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Studies on thermal stability of Metal Chelates of Th and Tb metals with p-chlorobenzaldehydethiosemicarbazone

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Abstract
Co-ordination compounds shows their importance in various and wide spectrum it is useful in agriculture[8] it is very important in petroleum industries ,in biochemistry, B_{12}, hemoglobin, chlorophyll and various complex catalyst are useful in biochemical reactions in animals and plants and in medicinal chemistry, drugs can be easily absorb in human body as well as bio-available efficiently if these are in complex form[16] and in analytical chemistry etc. and deserves highest attention.
Complex of p-chlorobenzaldehydethiosemicarbazone with heavy like Platinium, Paladium . Thorium, Tb metals also shows anti-bacterial as well as anti-carcinogenic[1-7] properties etc in order to understand its chemistry and mode of action its thermal stability and its structural aspects are very important. Pyrolysis (TGA) is an very important analytical approach and has been useful in contributing to ascertaining some of the structural aspects, like thermal stability, stoichiometry of compound including metal complexes.
In this present study it includes pyrolysis study of solution of complex Th(IV) and Tb(III) metals with p-chlorobenzaldehydethiosemicarbazone and the resulting data analysed .
Key Words*- Pyrolysis, medicinal , Co-ordination compounds,Thermal stability*

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I. Introduction

Co-ordination compounds ane useful in agriculture industries pestisides use aa a complex shows better results (17) . Coordination compounds not only having biological importance but also usful in medicines field , co-ordination compounds of heavy metals with thiosemicarbazone shows anti-cancer properties [1-7], many of the thiosemicarbazone complexes are anti- bactira. Thiosemicarbazone are very good ligands it shows significant biological volues and importance ,when this ligands bind with heavy metals ions then it shows properties like antioxidant ,anticancer etc.[11]. Due to such medicinal properties researchers and scientists showing interest in recent years to know the structural aspects of heavy metal complexes of thiosemicarbazones, which possessing the carcinostatic properties with the view to understand their action and mechnics . The studies of complex is now being greatly addressed due to its promising role in fighting cancer disease,

Besides exploration the metal-thiosemicarbazones complex in its solid state, their solution chemistry also deserve the high attention of the researchers[1-7].Observation on the increasing activity of some drugs when administered as metal complexes by William Frunst et al.[9] and inhibition of tumor growth with metal complex of thiosemicarbazones by Dwyer et al.[10] have further increased the interest on coordination compounds..

Synthesis of P-chlorobenzaldehydethiosemicarbazone

Synthesis of thiosemicarbazide is carried out by the reaction of carboxylic acid hydrazide and isothiocyanate in the presence of polar solvent [12] .Equ**i**molar aquous solution of thiosemicarbazide is reacted with equimolar solution of p-chlorobenzaldehyde (acetone base) at room temperature with constant stiring. When the reaction completed, a dirty white ppt obtained which then filtered, washed and kept in oven overnight at 35^{o}-40^{o}C. for drying

Instruments:

Elemental Analyser of Model no. 4208, for pyrolysis study of the complexes, manually operated assembly which is equipped with Toshmiwal furnace, duly standardized with calcium oxalate are employed.

II. Experiment

All the chemical which used were of analytical grade, For isolation of metal complexes of Th, Tb, three moles of p-chlorobenzaldehydethiosemicarzone(in acetone) were mixed with a mole of metal ion, the deepening of colour resulted on mixing. On mild shaking followed by cooling the precipitate appeared in both the case . The precipitate was filtered, washed with ice cooled water and dried at 35°-40°C. the elemental analysis agreed to the accompanying composition;

$ThR_4 . H_2O$ and $Tb.R_3 1½ H_2O$ where R = p- $Cl.C_6H_4.CH=N-N=CS-NH_2$.

III. Result and Discussion:

tris-(p-chlorobenzaldehydethiosemicarzone) Th(IV). H_2O

tris-(p-chlorobenzaldehydethiosemicarzone) Tb(III). 1½ H_2O

Like metal complexes other than Eu(III) complexes, Tb(III) – p-chlorobenzaldehydethiosemicarbazone is hydrated one having the composition. Tb. R_3 . $1½H_2O$ and thermal stability upto 100^0C. 1½ H_2O molecules were lost from 100^0 to 120^0C and the dehydrated complex Tb. R_3 could be constant by weight in the temperature range : $120^0 – 156^0$C. Further a molecular of R_3 spilt off from 150^0 to 272^0C forming an intermediate of definite composition : Tb. R_2 (272^0C – 304^0C), which after 304^0C lost two R molecules upto 560^0C
levelling off hence forth; the residue off hence forth; the residue left was nothing but Tb_2O_3. Possible of decomposition may be given by following steps .{Table 1)

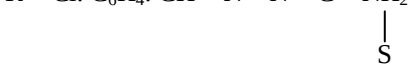
$$Tb.R_3.\ 1½H_2O \xrightarrow{1½H_2O} Tb.R_3 \xrightarrow{-R} Tb.R_2 \xrightarrow{-2R} Tb_2O_3$$

Table : 1

PYROLYSIS DATA ON PROGRESS OF DECOMPOSITION OF tris-(p-chlorobenzaldehydetiosemicarbazonato) Tb (III) $1½H_2O$

Stability/Phase (Temp. Range)°C	Loss (Temp. Range) °C	% Loss		% Tb_2O_3	
		Calc.	Found	Calc.	Found
Tb. R_3 $1H_2O$ (upto 100)	1 ½H_2O (100-120)	03.13	03.09		
Tb. R_3 (120-156)	R (156-272)	26.16	25.77		
Tb. R_2 (272-304)	2R (304-560)	78.47	79.90	21.12	19.60
Tb2O3 (560 onwards)	--	--	--	21.74	23.21

% losses are accumulated



The metal chelate of ligand with 5f block element [(Th(IV)] displayed five plateaus on its thermogram coinciding with the accompanying definite composition.upto temperature 112°C it loses one molecule of H_2O then the weight is constant upto 126°C-152°C, at this stage $Th.R_4$ decompose , %of weight loss suggest formation of $Th.R_2$ this composition is remains stable upto temperature 392°C further decomposition gives Th 3/4R its stabilty range is given below and finally above 600oc ThO_2 is residue and thermally stable compound

I.	$Th.R_4.H_2O$	(upto 112^0C)
II.	$Th.R_4$	($128^0 – 152^0$C)
III.	$Th.R_2$	($360^0 – 392^0$C)
IV.	Th. 3/4R	($480^0 – 512^0$C)
V.		(600 onwards)

The successive losses as shown below:

I ----- II	H_2O	($112^0 – 128^0$C)
II ---- III	2R	($152^0 – 360^0$C)
III ---- IV	1¼R	($392^0 – 480^0$C)

IV ---- V 3/4R ($512^0 – 600^0$C)
were observed

$$\text{Th. } R_4.H_2O \xrightarrow{-H_2O} \text{Th. } R_4 \xrightarrow{-2R} \text{Th. } R_2 \xrightarrow{-1¼R} \text{Th. } 3/4R \xrightarrow{-3/4R} ThO_2$$

Tables : 2 contain the analytical data on the course of decomposition of the metal complexes.
Piecing together the solution data and thermal data, the final following structures may be suggested.

Table : 2
PYROLYSIS DATA ON PROGRESS OF DECOMPOSITION OF Tetra-(p-chlorobenzaldehydetiosemicarbazonato) Th(IV) Monohydrate

Stability/Phase (Temp. Range)^{0}C	Loss (Temp. Range) ^{0}C	% Loss		% ThO_2	
		Calc.	Found	Calc.	Found
Th. $R_4.H_2O$ (upto 92)	H_2O (112-128)	01.56	01.89		
Th. R_4 (128-152)	2R (152-360)	39.16	37.74		
Th. R_2 (360-392)	1¼R (392-480)	63.69	63.20		
Th. 3/4R (480-512)	3/4R (512-60)	78.33	75.00		
ThO_2 (600 onwards)	--	--	--	22.96	25.00

% losses are accumulated

R = Cl. C_6H_4. CH = N – N = C – NH_2
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