Spectrophotometric Determination of Iron with Xylenol Orange

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Summary: Iron has been determined spectrophotometrically with xylenol orange as a chromogenic reagent 1:1 complex formed in highly acidic medium is measured for its absorbance as 585 nm. In the present work, effect of buffer solution, reagent concentration, solute concentration, time, temperature and H_2O_2 has been studied. Interference of 23 cations and 5 anions is also reported.

Introduction

Iron is an extremely important constituent of the blood and tissues of the animal body. Most of the iron in the body is present as iron porphyrin or heme proteins which include hemoglobin in the blood, myoglobin and the heme enzymes [1]. Iron deficiency causes an anemia resulting from an inadequate dietary intake [2]. Various gravimetric [3,4] and volumetric [3,5] methods are known for the determination of iron but the chances of error are great especially in weighing. Small amounts of iron can be. determined spectrophotometrically using 1,10-phenanthroline [6,7], nitroso-R-salt [8], tiron [9], thiocyanate [7], acetyl acetone [10,11] and anthranilic acid [12]. In the present work xylenol orange is used to make a complex with iron and most of the experimental conditions are studied carefully and adjusted to suit better results.

Results and Discussion

The reaction between iron and xylenol orange was very quick. A purple coloured complex was immediately formed on the mixing of reactants and attained intensity when heated to 45°C and pH 1.5 (Fig. 2) λ_{max} was 585 nm (Fig. 3). The complex is stable for two hours.

Effect of buffer solution

Complex formation is pH dependent and the most stable complex was formed at pH 1.5 at which the reaction mixture showed maximum absorbance. 6 ml of sodium acetate buffer solution per 50 ml was quite sufficient to attain maximum colour intensity (Fig. 4).

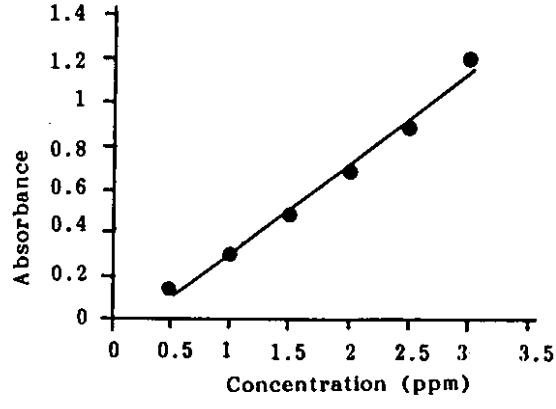

Fig. 1: Calibration curve.

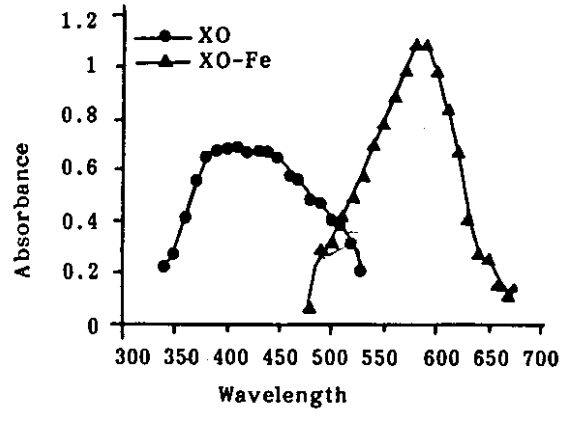

Fig. 2: max determination.

Effect of reagent concentration

The reaction was studied for xylenol orange concentration effect, different quantities of xylenol

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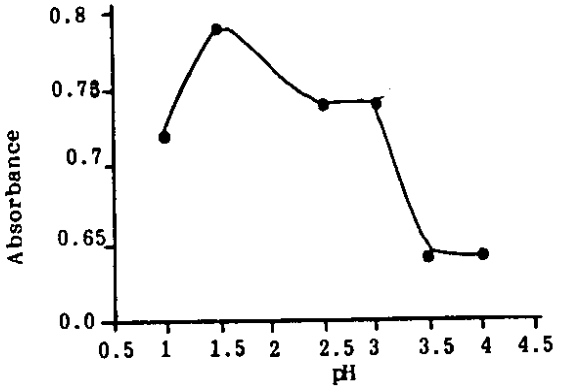


Fig. 3: Effect of pH.

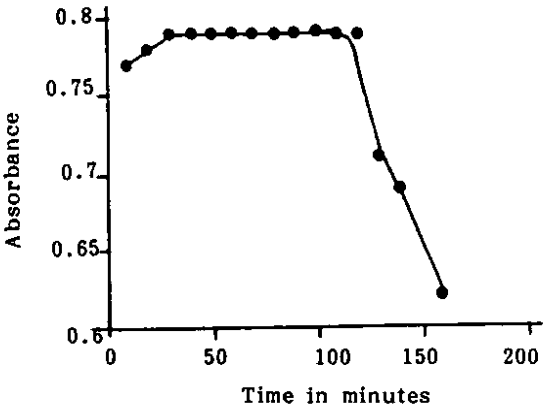


Fig.6: Effect of time.

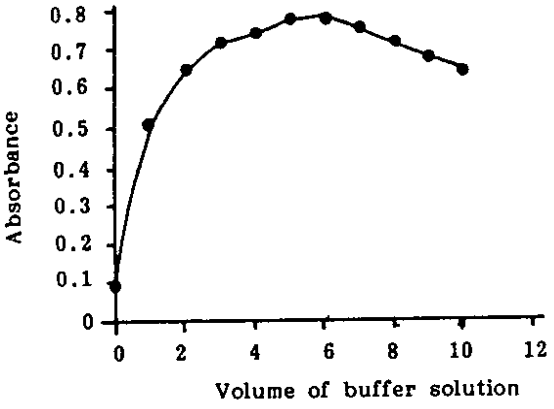


Fig. 4: Effect of volume of buffer solution.

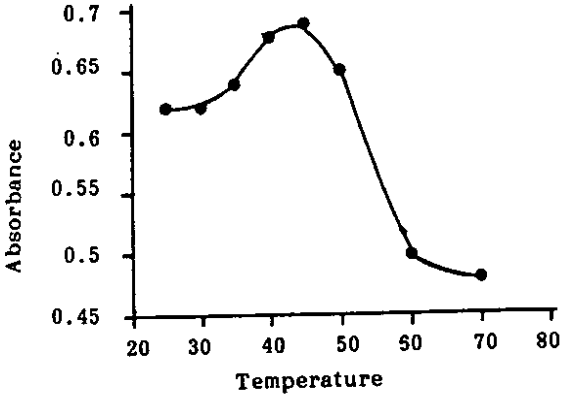


Fig. 7: Effect of temperature (°C)

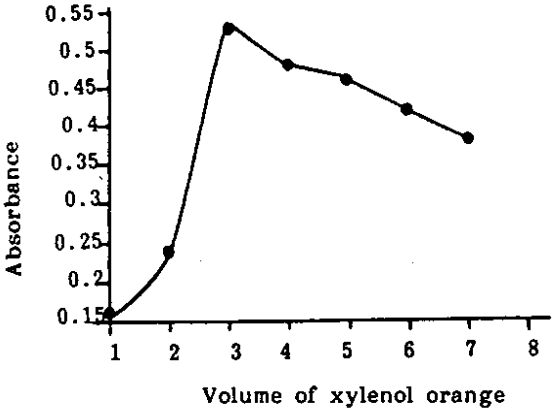


Fig. 5: Effect of reagent concentration at 585 n.m.

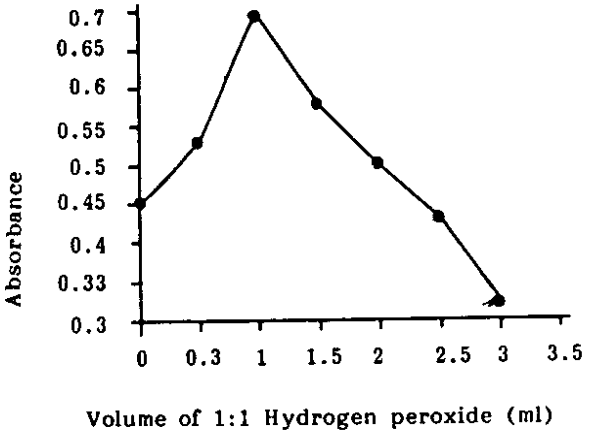


Fig. 8: Effect of volume of hydrogen peroxide (ml).

orange were used and it was observed that its 3 ml (0.05% w/v) per 50 ml solution were sufficient for complete complex formation upto 3 ppm (Fig. 5).

Effect of time and temperature

The complex is quit time dependent.The complex is stable for two hours. Beyond this lapse of time purple colour begins to fade and after 24 hours it completely disappears (Fig. 6). The effect of temperature was also noted and best results were obtained at 45°C (Fig. 7).

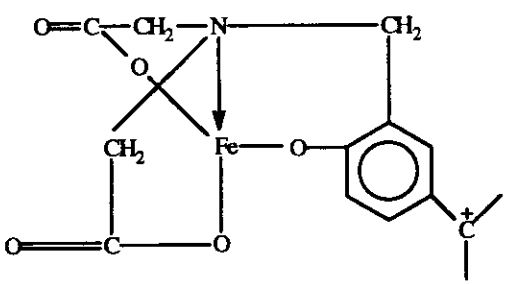
Effect of H_2O_2

Hydrogen peroxide was used as an oxidizing agent, required only in the case of Fe(II). Only 1 ml 1:1 H_2O_2 was sufficient for stable complex formation (Fig. 8)

Complex formation

Ferric iron forms a 1:1 complex with xylenol orange. Formation constant was found to be 5×10^5. It is believed that the phenolic, amine and carboxylic group of xylenol orange are involved in chelation.

[The possible structure of iron - xylenol orange complex]



Interference study

Interference of 23 cations and 5 anions in the determination of iron was studied. Table-1 and Table-2 list the changes in absorbance of the iron complex offered by various interferents when added in equal concentration. The study indicates that only bismith interfered positively and rest of the cations almost behaved normally. Among anions except sulphate ion (SO_4^{n}) all interfered positively.

Table-1: Effect of interferring cations on the determination of iron

Sr.No.	Absorbance of copper complex	Metal added	Absorbance with metal	Error ± %
1	0.69	Cr	0.67	2
2	0.69	Co	0.68	1
3	0.69	Ni	0.66	3
4	0.69	Tm	0.66	3
5	0.69	Al	0.67	2
6	0.69	Bi	0.92	23
7	0.69	Mo	0.69	0.00
8	0.69	Ag	0.70	1
9	0.69	Ba	0.68	1
10	0.69	Se	0.69	0.00
11	0.69	Pb	0.71	2
12	0.69	Zn	0.72	3
13	0.69	Na	0.68	1
14	0.69	Zr	0.70	1
15	0.69	Cu	0.69	0.00
16	0.69	Mn	0.66	3
17	0.69	V	0.73	4
18	0.69	K	0.69	6
19	0.69	Mg	0.63	2
20	0.69	Ca	0.67	2
21	0.69	Ca	0.68	1
22	0.69	Mg	0.68	1
23	0.69	Sr	0.67	2

Table-2: Effect of interferring anions on the determination of iron

Sr.No.	Absorbance of copper complex	Anion added	Absorbance with metal	Error ±%
1	0.69	Cl	0.80	0.00
2	0.69	SO_4	0.69	0.00
3	0.69	CH_3COO	0.80	
4	0.69	NO	0.79	
5	0.69	PO_4	0.80	

Table-3: Spectrophotometric determination of iron with xylenol orange at 1.5 pH and 585 nm wavelength.

No.	Taken µg/25 ml	Found µg/25 ml	Error
1	10	10	± 0.00
2	30	30	± 0.00
3	40	40	± 0.00
4	60	59.90	–0.10
5	70	70	±0.000

Experimental

Reagents

All reagents were of analytical grade or of comparable purity. Deionized water was used throughout. The stock iron solution was prepared by dissolving 4.975 g of $FeSO_4$ $7H_2O$ in water.

Before making up the volume 1 to 3 ml of concentrated sulphuric acid was added to stop the hydrolysis of $FeSO_4$. Then the solution was diluted to 1000 ml with water. Buffer solution was prepared by dissolving 0.266 g of sodium acetate in 1000 ml of 5 M acetic acid. Chromogenic reagent was prepared by dissolving 0.125 g of xylenol orange in water by adding 0.25 ml of concentrated hydrochloric acid and diluted to 250 ml. Hydrogen peroxide was 30%.

Apparatus

All absorbance measurements were made with spectronic-20 "BAUSCH and LOMB" using a paired 1 cm quartz cell. The pH meter was a Pye Unicam. All other volumetric glassware used was of A-garde calibration.

Procedure

A neutral sample solution was mixed thoroughly with 1 ml H_2O_2 (1:1), 5 ml of buffer solution (1.5 pH) and 3 ml of xylenol orange (0.05%) in a beaker and warmed upto 40-45°C. It was cooled and transferred to a 50 ml measuring flask and volume made upto the mark with water. The absorbance was measured at 585 nm against a reagent blank. The same sequence was repeated by taking different concentrations of iron (0.5 - 3.0 ppm) to prepare a calibration curve (Fig. 1).

Conclusion

The iron xylenol complex offers a higher sensitivity (ε = 34000) than all other common reagents used for the determination of iron. Iron in concentration as low as 0.5 to 3.0 ppm can be determined with appreciable accuracy and recovery is almost 100% (Table-3). It is expected that xylenol orange would find many applications where a highly sensitive reaction for iron is required. The method is simple and collective under experimental conditions mentioned above. It can be applied to analysed iron containing fruits and vegetables. As interference of the metal ions is minor, determination of iron can be achieved without any serious problem.

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