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Synthesis characterization and spectrophotometric study of Fe(III) complex with hydrazone

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ABSTRACT

The stability constant of Fe (III) complexes of metal with 8-aceto-7-hydroxy-N-[4',6'-dichloro-1',3',5'-S-triazino] coumarin hydrazone was determined by Job's method at Wavelength 440 and 570 nm by keeping the metal:ligand ratio 1:1. The structure elucidation have been done by elemental analysis, IR and Magnetic studies. © 2009 Trade Science Inc. - INDIA

KEYWORDS

Coumarin;
Hydrazone;
Triazine;
Metal complex;
Spectrophotometric study.

INTRODUCTION

Hydrazones are Interesting analytical reagents^[1]. Although hydrazones are used for the spectrophotometric determination for metal ions, heterocyclic hydrazones are not exploited much. Some authors have reported the antifungal activity of co(II) , Ni(II) and cu (II) metal complexes devived from hydrazones^[2].

Metal Complexes of coumarin derivatives are also reported as an antimicrobial agent^[3]. The S-triazine and it's derivatives have their own importance in hetero cyclic compounds due to their good activities such as anticancer^[4] antiviral^[5] and antimicrobial^[6]. Some hydroxyl Coumarin derivatives are reported as anti HIV agent^[7].

In the view of these observations we have synthesized metal complex derived from 5-triazine hydrazone Coumarin and studied their stability constant by Spectrophotometer.

EXPERIMENTAL

All chemicals used in the present work were of A.R. grade. Melting point of compound was determined on open capillary tubes and is uncorrected. Job's method of continuous variation has been applied for confirming metal-ligand ratio and stability constant. The magnetic measurements were made at room temperature by the Gouy balance method^[8]. Infrared spectra were measured in the range 4000-400cm⁻¹ on a schimadzu FT IR-801 spectrophotometer with KBr pellets.

Preparation of [8-Aceto-7-hydroxy-N-(4',6'-dichloro-1',3',5-s-triazino)] coumarin hydrazone

8-Aceto-7-Hydroxy- Coumarin hydrazone (2.18gm 0.01M) was dissolved in acetone and mixed with a solution of cyanuric chloride (1.84gm 0.01M) dissolved in acetone, in a round- bottomed flask fitted with a water condenser. The mixture was constantly

stirred for 3 hrs maintaining the pH of the reaction mixture between 6 to 7 and temperature at 5°C. The content was successively poured over crushed ice, filtered and crystallized from ethanol as dull pink palates. m.p.147°C, yield: 68%.

Preparation of metal complex

1% ethanol solution of 8-aceto-7-hydroxy-N-(4',6'-dichloro-1',3,5'-s-triazino)coumarin hydrazone was added dropwise to a warm Ferric chloride solution maintaining the pH of the mixture at pH 6.5 to 7 during the reaction. Light brownish yellow precipitates thus obtained were washed with warm ethanol. The precipitates are soluble in chloroform, carbon tetrachloride and benzene. The m.p. of Bis[8-aceto-7-hydroxy-N-(4',6'-dichloro-1',3',5'-s-triazone) coumarin hydrazone] Ferric(III) is 289°C.

Spectrophotometric study

The composition of Fe(III) chelate with the reagent 8-aceto-7-hydroxy-N-(4',6'-dichloro-1', 3',5'-s-triazino)coumarin hydrazone has been determined on the basis of Job's continuous variation method.

Composition of bis[8-aceto-7-hydroxy-N-(4',6'-dichloro-1',3',5-S-triazone)] coumarin hydrazone] Ferric(III) complex By Job's continuous variation method

A 0.002M solution of Fe(III) was prepared by suitable dilution of the standard solution.

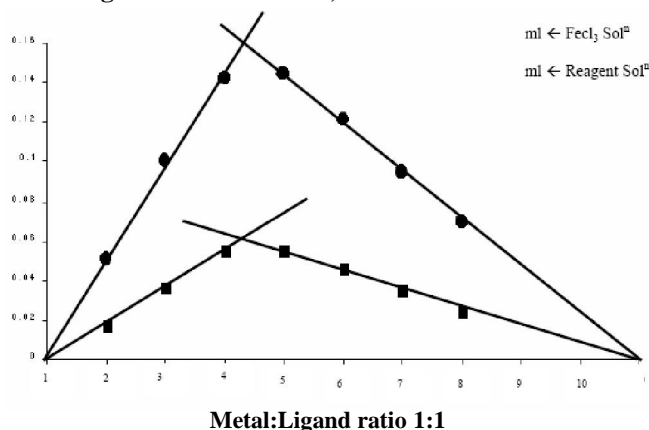
The solution of reagent 8-aceto-7-hydroxy-N-

Reagent: 8-Aceto-7-hydroxy-N-(4',6'-dichloro-1',3',5'-s-triazino) coumarin hydrazone

Reagent: 0.002M in C₂H₅OH

FeCl₃ 6H₂O 0.002M

Wavelength used λ=○ 440 nm; λ=□ 570 nm



(4',6'-dichloro-1',3',5'-S-triazone) coumarin hydrazone was prepared in absolute alcohol or DMF. The solution of metal salt and the reagent were mixed in varying proportions as under.

Metal ion solution. 1 2 3 4 5 6 7 8

Reagent solution. 9 8 7 6 5 4 3 2

pH of the solution was adjusted to 6.5.

The precipitated complex was extracted with three 5ml portion of chloroform and final volume of chloroform to 25 ml. The absorbance of chloroform extracts were measured at 440 and 570nm. The result are tabulated in TABLE 1.

TABLE 1: Job's method for bis[8-aceto-7-hydroxy-N-(4',6'-dichloro-1',3',5'-s-triazino)coumarin hydrazone]Ferric(III)

Nickel Solution Taken ml	Reagent Solution ml	Optical Density	
		λ=440 nm	λ=570 nm
1	9	0.09	0.004
2	8	0.019	0.085
3	7	0.026	0.011
4	6	0.036	0.016
5	5	0.044	0.021
6	4	0.033	0.013
7	3	0.021	0.007

It is evident from the graph that absorbance gradually increases up to molare composition of metal to the reagent and after that it becomes constant indicating 1:1 stoichiometry of the complex.

Evaluation of stability constant

$ML_n = M + nL$

$C(1-\alpha) = C [n.c \alpha]^n$

$K_s = c(1-\alpha)/c[n.c \alpha]^n$

Taking $n=2$ in this case the equation reduce to

$K_s = 1 - \alpha / 4 c^2 \alpha^2$ Where, $\alpha = (E_m - E_s) / E_m$

E_m = Maximum absorbance obtained from the horizontal portion of the curve, or at the intersect of extrapolated lines.

E_s = absorbance at the stoichiometry molar ratio of the metal to reagent in complex.

Calculation of stability constant

The stability constant is calculated from the above relation.

The standard free energy change ΔG° for the formation reaction of complex has been calculated at 25°C

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using the formula $\Delta G^\circ = -RT \ln K$

Bis [8-aceto-7-hydroxy-N-(4',6'-dichloro-1',3',5'-s-triazine) coumarin hydrazone] Fe(III)

Method Job's	λ_{nm}	Em	Es	α	Ks	ΔG°
Fe(III)	440	0045	0.044	0.022	9.78×10^8	-2.259k cal/mole
	570	0.022	0.021	0.045	3.18×10^8	-1.594k cal/mole

Metal solution: 0.002M

Ligand solution: 0.002M

Final volume of Chloroform extract: 25ml

Wavelength: 440nm, 570nm.

pH: 7.0

RESULT AND DISCUSSION

All the compounds were analyzed for carbon, hydrogen, nitrogen, oxygen, chlorine and metal and the result were within=0.4% of theoretical values.

TABLE 2 : Elemental data of compounds

Bis[8-aceto-7-hydroxy-N-(4',6'-dichloro-1',3',5'-s-triazino)coumarin hydrazone] Ferric (III)								
Mol. Formula	Mol. Wt.	% of Elements						μ_{eff} in B.M.
		M	C	H	(O)	N	Cl	
Fe[C ₁₄ H ₈ O ₃ N ₅ Cl ₂] ₂	720.85	Fe 13.30 (13.32)	39.86 (39.89)	1.89 (1.89)	11.37 (11.39)	16.61 (16.62)	16.88 (16.86)	6.86 (Paramagnetic)

CONCLUSION

The spectral study, Spectrophotometric study and magnetic study of these metal complex of hydrazone reveals that metal complex is having high spin tetrahedral geometry and the metal ligand ratio is 1:1. The ligand can be good analytical reagent for some metal ions.

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