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Spectrophotometric Determination Of Iron (III) In Biological Samples

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Abstract
Iron (III) serves as a key component of the enzyme systems and proteins with vital functions at optimum levels. Its excessive intake causes siderosis while its deficiency causes anemia therefore, it is of interest to determine iron (III) in biological samples, foodstuffs and pharmaceutical formulations. A rapid sensitive and selective spectrophotometric method for the determination of trace amount of iron(III) has been developed based on the extraction of iron(III) from an aqueous solution of pH 1.9-2.5 with bis(2,4,4-trimethyl pentyl dithiophosphonic acid in toluene. The optimum shaking time and reagent concentration for the maximum absorption have been evaluated. The green-colored toluene extract exhibits maximum absorption at 613nm the molar absorptivity was found to be $5.048x10^3$/mol-1cm-
1. Beers law is obeyed in the range of 0.2-4.8 ppm of iron (iii). sandell's sensitivity is 0.102µg iron (III) cm.
2. The log-log plot at pH 2.2 gave a linear graph with a slope of 2.8 indicating the complex extracted into toluene is 1:3 complex of metal reagent. The effect of various diverse cations and the anions on the extraction of 50µg of iron (III) have been studied and found that method is selected. The method has been applied for the determination of iron (III) in the human hair sample, some pharmaceutical formulations and foodstuffs and compared with the ICP-AES method.

Keywords:
Iron (III), bis-(2-4, 4-trimethyl pentyl dithiophosphonic acid, spectrophotometry.

Introduction:
Iron (III) serves as a key component of enzyme systems & proteins with vital functions at optimum levels. Its excessive intake causes siderosis while its deficiency causes anemia (1). Therefore, it is interesting to determine iron (III) in biological samples, foodstuffs & pharmaceutical formulations.

Earlier reported methods (2-6), for the spectrophotometric determination of iron (III), suffer from drawbacks like low sensitivity, variation in color intensity & interference due to the formation of colored products of other transition metals. We have developed a new spectrophotometric method for the determination of iron (III) based on the extraction of the colored complex of iron (III) with bis-(2, 4, 4-trimethyl pentyl dithiophosphinic acid) in toluene. The method is rapid, sensitive & selective.

Experimental:
1. Instruments used:
A Shimadzu UV-160A visible spectrophotometer with quartz cells of 1 cm. and a control dynamic digital pH meter with combined glass electrode were used for absorbance and pH measurements.
2. The stock solution of iron (III):
2.1585 gm. of NH_4 Fe (SO_4), 12H2O/250ml distilled water containing 3ml H_2SO_4. Standardized by titrimetrically 0.1N $K_2Cr_2O_7$ solutions. Working standards were prepared by suitable solutions of this stock solution.
3. Stock solution of reagent (0.1 mol dm^3):
3.226g of bis-(2, 4, 4-trimethyl pentyl dithiophosphonic acid)/100ml of toluene.

4. General procedure:
Adjust the pH of an aliquot aqueous solution containing iron (III) to the desired pH in a total volume of 25 ml. Transfer the solution to a separating funnel. Add 10ml of reagent solution in toluene & shake for 3 min. allow the two phases to separate. Collect the organic phase in a beaker & dry over anhydrous sodium sulphate. Measure the absorbance at 613 nm against the blank prepared similarly. The above method was applied in presence of various diverse ions the results are given in Table No.1.The tolerance limit for magnesium ion as a foreign ion was more than 2% in the extract causing error and not the absorption value. Most of the ions were tolerated in the range of 1:10, expect

Cu(II),EDTA(1:1), Co(II)(1:2),Mo(VI)(1:5). In most of the pharmaceutical, biological and food samples the amount of copper is generally less than iron. Therefore the present method is selective in the estimation of iron in these samples.

5. Procedure for Analysis of Human Hair:
The hair samples were first washed with acetone 2-3 times in a beaker with constant stirring & dried in an oven at 110 c for 3-4 hrs. 2g of hair digested with 1:1 HNO_3:$HClO_4$ on a hot plate. The solution was evaporated near to dryness. The ash was taken up with 5ml of 5ml dm^3 sulphuric acid, filtered and made up to 50 ml with water. An adequate of the solution was then treated according to the present procedure & ICP-AES.

6. Procedure for Analysis of foodstuff:
Rice, wheat flour, banana & tomato were oven-dried at 90ºC for 24 hrs. A 5g sample was digested with nitric acid & perchloric acid & heated gently on a hot plate to dryness. The ash was taken up with 5ml HCL and evaporated to dryness again & dissolved in 2ml conc. HCL & water, filtered & made 25ml. an aliquot of each solution was treated according to the present procedure by ICP-AES. The result for the determination of iron (III) in foodstuffs & human hairs is given in Table No.2

7. Procedure for Analysis of Pharmaceutical samples:
The contents of capsules or tablets were ignited in a muffle furnace at 400 c for 2 hrs. The ash was dissolved in 5ml conc. HCL, filtered and diluted to 100ml with distilled water. By taking a suitable aliquot the concentration of iron (III) was determined by the present method ICP-AES. The result for the analysis of the samples is given in Table No.3

RESULTS & DISCUSSION:
1. The absorption spectrum of the extract:
The wavelength range studied 400-700 nm.
Max is 613nm.
Molar absorptivity 5.048*103 mol^{-1} cm^{-1}.

1. Variations of parameter on the maximum absorption of extract:

Parameter:	Range max. absorption	Optimum used	Studied obtained
pH	1.0-3.0	1.9-2.5	2.2
Conc. range	0.0001-0.005	0.001-0005	0.001
Shaking time	0.5-5 mints	3-5 mints	3 mints

2. Effect of diluents on the max. absorption:
Diluents Studied: Benzene, Toluene, Xylene, Chloroform, Hexane and CCL_4 (0.005 mole dm^3/reagent solution). Absorption of extract found to decrease in the following order Toluene=Xylene> Benzene > Chloroform >CCL4> Hexane. The optimum solvent used is toluene because Cheaper than Xylene.

3. Stability of Extract:
The absorption of the extract remains constant for up to 45 minutes. Thereafter it decreases therefore spectrometric readings should be taken before 45 minutes.

5. Composition:
Log-log plot at optimum condition gave linear graph with slope 2.8, therefore, Composition of the complex in the ratio of 1:3.

6. Linearity:
Beers law obtain in the range 0.2-4.8 ppm of iron (III). Sandells Sensitivity =0.102 ug of Iron (III) cm^2

TABLE No. 2: Determination of iron (III) in foodstuffs & human hairs

Samples	Iron found (ug/g) Present method	R.S.D %	Iron obtained ICP-AES method(ug/g)
Rice	50.04	1.70	50.98
Wheat flour	21.58	2.67	21.11

Banana	35.53	1.59	35.08
Tomato	59,69	1.01	60.18
Human hair	94.46	1.27	95.39

TABLE No. 3: Determination of RSD percentage

Sample	Composition	Iron (III)	RSD %	
		Certified value, (mg)	Found, (mg)	
Nutrisan (Sandoz, India)	Ferrous fumarate I.P 20mg manganese chloride U.S.P 0.5mg, magnesium oxide I.P 1.0mg, Calcium gluconate 150 mg, zinc oxide I.P 2 mg	6.57	6.55	0.69 %
Supradyn (Roche, India)	Dried ferrous sulphate I.P 32.04 mg, copper sulphate I.P 3.39mg, Zinc sulphate I.P 0,25 mg, Calcium phosphate I.P 129mg, Manganese sulphate B.P 25.8 mg	11.78	11.7	1.36 %
Multivitamins iron minerals (Meyer Organics, India)	Ferrous fumarate I.P 55mg, Zinc sulphate I.P 20mg, Copper sulphate U.S.P 1mg, Manganese sulphate U.S.P 1mg, Magnesium Hydroxide B.P 30 mg	18.08	18.12	1.02 %

Table1. Effect of diverse ions on the determination of Iron (III).

Iron (III) taken = 50 ug.

Foreign ion	**Source**	**Tolerance limit (mg).**
Ni2+	$NiSO_4$. $7H_2O$	1.5
Cr6+	$K_2Cr_2O_7$	0.5
Pb2+	$Pb(NO_3)_2$	2.5
Mn2+	$MnSO_4$. $4H_2O$	10.0
Mo6+	$(NH_4)6\ Mo7O_{24}$	0.25
Zn2+	$ZnSO_4.7H_2O$	1.0
Mg2+	$MgSO_4$. $7H_2O$	7.5
Sr2+	$SrCl_2$. $6H_2O$	10.0
Ba2+	$BaCl_2.2H_2O$	7.5
Al3+	$Al(NO_3)_3$	2.5
Ca2+	$CaCl_2$	15.0
Cd2+	$CdSO_4.5H_2O$	1.0
Sn2+	$SnCl_2.2H_2O$	0.5
Cu2+	$CuSO_4.5H_2O$	0.05
Co2+	Co $(NO_3)_2$. 6H2O	0.10
Thiocyanate	NH_4SCN	5.0
Thiosulphate	$Na_2S_2O_3.2H_2O$	7.5
Thiourea	Thiourea	5.0
Tartarate	Tartaric acid	2.5
Oxalate	Oxalic acid	0.5
Citrate	Citric acid	0.5

Phosphate	Na_3PO_4	1.0
EDTA	EDTA disodium salt	0.05
Nitrite	$NaNO_2$	7.5
Nitrate	$NaNO_3$	7.5
Sulphate	Na_2SO_4	5.0
Chloride	NaCl	7.5
Fluoride	Sodium fluoride	1.0
Iodide	I_2	7.5

Conclusion:
The method is simple, rapid, sensitive & selective for the extractive spectrophotometric determination of iron (III) in biological, foodstuffs and pharmaceutical samples. This method is comparable with the ICP-AES method.

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