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Original Article

DEVELOPMENT OF EXTRACTIVE SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF IRON (III) WITH SCHIFF BASE 2-[(2-HYDROXYPHENYLIMINO) METHYL]-4-NITROPHENOL

DATTA B. MANDHARE, VASANT D. BARHATE*

V. E. S. College of Art, Science and Commerce, Sindhi Society Chembur, Mumbai 400071, India
Email: vasantbarhate@yahoo.co.in

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ABSTRACT

Objective: A simple spectrophotometric method has been developed for the determination of Iron (III) by using Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP].

Methods: HPIMNP extracts Fe (III) quantitatively (99.95%) into chloroform from an aqueous solution of pH range 4.0-6.0.

Results: The chloroform extracts show maximum absorption at 510 nm (λ max). Beer's Law is obeyed over the Fe (III) concentration range of 0.5 to 20.0 µg/ml. The Molar absorptivity and Sandell's sensitivity for Fe–HPIMNP system is 5000 L mol $^{-1}$ cm^{-1} and 0.011 µg cm^{-2}respectively. The composition of extracted species is found to be 1: 3 [Fe-HPIMNP] by Job's continuous variation and Mole-ratio method. Interference by various ions has been studied.

Conclusion: The proposed method is rapid, sensitive, reproducible and accurate and it has been satisfactory applied for the determination of Iron in Pharmaceutical Samples.

Keywords: Solvent Extraction, Extractive Spectrophotometry, Iron (III), Schiff base, 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP], Pharmaceutical sample.

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INTRODUCTION

Various reagents [1] are available for the spectrophotometric determination of Iron (III) of which Oximes, Schiff bases and it derivatives constitute an important class [2, 3]. Synthesis and Antimicrobial Activity of Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP] have been reported [4]. However, Analytical application of HPIMNP was not studied. In the present communication, we describe the extractive spectrophotometric determination of Fe (III) with 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP].

MATERIALS AND METHODS

ELICO-SL 159 spectrophotometer with optically matched quartz or glass cells of 1 cm path length were used for absorbance measurement. An ELICO–LI 127 pH meter was employed for pH measurements. The reagent HPIMNP was synthesized by condensation of 5-Nitro salicylaldehyde with 2-amino phenol as per reported procedure [4]. The resulting product was recrystallized by using ethanol [5] and characterized by elemental and spectral analysis. Its 0.5 % solution was prepared in dimethyl formamide (DMF). A stock solution of Fe (III) was prepared by dissolving ferric ammonium sulfate in double distilled water containing dilute sulphuric acid. It was standardized by thiocyanate method [6]. Working solutions of Fe (III) were made by suitable dilution. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

Extraction and separation of Fe (III)

To an aliquot of an aqueous solution containing 500 µg of Fe (III) and 2 ml of 0.5 % solution of HPIMNP prepared in DMF were mixed in 25 ml beaker. The pH of the solution was adjusted to desired value with a dilute solution of HCl/NaOH, Keeping the total volume to 10 ml with distilled water. The resulting solution was then transferred into 125 ml separatory funnel. The beaker was then washed with 5 ml portion of organic solvent twice, and each washing was added to the solution in a separatory funnel. The two phases were equilibrated for one minute and allowed to separate. After the separation of two phases, pH of the equilibrated aqueous phase was measured and Iron content in each phase was determined by Thiocynate method [6]. The extraction was carried out with different solvents to find out the best extracting solvent. On the basis of Iron content in aqueous and an organic phase, extraction coefficient and percent extraction were calculated.

Extractive spectrophotometric determination of Fe (III*)*

To an aliquot of an aqueous solution containing 5-200 µg of Fe (III), 2 ml of Potassium hydrogen phthalate (KHP) and sodium hydroxide buffer solution of pH 5 and 1 ml of 0.5 % solution of HPIMNP prepared in DMF were added. The volume of solution was made up to 10 ml with distilled water. The solution was equilibrated for one minute with 10 ml of chloroform and the phases were allowed to separate. The chloroform extract was collected in a 10 ml measuring flask and made up to mark with chloroform. The absorbance of chloroform extract was measured at 510 nm against a reagent blank prepared under identical conditions. The Fe (III) content of the sample solution was determined from the calibration curve. To study the effect of other ions, the respective foreign ions were added to aqueous phase before the extraction and adjustment of pH.

Determination of iron in pharmaceutical sample

2 ml of pharmaceutical sample was dissolved in boiling with 10 ml aquaregia. The resulting solution was evaporated to dryness, and the residue was then dissolved in 10 ml of 1 M HCl, filter if required, and the solution was diluted to 100 ml with double distilled water. To an aliquot of this solution (1 ml) was analyzed for Iron by the procedure as described earlier.

RESULTS AND DISCUSSION

Iron (III) could be extracted quantitatively (99.95%) by HPIMNP into chloroform from an aqueous solution of pH range 4.0–6.0 (fig. 1). Organic solvents used for extraction of Fe (III) can be arranged on the basis of their extraction coefficient values as chloroform> carbon tetrachloride>xylene>toluene>benzene>Nitro benzene> ethyl acetate>n-butanol>Iso amyl alcohol>benzyl alcohol (fig. 2). Chloroform was found to be the best extracting solvent; hence, it was selected for extraction throughout the work.

The chloroform extract of Fe-HPIMNP complex showed an intense peak at 510 nm. The absorbance due to the reagent is negligible at this wavelength, so the absorption measurements were taken at this wavelength (fig. 3). The result shows that the system confirmed to Beer's law at this wavelength over a Fe (III) concentration range 0.5 to 20 µg/ml (Fig-4). The molar absorptivity and Sandell's sensitivity of the extracted species on the basis of Fe (III) content were calculated to be 5000 L mol^{-1} cm^{-1} and 0.011µg. cm^{-2} respectively. It was found that 1 ml of 0.5 % solution of HPIMNP prepared in DMF was sufficient to extract 200 µg of Fe (III). The color of the chloroform extract was found to be stable at least 48 hrs at room temperature.

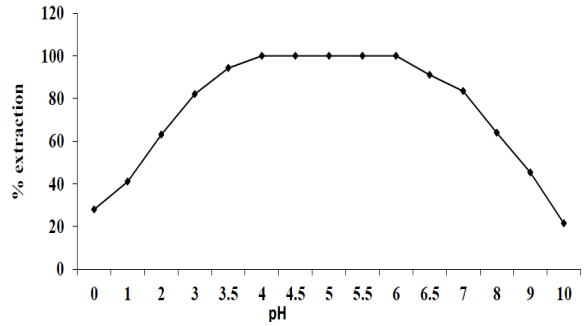

Fig. 1: Effect of pH on the extraction of iron with HOIMNP into Chloroform

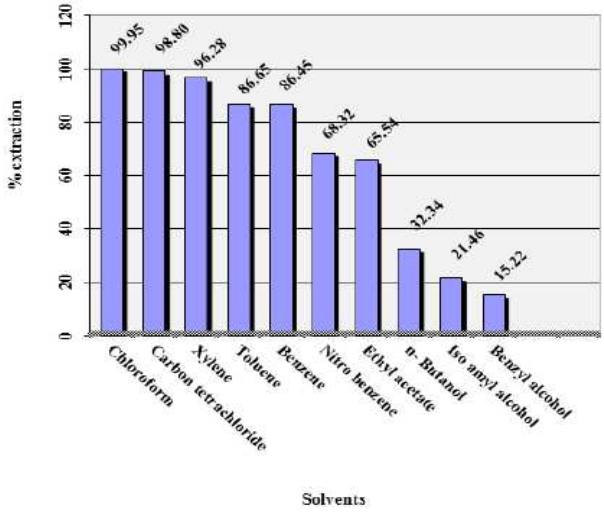

Fig. 2: Effect of solvents on extraction of Fe (III)

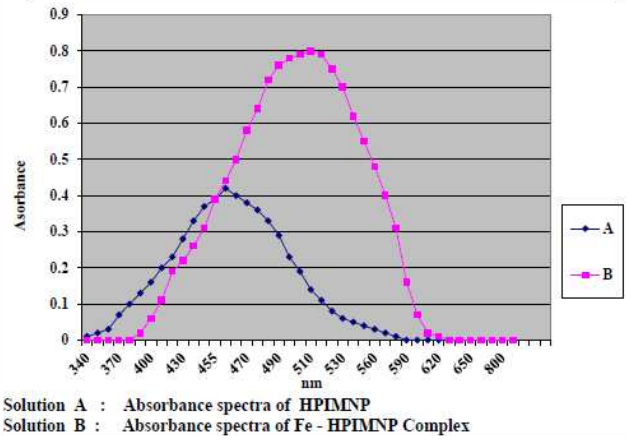

Fig. 3: Absorption spectrum of HPIMNP and Fe-HPIMNP in Chloroform

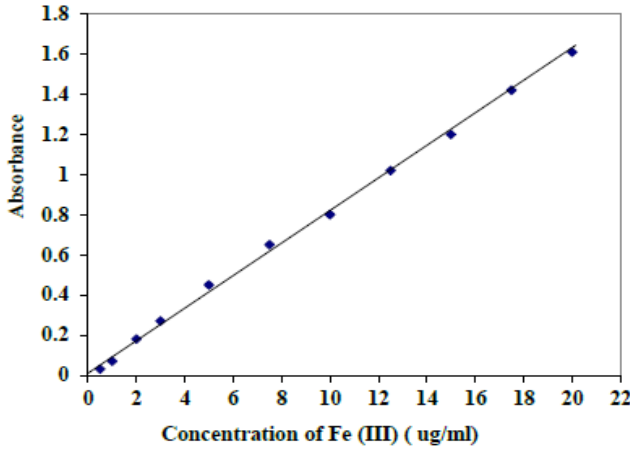

Fig. 4: Calibration curve for iron

Effect of other ions

Fe (III) (100 µg) was determined in the presence of various ions. The following ions in the amount indicated, did not interfere in the spectrophotometric determination of Fe (III)(100 µg): 10 mg each of Li (I), Be (II), Ba (II), Ca (II), Sr (II), Al (III), Ti (III), V (V), Mo (VI), U (VI) & Ni (II). 100 ppm of each Os (IV), Pd (II), Pt (IV), Ru (III) and Rh (III). And 20 mg each of chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, sulphide, phosphates, tartrate, acetate, citrate and thiosulphate, triethanolamine, ascorbic acid.

Interference by various ions was removed by using appropriate masking agent (table 1).

Table 1: Masking agents required for suppressing the interference by other ions:

Interfering ion	Amount added in mg	Masking agent added 1 ml of 2M solution
Cu (II)	10	Sodium thiosulphate
Co (II)	10	Ascorbic acid
Cr (III)	10	Triethanolamine
Ag (I)	10	Potassium bromide

Composition of the extracted complex

The composition of the extracted complex was found to be 1:3 (Fe: HPIMNP) by Job's continuous variation and Mole ratio methods (fig. 5 & fig. 6).

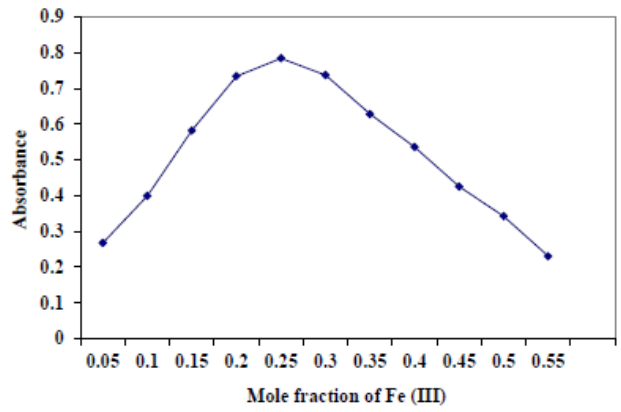

Fig. 5: Job's continuous variation meth

Table 2: Determination of iron in pharmaceutical sample

Pharmaceutical Sample	Certified amount of Iron present	Iron (III) found	
		Present method	Thiocyanate method
Impheron Injection	50 µg/ml	49.8 µg/ml	49.6 µg/ml
Ferrochelate drops	1.33 mg/ml	1.32 mg/ml	1.31 mg/ml

Results are the average of three independent determinations.

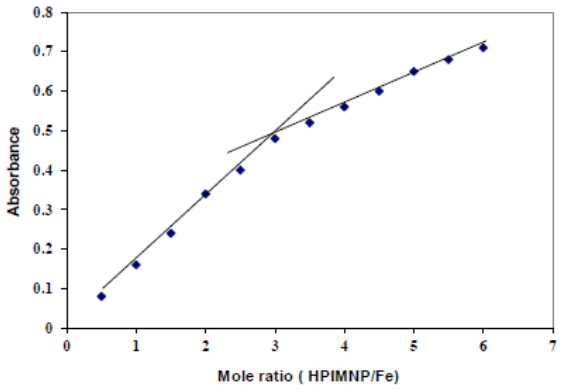

Fig. 6: Mole ratio method

Precision, accuracy, sensitivity and application of method

The precision and accuracy of the method were tested by analyzing the solution containing a known amount of Fe (III) following the recommended procedure. The average of 10 determination of 5 µg of Fe (III) in 10 cm^3solutions was 4.98µg, which is varied between 4.92 and 5.08 at 95% confidence limit and standard deviation is±0.113. The proposed method has been applied for the determination of Iron in Pharmaceutical sample. The results of the analysis of the sample were comparable with those obtained by the thiocyanate method [6] (table 2).

CONCLUSION

From the above discussions, it is found that Schiff base, 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP] is a good sensitive reagent for the development of rapid and sensitive extractive spectrophotometric method for the determination of Fe (III) and it has been satisfactory applied for the determination of Iron in Pharmaceuticals Samples.

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CONFLICT OF INTERESTS

Declared None

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