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Analytical method development for extractive spectrophotometric determination of Fe(II) as a complex with 1, 2 Propanedione,1-phenyl-1-(2-hydroxy-5-bromobenzilidine azine) -2-oxime (PDPHBBAO)

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Abstract: 1, 2 Propanedione,1-phenyl-1-(2-hydroxy-5-bromobenzilidine azine) -2-oxime (PDPHBBAO) was synthesized and characterized by FTIR, NMR, elemental analysis as well as Mass spectrometry. This reagent was then applied for the development of the analytical method for the extractive spectrophotometric determination of Iron (II). Iron metal forms faint brown coloured complex, which can be extracted in chloroform at pH 8.0 having absorption maxima at 445 nm. Beer's law is obeyed in the concentration range 1-10 µg. The molar absorptivity and Sandell's Sensitivity of the extracted species are found to be 4.96 X 10^3 Lit mol^{-1} cm^{-1} and 11.27 X 10^{-3} µg / cm^2 respectively

The developed method is highly sensitive, selective, simple, rapid, accurate, and has been satisfactorily applied for the determination of iron in the synthetic mixtures, pharmaceutical samples, and alloys.
Keywords: Characterization; Iron; Extractive Spectrophotomertic determination.

Introduction

Iron is essential to nearly all known organisms. In cells, iron is generally stored in the centre of metalloproteins. The use of iron metal filings in organic synthesis is mainly for the reduction of nitro compounds.(1) Additionally, iron has been used for desulfurizations,(2) reduction of aldehydes,(3) and the deoxygenation of amine oxides.(4) Knowing the importance of Iron, a method highly sensitive, selective, simple, rapid, accurate and superior to reported methods in the literature(5-30) has been developed for its extractive spectrophometric determination The developed method has been satisfactorily applied for the determination of iron in the synthetic mixtures, pharmaceutical samples, and alloys.

Experimental

The **PDPHBBAO** was synthesized[31,32,33], characterized[34] and used for extractive spectrophotometric determination of Fe(II) A stock

solution of PDPHBBAO (Concentration 0.005%) was prepared in methanol.

Iron (II) Solution

A weighed quantity of ferous sulphate was dissolved in double distilled water containing dilute sulphuric acid and then diluted to the desired volume using double distilled water. The iron solution was then standardized by o-phenanthroline method[35].

Recommended procedure

Mix 1cm^3 aqueous solution containing 1-100µg of iron and 1 cm^3 of 0.005 % methanolic solution of PDPHBBAO in 25 cm^3 beaker. Adjust the pH of the solution to required value with dilute solution of H_2SO_4 and NaOH. Make the final aqueous volume up to 10 cm^3. Transfer the solution into 125 cm^3 separating funnel and equilibrate for 1min with 10 cm^3 chloroform. Allow the two phases to separate and measure the absorbance of the organic extract containing the complex at 445 nm against reagent blank.

Effect of foreign ions

The effect of diverse ions on the iron (II) determination was studied, in presence of a definite amount of a foreign ion. Various cations and anions were investigated in order to find the tolerance limit of these foreign ions in the extraction of iron (II) (Table 2). The tolerance limit of the foreign ion was taken as the amount required causing an error of not more than +2% in the recovery of Iron (II). The ions which interfere in the spectrophotometric determination of iron were masked by using appropriate masking agents. (Table 3).

Table : 1 Results and Discussion

Condition	Results
Absorption Maxima	445 nm
Solvent	Chloroform
pH range	7.5 to8.5.
Equilibration time	1.0 min
Stability of Iron-PDPHBBAO	8 h
Beer's range	1 to 10 µg/ cm^3
Molar absorptivity	4.96 X 10^3 Lit mol^{-1} cm^{-1}
Sandell's sensitivity	11.27 X 10^{-3} µg / cm^2
Mole Ratio of Fe : PDPHBBAO	1 :3

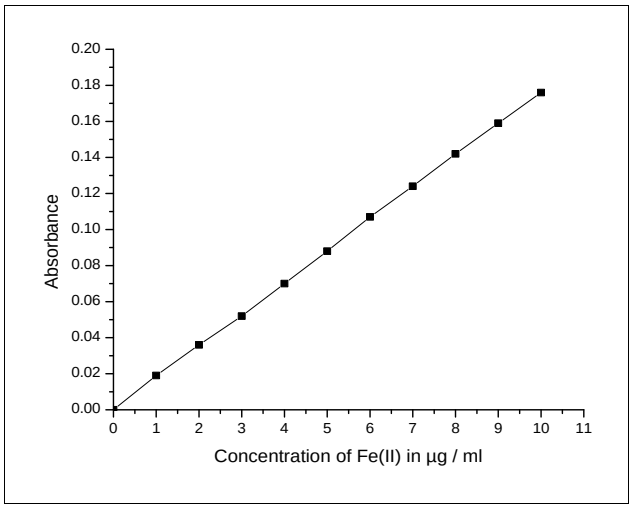

Fig. 1 Calibration Plot

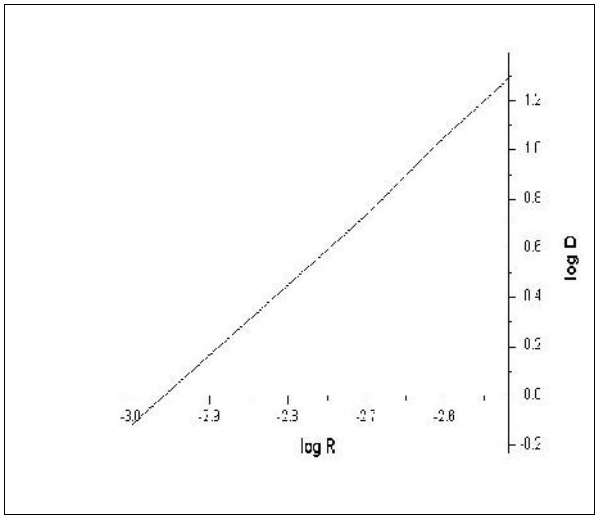

Fig. 2 Slope Ratio Method

Table 2 Effect of foreign ions

Anion added	Amount added in mg.	Cation dded	Amount added in mg.
Chloride	15	Ca	5
Fluoride	15	Sr	5
Bromide	15	Ba	10
Iodide	10	Mo	10
Bromate	10	Mn	10
Iodate	10	Mg	10
Chlorate	10	V	5
Chromate	20	Rh	2
Dichromate	10	Tl	5
Carbonate	10	U	5
Phosphate	10	Th	5
Urea	20	Li	10
Thiourea	10	Ce	5
Acetate	20	Zr	5
Thiosulphate	20	Cd	5
Oxalate	20	Al	5
Nitrate	20	Hg	10
Nitrite	20	As	10
Sulphate	20	Zn	5

Table No 3 Effect of masking agent

Interfering Ion	Masking agent added	Interfering Ion	Masking agent added
Ag(I)	Potassium iodide	Citrate	Sodium molybdate
Cd(II)	Potassium iodide	Tartarate	Sodium molybdate
Pb(II)	Sodium thiosulphate	EDTA	Boiling with concectrated HNO_3
Mn(II)	Sodium fluoride	CN^{--}	Boiling with concentrated HNO_3 and formaldehyde
Ce(IV)	Sodium fluoride	Zr(IV)	Sodium fluoride
Cr(II)	Ammonium acetate	Ni(II)	Thiourea

Applications

The present method was applied for determination of amount of Iron (II) in various samples as alloys, synthetic mixtures, injection vial, tablets and industrial waste water. The results obtained were well in agreement with those of standard methods.Table 4.

Table 4 Applications

Sr. No.	Sample	Amount of Fe(II)	
		Standard Method	**Present Method**
1.	**Alloy /Ore**		
a)	Hematite	35.0%	34.98%
b)	Steel	67.2%	66.99%
2.	**Capsule/tablet**		
a)	Injection Viol	50 mg/ml	49.9 mg/ml
b)	**2B12 (Capsule)**	1.5 mg	1.49 mg
3.	**Synthetic Mixture**		
a)	Fe(II)(5)+Zn(II)(5)	4.98 ppm	4.99 ppm
b)	Fe(II)(5)+Mg(II)(5)	4.98 ppm	4.99 ppm
4.	**Industrial Waste Water at Ulhasnagar Creek**	0.5 ppm	0.49 ppm

Every result is average of three independent determinetions.

Conclusion

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

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