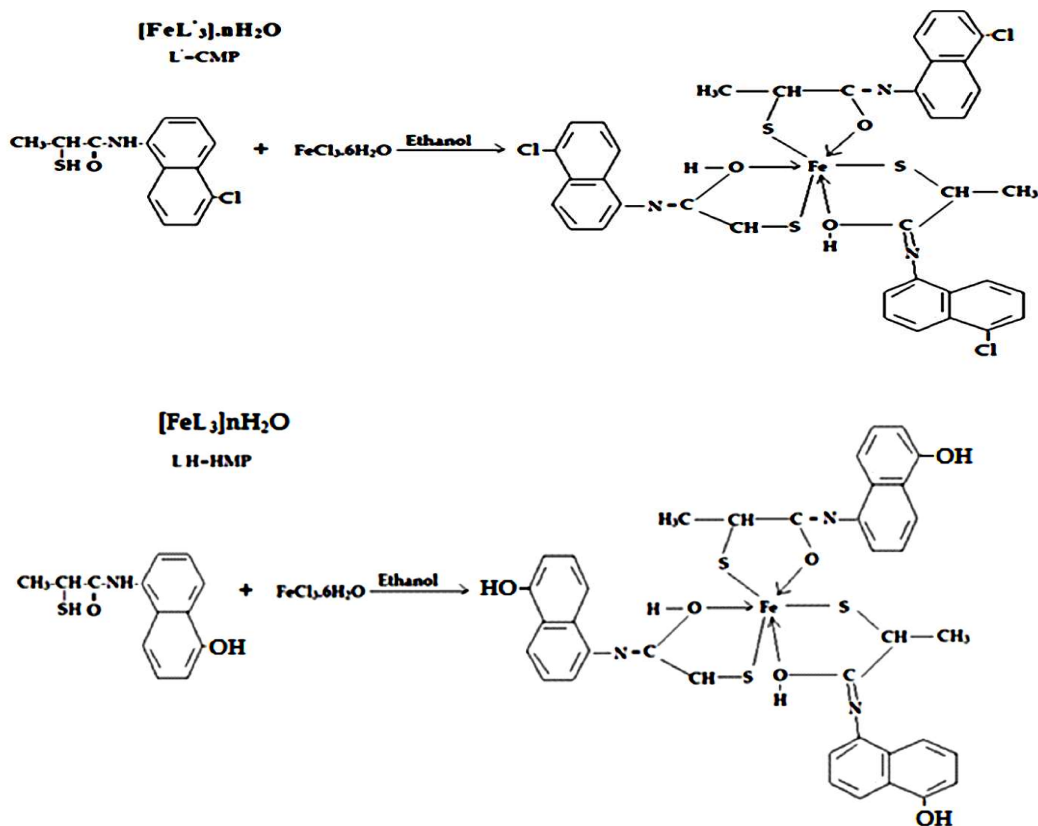


Table -1 Analytical

S.No.	Complexes	%M Cal (Found)	% C Cal (Found)	% H Cal (Found)	% N Cal (Found)	% S Cal (Found)	% Cl Cal (Found)
01	$[\text{Fe}(\text{CMP})_3] \cdot 2\text{H}_2\text{O}$	6.30 (6.31)	52.86 (52.84)	4.17 (4.15)	4.74 (4.72)	10.84 (10.85)	12.02 (12.05)
02	$[\text{Fe}(\text{HMP})_3] \cdot 2\text{H}_2\text{O}$	6.73 (6.75)	56.39 (56.38)	4.82 (4.81)	5.06 (5.07)	11.75 (11.58)	- (-)

Result and Discussion

The above complexes of Fe(III) are soluble in pure organic solvent especially in DMF. The μ_{eff} value of Fe(III) complexes have been observed in the range 4.25-4.40 B.M. which are two high for the half quenched spin 3/2 situation but intermediate between these corresponding to high and low spin complexes and may on the basis of analytical finding be suggested octahedral geometry exist in spin free ($s=5/2$) and spin paired ($s=1/2$) equilibrium. The compounds may be assumed to be monomeric as the paramagnetic susceptibility remains unaltered even in solution.

Fe(III) has ground state term 6S . In high spin octahedrally coordinated Fe(III) complexes,

the lowest configuration $(t_{2g})^3(e_g)^2$ gives rise to the ground state A_{1g}^6 . As there is no. sextet level present and absorption are spin forbidden are of very low extension co-efficient (19-29).

Biological activity - The complexes of Fe(III) with the CMP-H and HMP-H ligands show higher biological activity than free ligands and metal salt.

They exhibit higher activity against *S.aureus*, *B.Subfiles*, *B.cerus*, *E.Coil*, and *S.Marcscens*. Generally Fe(III) complexes exhibit marked augment in biological activity may be due to the effect of the metal ion on normal cell process (30-31).

Table -2 Important IR bonds of LH and L'H complexes

Compounds	>N-H Cm ⁻¹	>C=O Cm ⁻¹	-S-H Cm ⁻¹	Fe-N Cm ⁻¹	Fe-S Cm ⁻¹
LH	3260s	1655s	2565w	-	-
L'H	3270s	1665s	2575w	-	-
[Fe(CMP) ₃].2H ₂ O	3255s	1650s	-	315m	285m
[Fe(HMP) ₃].2H ₂ O	3240s	1660s	-	310m	270m

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