

Synthesis and characterization of chromium (III) complexes with N-(5-chloronaphthyl)-2- mercaptopropanamide (CMP-H) and N-(5-hydroxynaphthyl)-2-mercaptopropanamide (HMP-H) ligands.

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Abstract: Cr(III) have been combined with CMP-H and HMP-H ligands having formula of complexes $[\text{CrL}_3] \cdot n\text{H}_2\text{O}$ and $[\text{CrL}'_3] \cdot n\text{H}_2\text{O}$. These complexes have been characterized on the basis of usual methods such as elementary analysis, measurement of magnetic moments, IR, UV studies. On characterization all the complexes have been found to octahedral geometry and paramagnetic in nature. These provide a new supplement containing Cr (III) for patients with type-2 diabetes mellitus.

(Keywords : Cr (III), N-(5-Chloronaphthyl)-2-mercaptopropanamide, N-(5-hydroxynaphthyl)-2-mercaptopropanamide)

Introduction

Cr (III) is a trace mineral which is needed at supplement in a management of diabetes mellitus function. The diabetes relevant interaction of Cr(III) is with the hormone insulin and its receptors¹⁻². Chromium increases insulin binding to cell, insulin receptor number and activate insulin receptors³. The disadvantage of Cr (III) compound is the effect in DNA damage. The search for compounds with novel properties to deal with disease condition is still in progress chromium as Complex act as Cr (III) supplement. There is a direct relationship between the charge of these Cr(III) species and their reactivity with DNA⁴⁻⁵. Cr(III) in the form of oxides, Hydroxide or sulphates is much less mobile and exist mostly bound to organic matter in soil and aquatic environment. Free Cr(III) metal ions would quickly become a adsorbed or hydrolysed and

precipitated in the absence of soluble complex ligands. The presence of organic ligands kept Cr(III) in solutions and prevented removal of Cr at pH >5.5⁶. There are many published methods for chromium speciation such as atomic spectrometry, plasma mass spectrometry and visible spectrometry, Ion chromatography, reversed phase liquid chromatography. The most widely employed procedures for chromium speciation by atomic mass spectrometry are based on selective Proconcentration of Cr(III).⁷⁻¹³ In this paper we have initiated through the study of Cr(III) Complexes with CMP-H and HMP-H ligands having 'O', 'S' donor atoms¹³. Cr (III) complexes of the ligands reported have not been communicated earlier.

Experimental

A) Materials - All the chemicals used were of analytical grade 1-amino-5-hydroxynaphthalene, 1-amino-5-chloronaphthalene, 2-mercapto- propanoic acid, $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$, KOH, ethanol were purchased from the E.Merck (India) Limited Mumbai.

Preparation of ligands

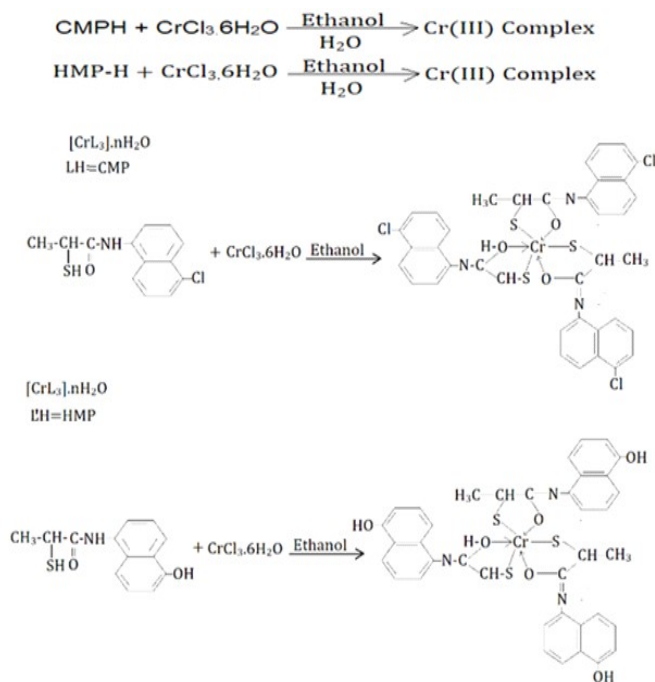
a) Preparation of N-(5-hydroxynaphthyl)-2-mercaptopropanamide- This ligand was prepared in a manner analogous to the preparation of the thioglycolanilide. For this equimolar proportion of 1-amino-5-hydroxynaphthalene and 2-mercapto- propanoic acid were mixed in 500 ml

conical flask and heated in a glycerene bath at 110°C-120°C for about 2-3 hours in slow current of CO₂. 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 the ligand obtained is dried in a Vacuum desiccator. The ligand obtained shows melting point 143°C-144°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 1-amino-5-hydroxynaphthalene. The ligand obtained has melting point 133°C-135°C.
- c) Preparation of Cr(III) complexes -The Cr(III) Complexes with CMP-H and HMP-H Ligands were prepared. Each

complexes was prepared when chromium (III) chloride (CrCl₃.6H₂O) solution (0.26gm.mol) in aqueous ethanol was mixed with ligand solution in the same solvent in the ratio of 1:1. The resulting solution was treated a few drops of ammonium hydroxide till the solution is alkaline. The resulting mixture was stirred under reflux for 1h and 80 oC. On Standing the pale yellow precipitate was collected by Buchner filtration, Wash with water and dried over anhydrous CaCl₂. This method was based on the experiment of Yang¹⁴ with same modification in pH adjustment and the sequence of the process. The yielding solid was weighing until the constant weight.

Synthesis Scheme



Analytical Table

S.No	Complexes	%Cr cal. (found)	%C cal. (found)	%H cal. (found)	%N cal. (found)	%S cal. (found)	%Cl cal. (found)
1	[Cr(CMP) ₃] ₂ H ₂ O	5.90 5.92	53.09 53.08	4.19 4.18	4.76 4.74	10.89 10.85	12.80 12.10
2	[Cr(HMP) ₃] ₂ H ₂ O	6.29 6.25	56.66 56.65	4.84 4.87	5.08 5.06	11.62 11.66	—

Results and Discussion

The Resulting complexes of Cr(III) in this work are soluble in pure organic solvent especially in DMF. First of all Longavin¹⁵ reported a theory to explain diamagnetic substances.

Nyholm et al¹⁶ has attempted to predict the geometry of transition metal complexes on the basis of magnetic moment value^{17,18}. Magnetism has been of considerable utility in establishing the structure of many complexes. Cr(III) complexes [CrCMP₃] and [CrHMP₃] have μ_{eff} value 3.86 and 3.55 BM respectively which corresponds to spin only value.

On the basis of magnetic moment value the resulting complexes are paramagnetic in nature and have octahedral geometry.

Spectral behaviour:

The electronic spectra of Cr(III) complexes have been studied. They are of little use for structure determination. It is formed that ethanolic or DMF solution of complexes do not exhibit absorption and b/w 450-1000 nm. At lower wavelength they display either charge transfer or ligand absorption i.e $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ transition in almost one or two shoulders b/w 400 and 600 nm.

IR spectral studies of the complexes

IR spectrum arises from the absorption by a molecules in the region between 4000cm⁻¹ to 200cm⁻¹. IR spectra were recorded in KBr Discs NUJOL and HCB Mulls using a Perkin-Elmer 1330 spectrophotometer.

Important IR bands of LH and L'H complexes

Compounds	>NH cm ⁻¹	>C=O cm ⁻¹	-SH cm ⁻¹	Cr-N cm ⁻¹	Cr-S cm ⁻¹
LH	3260	1655	2565	-	-
L'H	3270	1655	2575	-	-
[Cr(CMP) ₃] ₂ H ₂ O	3250	1650	-	300	275
[Cr(HMP) ₃] ₂ H ₂ O	3250	1660	-	310	270

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