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SPECTROPHOTOMETRIC DETERMINATION OF MICRO AMOUNTS OF IRON(III)

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Abstract. A spectrophotometric method for the determination of iron in microgram quantities is described which is based on colour reaction between iron(III) and tannic acid having maximum absorption at 550 nm. Acetic acid sodium acetate (7:3) has been found a suitable buffer for the colour reaction. Maximum tolerable amounts of other metal ions have also been studied.

Many colorimetric and spectrophotometric methods[1–15] have been reported for the determination of iron but most of the methods are not free from interference and cannot be used selectively. Salicylate method,[1,3] is particularly useful when excess amount of material is available but it also requires rigid conditions. 5,7-Dibromooxine-*N*-oxide[6] as a spectrophotometric reagent for iron does not offer any advantages over other commonly used colorimetric reagents. In 2,4-dihydroxyacetophenone method,[9] colour develops slowly. The thiocyanate method[10] is unsatisfactory and colour is not stable in the presence of light. The *o*-phenanthroline procedure[12] requires rigid conditions to get reliable results. Bathophenanthroline methods[16,17] have been only used after reducing iron(III) to iron(II). Methods based on the use of quinoline carboxylic acid, 8-hydroxyquinoline, salicylic acid, indoferron, 2,2′-dipyridylglyoxime, are not free from interference and are not very sensitive methods.

A systematic study of colour reactions had led us to a very sensitive and accurate procedure for the determination of iron(III) even when present in small quantities. Tannic acid[18] has been used as a selective reagent which gives bluish black colour with iron(III) and has been reported as a spot test for this metal ion.[19,20] Tannic acid[21,22] has been determined colorimetrically with iron salts. Reference to the literature[23–25] indicates that no attempt has been made to use the reaction for the colorimetric or spectrophotometric determination of iron(III) and no detailed study of the reaction has been made from this viewpoint.

In the present paper, the colour reaction of iron(III) with tannic acid has been studied in detail and procedure has been developed for the spectrophotometric determination of iron in microgram quantities. Maximum tolerable amounts of other metal ions which do not interfere in the determination of iron(III) have been investigated. The colour reaction has maximum absorption at 550 nm and 0.5 μg as visual limit of identification.

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Experimental

Reagents. All the reagents were of analytical grade or comparable purity.

Standard solution of iron (III) was prepared by weight from ferric alum (B.D.H.) in distilled water containing dilute 5N H_2SO_4(dil). The solution is standardized by the stannous chloride reduction method.[26]

The suitable buffer used in the colour reaction of iron(III) and tannic acid was 0.2M CH_3COOH and 1M $CH_3COONa.3H_2O$.

The colour producing reagent 0.001M was prepared by dissolving 0.425 g tannic acid (E. Merck) in approximately 100 ml distilled water and warmed until tannic acid was completely dissolved, cooled and put in a measuring flask of 250-ml capacity and volume was made up to the mark.

Apparatus. All absorbance measurements for the determination of iron(III) were made with SP 600 Unicam spectrophotometer. The pH meter was a Pye Dynacap and graduated pipettes (Technico A BS. 1583 Ex. 20°C), accurate to ±0.005 ml were used for measuring solutions.

Procedure. To 10 ml acetic acid sodium acetate buffer solution (7:3), add 1 ml 0.001M of tannic acid solution. Then add 0.5–5 mg of iron(III) solution and make the final volume to 12 ml with distilled water. The pH of the solution will be between 4.06–4.44. The bluish black colour appears spontaneously, the intensity of which remains constant at room temperature (30–32°) for 2–15 min. The experiment is repeated with different volumes of iron solution and the absorbance is measured at 550 nm. The reaction obeys Beer's law. The typical calibration curve is given in Fig. 1.

Result and Discussion

Iron(III) produces stable colour with tannic acid having 0.5 μg as visual limit of identification. The colour solution has maximum absorbance at 550nm, all spectrophotometric determination were carried out at this wavelength.

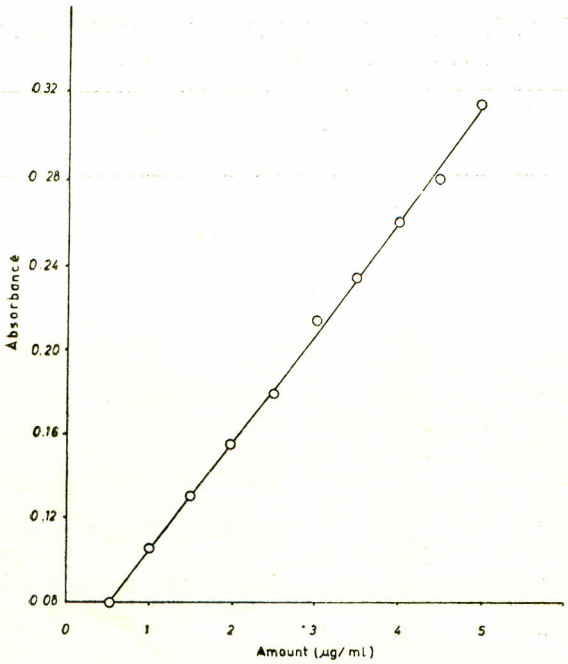

Fig. 1. Typical calibration curve for iron.

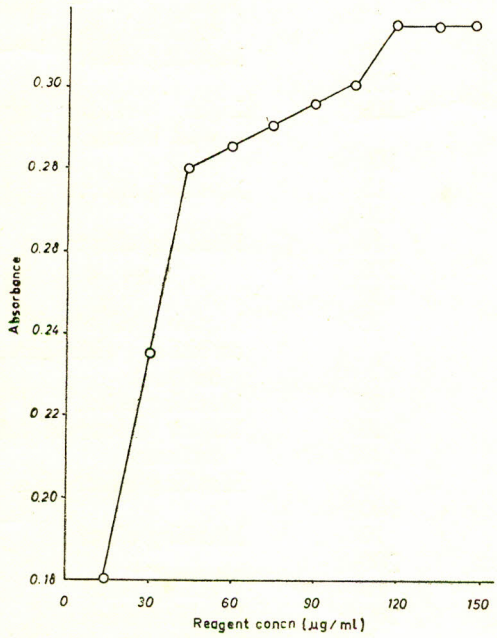

Fig. 2. Effect of reagent concentration on colour intensity.

The effects of reagent concentration, pH, time and temperature on the development of colour intensity has been studied.

The colour intensity remains stable if the concentration of the colour producing reagent (tannic acid) lies between 120–150 µg/ml (Fig. 2).

The effect of pH, temperature and time on colour intensity are given in Figs. 3, 4 and 5 respectively. Since the colour intensity is reproducible at a particular pH, temperature and after the same interval of time, the determination can be carried out at any set of conditions so long as they remain constant throughout the experiment.

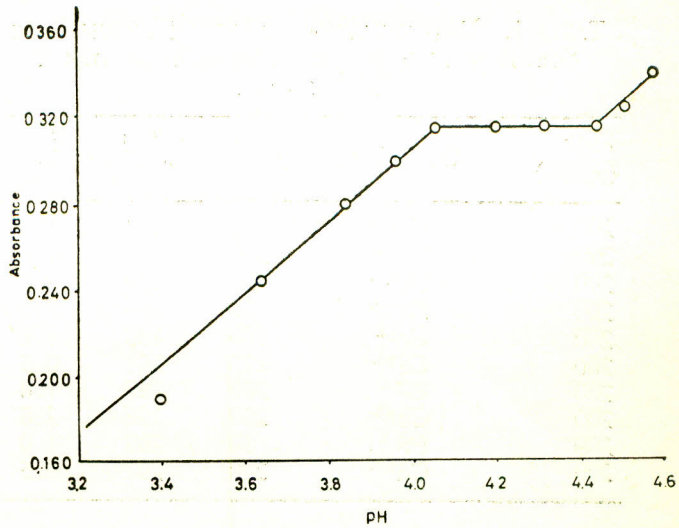

Fig. 3. Effect of pH on colour intensity.

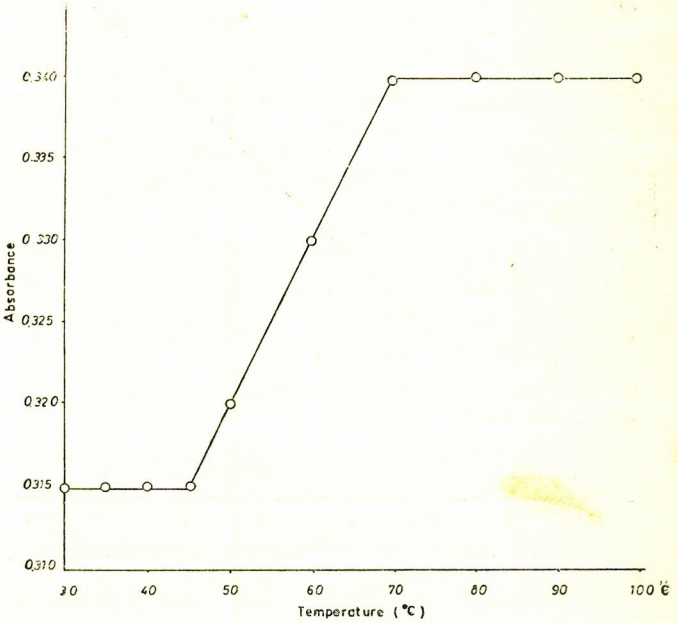

Fig. 4. Effect of temperature on colour intensity.

Since the colour intensity is fairly constant at room temperature (30–40°) and maximum between pH 4.06–4.44, all measurements were made on following conditions (Figs. 3 and 4).

The colour develops spontaneously after the addition of the colour producing reagent. The intensity of colour remains constant between 2–15 min as shown in Fig. 5.

The order of mixing of reagents is an important factor. The intensity of colour is fairly good if tannic acid is added before the addition of ferric solution.

The results for the determination of iron(III) from made-up solutions are shown in Table 1 which indicate the reliability of the method. The method can also be used for the determination of iron(III) in

TABLE 1. DETERMINATION OF IRON(III) FROM PURE SOLUTION AND IN THE PRESENCE OF OTHER METAL IONS.

Fe^{3+} (μg/ml)	Other ion	(μg/ml)	Fe^{3+} found	Error (%)
200	Co^{2+}	180	202	1.0
200	Ni^{2+}	200	200	0.0
200	Hg^{2+}	30	199	0.5
200	Au^{3+}	10	204	2.0
200	Cr^{3+}	100	198	1.0
200	Ca^{2+}	175	201	0.5
200	Bi^{3+}	200	198	1.0
200	Ce^{3+}	200	196	2.0
200	Pt^{4+}	10	202	1.0
200	Cd^{2+}	200	201	0.5

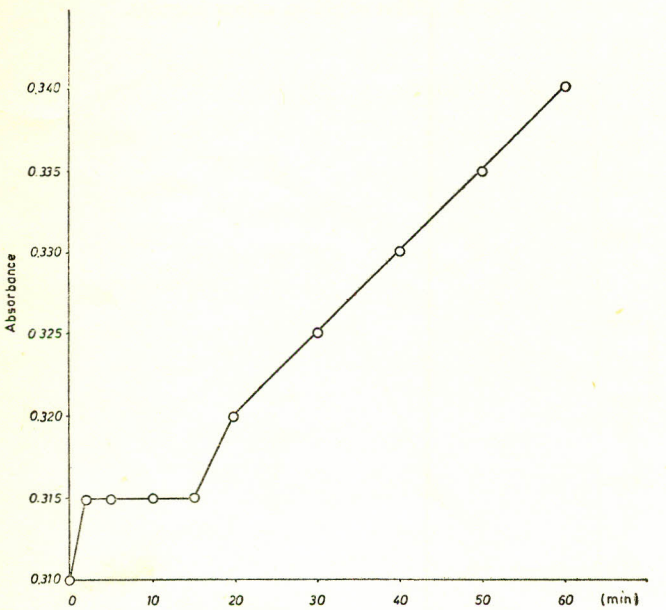

Fig. 5. Effect of time on development of colour intensity.

human blood and tissue. The method holds an excellent promise for the study of airborne particulates.

Maximum tolerable amounts of other metal ions, which do not interfere in the determination of iron(III) have been extensively studied and quantitative assessment is given in Table 2.

Conclusion

The colour reaction of tannic acid is selective for iron(III) and provides a very sensitive and accurate method for the determination of iron(III) in microgram quantities. Other cations do not interfere provided their amount do not exceed the tolerable amount (*cf.* Table 2). The method does not require rigid conditions and colour is stable for considerable time. This method has advantages over other methods because iron can be determined in very minute quantities. The mechanism of the colour reaction is not clear.

TABLE 2. MAXIMUM TOLERABLE AMOUNTS OF METAL IONS IN THE DETERMINATION OF IRON(III).

Metal ion	Not interfering* (μg/ml)	Remarks
NH^{4+}	150	Masking effect with slight turbidity
Na^{+}	1000	
K^{+}	800	
Ag^{+}	125	
Tl^{+}	125	
Be^{2+}	200	
Pd^{2+}	150	Masking effect
Zn^{2+}	200	
Mn^{2+}	175	
Hg^{2+}	30	Masking effect with pptn.
Co^{2+}	125	
Cu^{2+}	15	Masking effect with pptn.
Ni^{2+}	200	
Fe^{2+}	10	Slight ppt appears with increased amount
Cd^{2+}	200	
Ca^{2+}	175	
Uo^{2+}	250	
Sn^{2+}	10	Masking effect with increased amount
Pb^{2+}	10	Masking effect
Sr^{2+}	225	
Bi^{3+}	200	Light turbidity appears with increased amount
Cr^{3+}	100	
In^{3+}	15	Masking effect
Ce^{3+}	150	
Y^{3+}	10	Masking effect
La^{3+}	100	
Tl^{3+}	150	
Au^{3+}	10	Masking effect with slight turbidity with increased amount
Ti^{4+}	15	Masking effect
Pt^{4+}	10	Yellow colour of salt interferes
Th^{4+}	10	Masking effect with pptn.
Zr^{4+}	100	
Te^{4+}	10	Masking effect
Sn^{4+}	200	
Ce^{4+}	15	Masking effect
W^{6+}	200	Masking effect
V^{6+}	2	Interferes seriously due to the original colour of salt

*Solution containing 1μg/ml of iron(III) was taken and different amounts of various metal ions were added under experimental conditions. The amount of various metal ions is with respect to the amount of iron(III).

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