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New Analytical Technique for Determination of Trace Amount of Fe (III) by Using UV-Visible Spectrophotometric Method with Photometric Reagent

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ABSTRACT

3-(2-Hydroxylphenylimino)indolin-2-one [HPI2O], a new Analytical reagent is proposed as a sensitive spectrophotometric reagent for Fe(III). The reagent HPI2O is synthesized in the laboratory and characterized by NMR, IR and elemental analysis. A selective spectrophotometric method is presented for the trace determination of Fe(III) using HPI2O as spectrophotometric reagent (λ_{max}=430 nm) in acidic aqueous solution (pH=4.2). The Beer's law is obeyed in the concentration range from 1 to 5 ppm. The HPI2O forms a 1:2 brown coloured complex. The Sandell's Sensitivity is 0.016194 µg cm^{-2} with molar absorptivity 3448.7 L mol^{-1} cm^{-1}. The proposed method has been successfully applied to the determination of Iron in various real and synthetic samples. The precision and the accuracy obtained were satisfactory.

Keywords: Iron, Spectrophotometric determination, n-Butanol, Reagent, HPI2O.

INTRODUCTION

Iron and its compounds are widely used in alloys and various biological samples. Trace concentrations of Iron can also affect the physical and mechanical properties of metal and alloys. Iron is essential to nearly all known organisms. The use of iron metal fillings in organic synthesis is mainly used for the reduction of nitro compounds. Therefore, precise knowledge of the Iron present in a various samples is required, for which an accurate assessment of the Iron is an increasing need of analytical methods for determination of micro-trace or ultra-trace level[10,11]. Many methods have been reported for the determination of Iron such as inductively coupled plasma atomic emission or mass spectrometry[1],[2] require expensive instruments and well-trained operators. Some of the reagents used for the spectrophotometric determination of Iron are Bathophenanthroline[3], Mercapto pyridine-1-oxide[4], Pyridazine-3,6-diol[5], HMBAINH [16] etc. However, most of these methods suffer from certain limitation, such as interference by number of ions[6], [7] of low sensitivity[8].

The purpose of this work is to find selective, sensitive, simple method for exact determination of iron by using new analytical reagent[17,18]. In this paper a new method has been developed using 3-(2-Hydroxylphenylimino)indolin-2-one [HPI2O] for extraction and spectrophotometric determination of Iron,

which is simple, selective and sensitive. spectrophotometric determination is widely used due to its simplicity[19].

MATERIALS AND METHODS

The pH measurements were made using a pH meter Elico, Model LI-129, India in conjugation with a combined glass and calomel electrode. Shimadzu UV-Visible 2100 spectrophotometer with 1.0 cm matched quartz cells were used for all absorbance measurements.

Reagent and chemicals: 0.1% HPI2O reagent is prepared by dissolving the requisite amount of HPI2O in a known volume of ethanol. All chemicals used were of analytical-reagent grade or the highest purity available. Doubly distilled de-ionized water and A.R. grade ethanol, which is were used throughout.

Fe (III) standard solutions: The stock solution of trivalent Iron was prepared by dissolving weighed amount of ammonium ferric sulphate in doubly distilled de-ionized water containing 1–2 mL of sulphuric acid. More dilute standard solutions were prepared from this stock solution as and when required.

PROCEDURE FOR THE EXTRACTION: 1 mL of aqueous solution containing 10 µg of Iron metal and 2 mL of reagent was mixed in a 50 mL beaker. The pH of the solution adjusted to 4.2, it must be noted that the total volume should not exceed 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 passed through anhydrous sodium sulphate in order to absorb trace amount of water from organic phase and then collected in 10 mL measuring flask and made up to the mark with organic solvent if required. The amount of Iron present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 430 nm and that in the aqueous phase was determined by thiocyanate method.

RESULTS AND DISCUSSION

The results of various studies are discussed below.

Extraction as a function of pH: The extraction of Iron with 3-(2-Hydroxylphenylimino)indolin-2-one has been studied over the pH range 1-10 and was observed that percentage extraction of Fe(III) is maximum at pH range 3.8 -4.4 . Hence, further extraction and determination carried out at pH 4.2.(Fig I).

Absorption spectrum: The absorption spectrum of Fe(III):HPI2O in n- Butanol shows the maximum absorption at 430 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 430 nm (Fig II).

Influence of diluents: The suitability of solvent was investigated using various organic solvents and the extraction of Fe(III): HPI2O was quantitative in n-Butanol. Hence, n-Butanol was used for further extraction studies as it gave better and quicker phase separation.

Effect of reagent concentration: It was found that 1 mL of 0.1% reagent is sufficient for the colour development of the metal Fe(III) in 10 mL of aqueous solution at pH 4.2

Effect of equilibration time and stability of the complex: The equilibration time of 1 minute is sufficient for the quantitative extraction of Iron. The stability of colour of the Fe(III):HPI2O complex with respect to time shows that the absorbance due to extracted species is stable up to 48 hours, after which slight decrease in absorbance is observed.

Calibration plot: The Beer's law is obeyed from 1 to 5 ppm. The molar absorptivity and sandell's sensitivity were calculated to be is 3448.7 L $mol^{-1}cm^{-1}$ and 0.016194 µg cm^{-2} respectively (Fig III).

LOD: LOD[10] (Limit Of Detection) of the present method was calculate at 98.3 % confidence level, it was 0.105 µg/mL.

Effect of divalent ions and foreign ions: The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10 ppm of Iron. The ions which show interference in the spectrophotometric determination of Iron were overcome by using appropriate masking agents (Table I).

Precision and accuracy: The precision and accuracy of the developed spectrophotometric method have been studied by analyzing ten solutions each containing 3 µg of Iron in the aqueous phase. The average of ten determinations was 3.0058 and variation from mean at 95% confidence limit was ± 0.01096.

Nature of extracted species: The composition of extracted Fe(III):HPI2O complex has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of Fe(III):HPI2O complex is 1: 2. (Fig IV).

APPLICATIONS

The proposed method was successfully applied for the determination of Iron from various alloys and pharmaceutical samples. The results found to be in good agreement with those obtained by the standard known method. (Table II)

Determination of Fe (III) in alloys/ore: About 0.3 to 0.5 g sample (alloy/ore) was dissolved in 15 cm^3 of aqua–regia. The solution was evaporated to dryness and the residue was treated with concentrated HNO_3 and diluted to 100 cm^3. An aliquot of a diluted solution was used for the extraction and spectrophotometric determination of Fe(III) by present method.

Tablet: To the tablet 1.0 cm^3 of concentrated HNO_3 was added and evaporated to dryness. It was treated with 5.0 cm^3 of 30 % H_2O_2 every time, till solution become colourless. The colourless solution was then treated with dilute HCl and evaporated to dryness. The residue was dissolved in 10 cm^3 distilled water and aliquot of this was used for Fe(III) analysis by the present method.

Synthetic mixture: The separation of Iron(III) from synthetic mixture of associated metals containing Co(II) and Cu (II) with varying combination was carried out. A definite aliquot of this solution was taken and after the adjustment of acidity of the aqueous solution to pH 4.2 and addition of 1 ml of 0.1% HPI2O solution, the iron complex formed was extracted into 10 ml of n-Butanol. The amount iron present was computed using the calibration curve method. The result obtained is compared with those obtained by standard method.

CONCLUSIONS

The results obtained show that the newly developed method in which the reagent HPI2O was used, can be effectively used for quantitative extraction and estimation of Fe(III) from aqueous media. The proposed method is quick and requires less volume of organic solvent. The result show good agreement with the standard method. The method is very precise, faster and simpler than other methods.

The developed method is compared with result obtained with the thiocyanate method for the estimation iron(III) and observed to be comparable. The method is precise, accurate, less time consuming and easily

employed anywhere, even in small laboratories as it requires only uv – visible spectrophotometer and not much sophisticated and costly measurement devices or instrumentation.

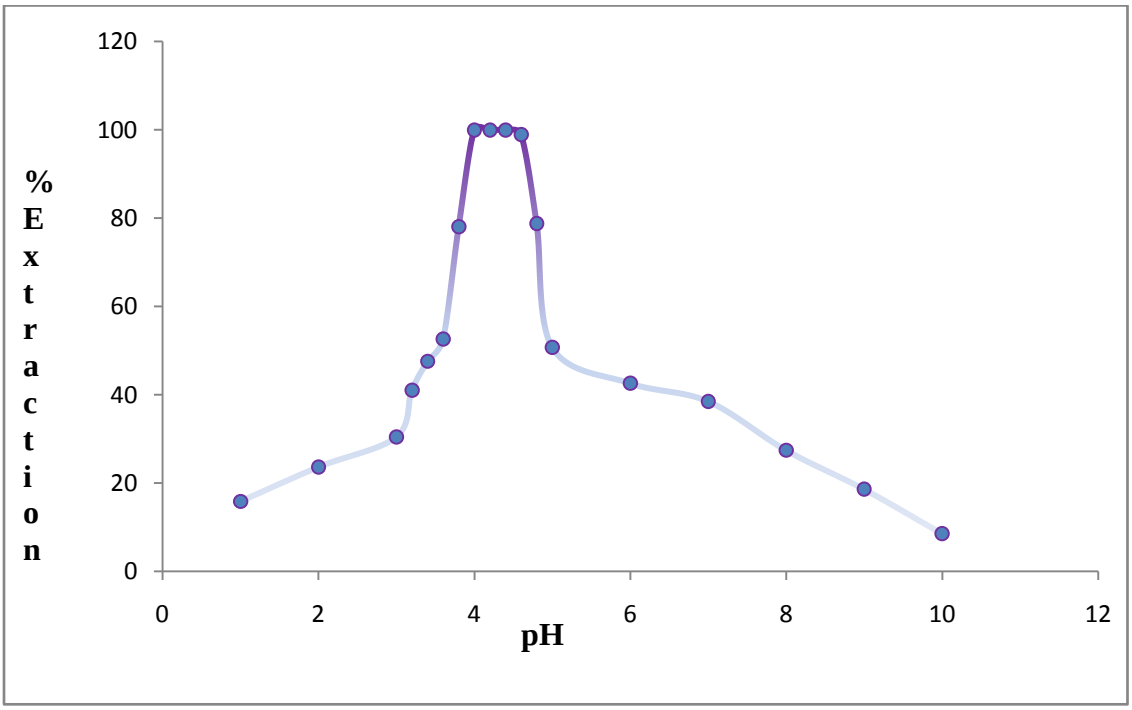

Fig I: Extraction as a function of pH

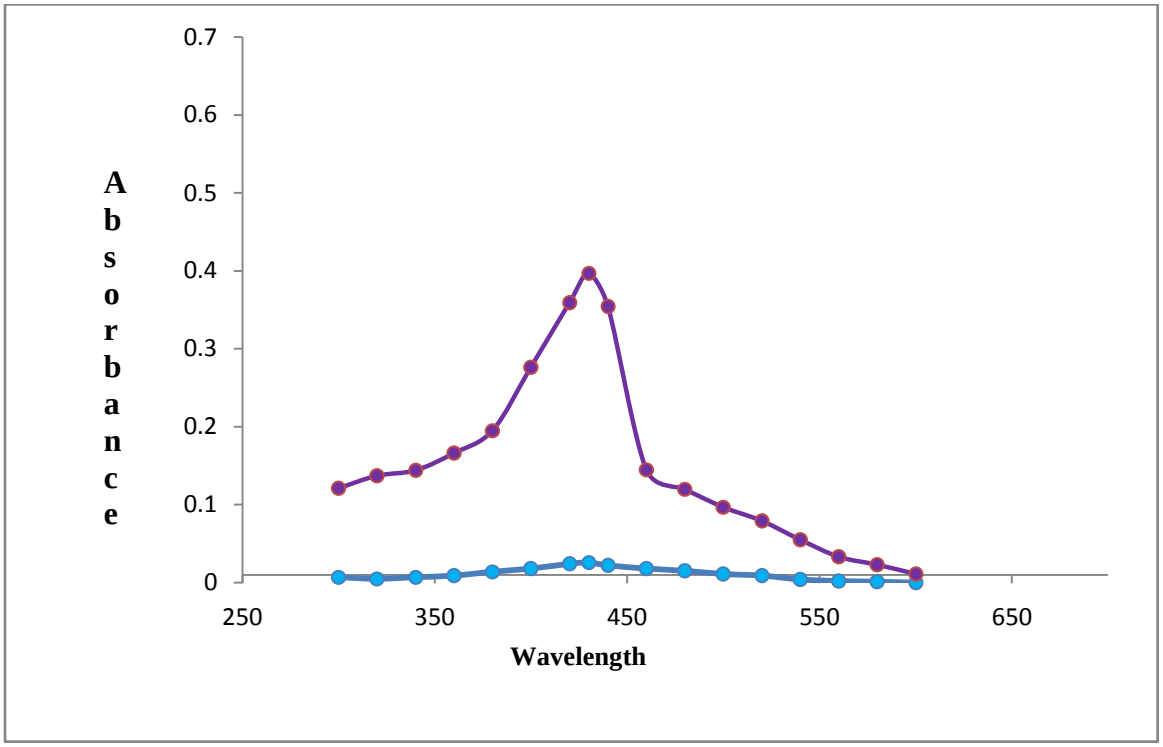

Fig. II: Absorption spectrum

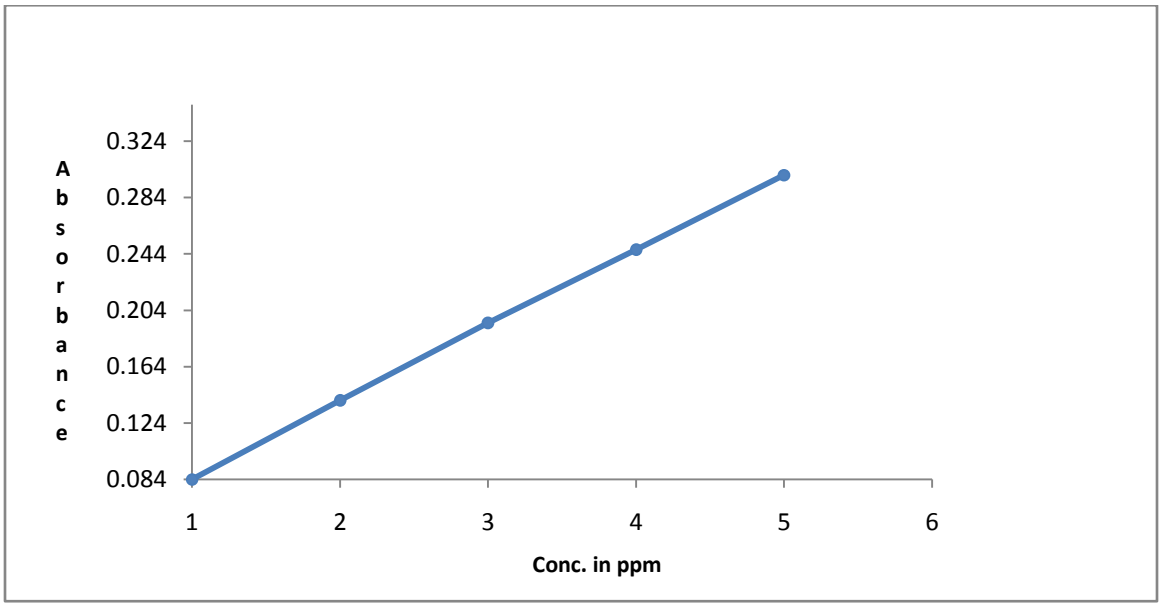

Fig III: Calibration plot

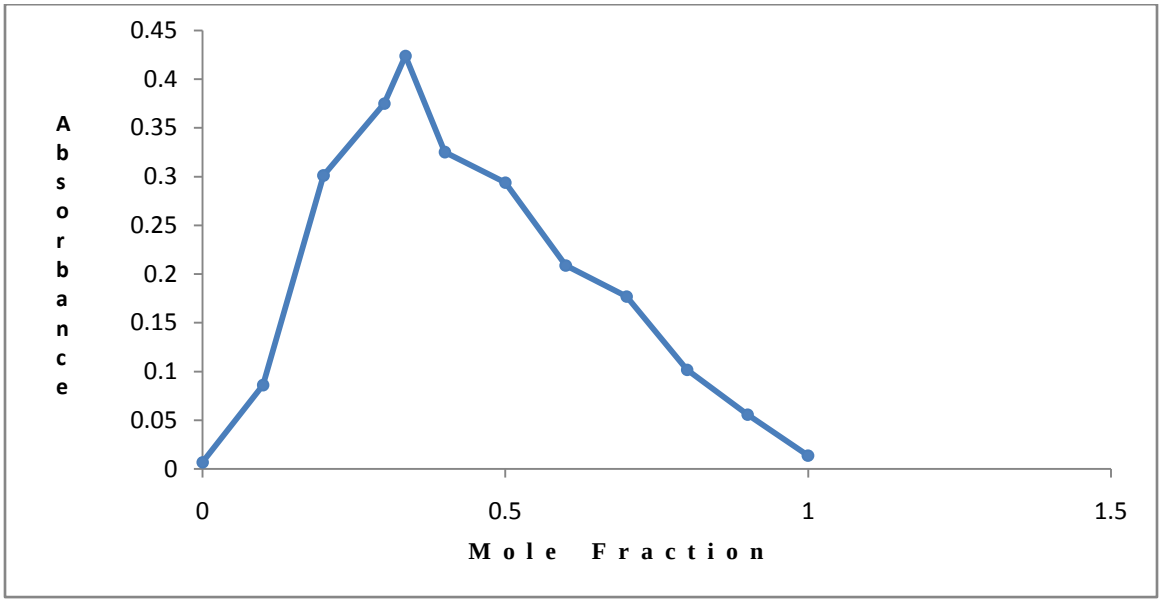

Fig IV: Job continuous variation Nature of extracted species

Table I: Use of masking agent

Sr. No.	Interfering Ion	Masking agent
1	Cu (II)	Thiosulphate
2	Ti (IV)	Ascorbic acid
3	Th(IV)	Sodium fluoride

4	EDTA	Boiled with concentrated HNO_3
5	CN^-	Boiled with concentrated HNO_3 and formaldehyde

Applications of proposed method

Table II: Applications

Sr. No.	Samples	Amount of Fe (III) Standard method	Amount of Fe (III) Present method
1.	**Alloy/ Ore (0.3 g)**		
a)	Hematite ore (53.06 % Fe)	0.159 g	0.156 g
b)	Elinver alloy (62.7 % Fe)	0.188 g	0.186 g
2.	**Pharmaceutical sample**		
	Supradyn (Multivitamin Tablet)	11.17 mg	11.22 mg
3.	**Synthetic mixture**		
a)	Fe (III) (5) + Cu (II) (2.5)	4.97 ppm	4.97 ppm
b)	Fe (III) (5) + Co (II) (5)	4.89 ppm	4.85 ppm

1. Each result is average of three independent determinations.
2. Each result is compared with standard thiocyanate method.

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