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PYROLYSIS STUDIES OF METALS CHELATES OF Gd and Eu METALS WITH P-CHLOROBENZALDEHYDETHIOSEMICARBAZONE

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ABSTRACT

Co-ordination compounds gaining attention due to its use in wide spectrum it is useful in petroleum chemistry, bio-chemistry, medicinal chemistry,as well as in analytical chemistry etc. and deserves high attention. Complex of p-chlorobenzaldehydethiosemicarzone with heavy metals also shows anti-bacterial as well as anti-carcinogenic.[1-7] properties etc in order to understand its chemistry its structural aspects are very important. TGA is an very important analytical tool and has been useful in contributing greatly to ascertaining some of the structural aspects, like thermal stability, stoichiometry of compound including metal complexes. Present study includes pyrolysis study of solution of complex Gd(III) and Eu(III) metals with p-chlorobenzaldehydethiosemicarzone and the resulting data were analysed.

KEYWORDS: Pyrolysis, TGA, Co-ordination compounds, Thermal stability.

INTRODUCTION

Co-ordination compounds ane not only having biological importance but also verry usful in medicines, co-ordination compounds of heavy metals with thiosemicarbazone shws anti-cancer properties,[1-7] many of the thiosemicarbazone complexes are anti- bactira. Thiosemicarbazone are very good Sciffs based ligands it shows significant biological volues and importance when this ligands bind with heavy metals ions then it shows properties like antioxidant anti-cancer etc.[14] 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 diseases

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,[8] 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.[15] Equimolar 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°-40°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.

Experiment

All the chemical which used were of analytical grade, For isolation of metal complexes of Gd, Eu, 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; $Gd.R_3.2H_2O$ and $Eu.R_3$ where R = p- $Cl.C_6H_4.CH=N-N=CS-NH_2$.

RESULT AND DISCUSSION

tris-(p-chlorobenzaldehydethiosemicarzone) Gd(III). $2H_2O$
tris-(p-chlorobenzaldehydethiosemicarzone) Eu(III).

Thermally Gd. $R_3.2H_2O$ was stable upto 72^0C. With the loss of two water molecules from the parent compound, a plateau referring to the formation of anhydrous metal chelate of Gd. R_3 composition was observed in the temperature range: 100^0 – 120^0C. The cleaving of 2 R organic matter started at 120^0C and continued till the temperature became 328^0C, creating another intermediate (Gd. 7/8R) which, displaying its constant weight in the temperature range: 328^0-360^0C, allowed further departure of 7/8R molecules;440°C the residue left conformed to Gd_2O_3.

$$Gd.R_3.2H_2O \xrightarrow{-2H_2O} Gd.\ R_3 \xrightarrow{-2\frac{1}{8}R} Gd.\ 7/8\ R \xrightarrow{-\frac{7}{8}R} Gd_2O_3$$

Table 1: Pyrolysis Data on Progress of Decomposition Of tris-(p-chlorobenzaldehydetiosemicarbazonato) Gd(III) Dihydrate.

Stability/Phase (Temp. Range)°C	Loss (Temp. Range) ^{0}C	% Loss		% Gd_2O_3	
		Calc.	Found	Calc.	Found
Gd. R_3. $2H_2O$(upto 72^0C)	$2H_2O$(72-100)	04.16	04.12		
Gd. R_3 (100-120)	2 1/8 R(120-328)	66.97	67.06		
Gd. 7/8R (328-360)	7/8R (360-400)	78.30	74.50		
Gd_2O_3 (440 onwards)	--	--	--	20.09	24.90

% losses are accumulated

$$R = Cl.\ C_6H_4.\ CH = N - N = \underset{\displaystyle S}{\underset{|}{C}} - NH_2$$

Tris (P-chlorobenzaldehydethiosemicarbazonato) Eu(III) is an anhydrous metal chelate which could keep up its identity thermally upto 92^0C, evidenced from first levelled off portion of the pyrolysis curve. The successive plateaus corresponded to the constant weight of Eu 15/16R and Eu_2O_3 in the respective temperature ranges: 272^0 – 312^0C and 504^0C onwards. The losses observed in reaching the second plateau was 2R molecules (92^0 – 272^0C) while the rest of the organic matter (15/16R) departed between the second and third plateau.504°C onward The residue left agree to Eu_2O_3.

$$Eu.\ R3. \xrightarrow{2\frac{1}{16}R} Eu.\ \frac{15}{16}\ R \xrightarrow{-1\frac{15}{16}R} Eu_2O_3$$

Table 2: Pyrolysis Data on Progress of Decomposition of tris-(p-chlorobenzaldehydetiosemicarbazonato) Eu(III).

Stability/Phase (Temp. Range)°C	Loss (Temp. Range) ^{0}C	% Loss		% Eu_2O_3	
		Calc.	Found	Calc.	Found
Eu. R_3 (upto 92)	2 1/16R (92-272)	56.20	56.87		
Eu. 15/16R (272-312)	15/16R (312-504)	78.88	78.92		
Eu_2O_3 (504 onwards)	--	--	--	21.12	19.60

% losses are accumulated

$$R = Cl.\ C_6H_4.\ CH = N - N = \underset{\displaystyle S}{\underset{|}{C}} - NH_2$$

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