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Synthesis of Hydrazine Carboxamide-2-[(2-Hydroxy-1-Naphthalenyl) Methylene] as an analytical reagent for the Extractive Spectrophotometric determination of Fe (II)

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ABSTRACT

The Hydrazine Carboxamide-2-[(2-Hydroxy-1-Naphthalenyl) Methylene] is used as a reagent for extraction and spectrophotometric determination of Fe (II). Iron forms gray colored complex which can be quantitatively extracted in n-Butanol as solvent at pH 7.4. Beer's law is obeyed in the range 1-10ppm giving a linear and reproducible graph under optimum conditions. The λmax is observed to be 500nm. The complex obtained is studied by Job's continuous variation method, mole ratio method and log-log method. The effect of pH as function of extraction, effect of solvents, effect of salting out agents, effect of reagent concentrations and interference study of foreign ions has been also studied. The molar absorptivity is found to be 0.1739×10^{4}L $mol^{-1}cm^{-1}$ and Sandell sensitivity is observed to be 0.2257μg/cm^{2}. The method developed is successfully applied to various commercial samples.

Keywords: HCHNM, Iron, Spectrophotometric determination.

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INTRODUCTION

The significance of iron as a transition metal lies in its wide spectrum of applications covering many frontier areas of study, particularly in industrial and consumer products. Excessive iron can be toxic because free ferrous iron reacts with peroxides to produce free radicals, which are highly reactive and can damage DNA, proteins, lipids and other cellular components. Hence, owing to the significance of iron, it's determination from associated elements by extractive spectrophotometry has been of considerable importance. A wide variety of reagent has been reported for the spectrophotometric determination of iron. A number of reagents such as hydrazone [1, 2], thiosemicarbazone [3], oxime [4-8], etc. have been used for the determination of Fe (II). However, these methods suffer from limitations such as interference of ions [9-15], requirement of masking agent [16, 17] etc. The synthesis of semicarbazone derivative and their applications towards metal ions have been reported method, far superior in sensitivity and selectivity to those reported in the literature, is developed for the extractive spectrophotometric determination of iron with Hydrazine Carboxamide-2-[(2-Hydroxy-1-Naphthalenyl) Methylene]. A close literature survey indicates that HCHNM has so far not been employed for either coordination or analytical studies. The proposed method is free from limitations.

EXPERIMENTAL

The absorption measurements were made on a Shimadzu UV visible 2100 Spectrophotometer with 1 cm quartz cells and standard buffer solutions and the digital pH meter Li- 120 model of Elico Pvt.Ltd. was used for pH measurement study. The chemicals used were of analytical reagent grade. Stock solution of iron was prepared by dissolving $(NH_4)_2$ Fe $(SO_4)_2.6H_2O$ in double distilled water and was standardized[7] by known method. The working solutions were prepared by appropriate dilution as required. The reagent was prepared as reported in the literature [18].

PROCEDURE FOR EXTRACTION

1.0 ml of aqueous solution containing 0.1mg of iron metal and 1 ml of reagent were mixed in a 50 ml beaker. The pH of the solution adjusted to 7.4 keeping the volume 10 ml. The solution was transferred to 100 ml separatory funnel. The beaker was washed twice with n-butanol and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was collected in 10 ml measuring flask and made up to the mark with organic solvent if required. After separation of the two phases, the pH of the aqueous phase was measured and the Fe (II) in each phase was determined by known method.

RESULTS AND DISCUSSION

The reagent forms gray colored complex with Fe (II), which was extracted in organic phase and the results obtained are as follows.

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EXTRACTION AS A FUNCTION OF PH

The extraction of iron with Hydrazine Carboxamide-2-[(2-Hydroxy-1-Naphthalenyl) Methylene] has been studied over the pH range1-10 and was observed that percentage extraction of Fe (II) is maximum at pH 7.4

ABSORPTION SPECTRUM

The absorption spectrum of Fe (II): Hydrazine Carboxamide-2-[(2-Hydroxy-1-Naphthalenyl) Methylene] in n-butanol shows the maximum absorption at 500 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 500 nm.

INFLUENCE OF DILUENTS

The suitability of diluents was investigated using organic solvents such as chloroform, ethyl acetate, ethyl methyl ketone, diethyl ether, toluene, n-butanol, carbon tetrachloride, MIBK, nitrobenzene, etc. The extraction of Fe (II) was quantitative with HCHNM in n-butanol. Hence, n-butanol was used for further extraction studies as it gave better and quicker phase separation.

EFFECT OF SALTING OUT AGENTS

The presence of 0.1M nitrate salts of alkali and alkaline metals does not show any effect over the absorbance value of Ni (II):Hydrazine Carboxamide-2-[(2-Hydroxy-1-Naphthalenyl) Methylene] complex extract.

EFFECT OF REAGENT CONCENTRATION

Various volumes of 0.1% reagent solution were added to the sample solution containing 100 μg of iron at respective pH values. The absorbance remained nearly constant when the volume of the reagent solution used was more than 1 ml. Therefore, 1 ml of 0.1% reagent was chosen for the quantitative determination of the metal.

EFFECT OF EQUILIBRATION TIME

The change in absorbance with variation in equilibrium time for extraction of Fe (II) shows that equilibrium time of 50 sec. are sufficient for quantitative extraction of Iron.

STABILITY OF THE COMPLEX WITH TIME

The study of stability of colour of the Fe (II): Hydrazine Carboxamide-2-[(2-Hydroxy-1-Naphthalenyl) Methylene] complex with respect to time shows that the absorbance due to

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extracted species is stable up to 72.0 hours, after which slight decrease in absorbance is observed. Throughout the experimental work, for practical convenience, the measurements have been carried out within one hour of extraction of Iron.

CALIBRATION PLOT

A calibration plot of absorbance against concentration of Fe (II) gives linear and reproducible graph in the concentration range 1 to 10 ppm of iron indicating that the Beer's law is obeyed in this range. (Fig 1). The molar absorptivity and sandell sensitivity were calculated to be is0.1739×10^{4} L mol^{-1} cm^{-1} and 0.2257µg/cm^{-2} respectively.

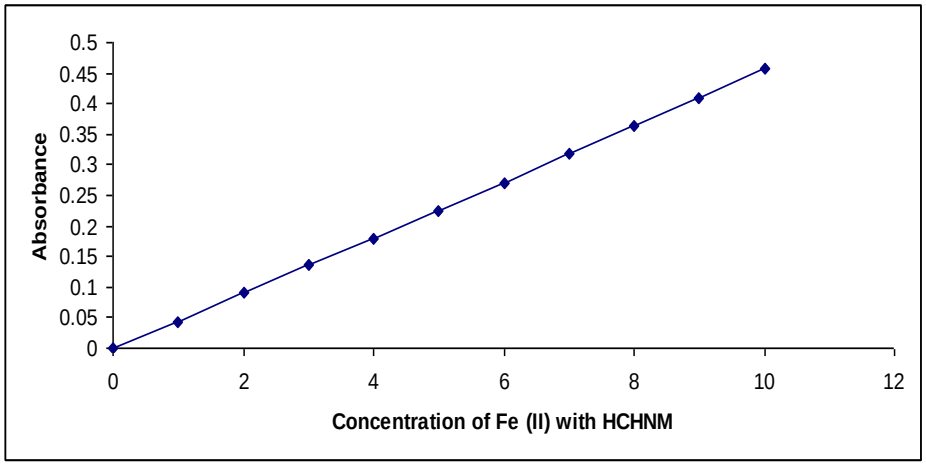

Fig 1: Calibration plot of Fe (II) with HCHNM

NATURE OF EXTRACTED SPECIES

The composition of extracted species has been determined by Job's continuous variation method (Fig 2), Slope ratio method (Fig 3), and Mole ratio method. It shows that the composition of Fe (II): HCHNM complex is 1:2.

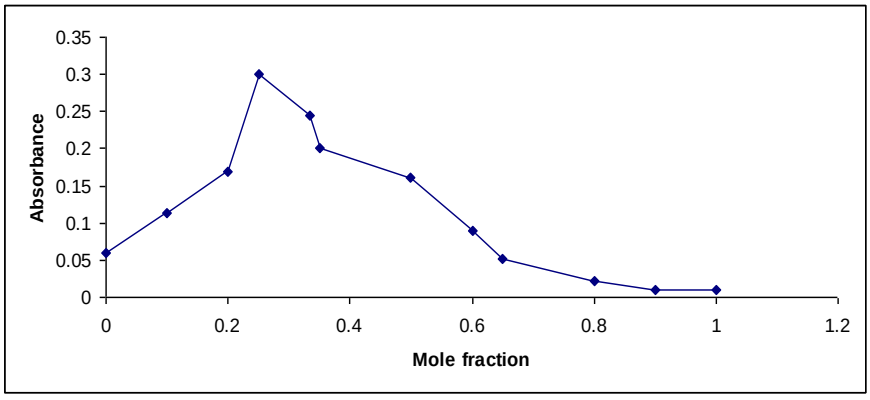

Fig 2: Job's Continuous variation method

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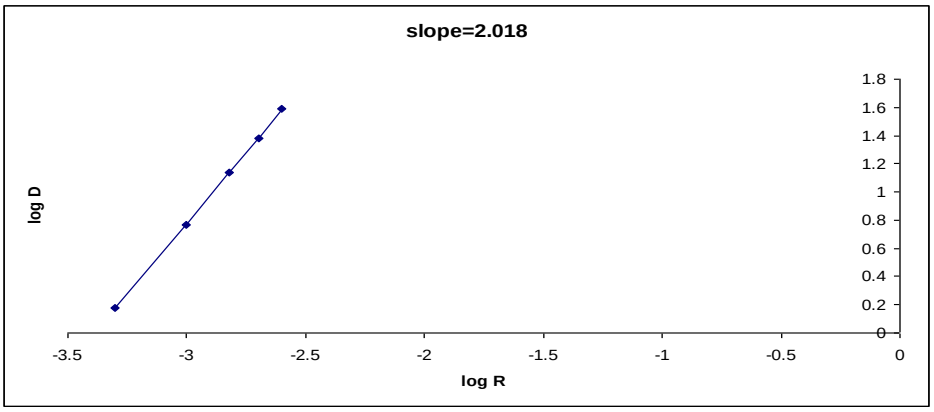

Fig 3: Slope ratio method for Fe (II): HCHNM complex

EFFECT OF DIVALENT IONS AND FOREIGN IONS

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 60 µg of Iron (Table 1)

Table 1: Effect of the interference of some cations on Absorbance of Fe (II): HCHNM complex in n-butanol

Sr.No.	Metal	Amount added in mg	Absorbance at 500 nm
1	--	--	0.136
2	Na(I)	13	0.136
3	K(I)	14	0.136
4	Ag(I)	9	0.136
5	Sr(II)	9	0.136
6	Mo(II)	7	0.136
7	Mn(II)	8	0.136
8	Mg(II)	10	0.136
9	Hg(II)	5	0.136
10	Rh(IV)	8	0.136
11	Tl(I)	9	0.136
12	Th(II)	8	0.136
13	V(VI)	9	0.136
14	Ce(III)	8	0.136
15	Cd(II)	7	0.136
16	Zn(II)	10	0.136
17	Al(III)	9	0.136
18	Ni(II)	5 **µg**	0.136
19	U(VI)	10 **µg**	0.136
20	Cu(II)	10 **µg**	0.136
21	Co(II)	10 **µg**	0.136

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PRECISION AND ACCURACY

The precision and accuracy of the spectrophotometric method have been studied by analyzing five solutions each containing 100 µg of Iron. Aliquot used is 100µg/ml

Standard deviation is 0.1673
Confidence limit at 99% is 49.96± 0.3016

APPLICATIONS

The newly developed method has been successfully applied for the determination of Iron from various alloys, ores and pharmaceutical samples. The results indicate that the developed method is compatible with the standard known method. (Table 2).

Table 2: Determination of Fe (II) using HCHNM from different samples

Sr.No.	sample	Amount of Fe(II) in mg	
		Standard method	**Present method**
1.	**Alloys**		
	1) Hematite	35.0 %	34.99 %
	2) Steel	67.2 %	67.19 %
2.	**Capsule/ tablets**		
	1) Austrin	32.86 mg	32.858 mg
	2) Globiro	50.0 mg	49.98 mg
	3) Raricap	25.0 mg	24.97 mg
	4)Injection Viol	50mg /ml	49.98 mg/ml
3.	**Synthetic mixture**		
	1)Fe(II)(5) +Zn(II)(5)	4.99 ppm	4.97ppm
	2)Fe(II)(5) +Mg(II)(5)	4.99ppm	4.98ppm

Every result is an average of three independent determinations.

CONCLUSION

The results obtained show that Hydrazine Carboxamide-2-[(2-Hydroxy-1-Naphthalenyl) Methylene] in n-butanol can be effectively used for quantitative extraction of Fe (II) from aqueous media. The proposed method is quick and requires less amount of organic solution. The equilibrium time required is very less and the complex is stable for 72 hours. The results show good agreement with the standard method. The method is very fast, accurate and precise.

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