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NOTE

Catalytic Polarographic Analysis of Trace Amounts of Iron in Water Samples and Leafy Vegetables

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A catalytic polarographic method for the determination of trace quantities of iron is developed using antipyrine as ligand in 0.05 M potassium chloride medium. The procedure is extended for the analysis of water and agricultural samples for iron content.

In continuation of our earlier work on development of sensitive catalytic polarographic method[1,2] for iron, a new reagent, antipyrine, is now introduced as a complexing agent in potassium chloride medium. Iron(II) forms complexes with antipyrine and gives catalytic current at DME. This is advantageously exploited for the development of simple and sensitive method for the determination of iron at ppm levels without much of interference from other metal ions.

Polarographic studies on iron(II) with antipyrine in potassium chloride as supporting electrolyte revealed that in the presence of iron(II) even at 0.5 ppm concentration a large current is obtained, the peak potential of which is about −1.4 V *vs.* SCE. Iron or antipyrine in potassium chloride individually do not exhibit any wave (Fig. 1). A detailed experimental procedure on the polarographic behaviour of iron(II) in the presence of a complexing agent, antipyrine, in potassium chloride medium is mentioned below.

A d.c. recording polarograph, model Cl-25, Elico, India and Perkin-Elmer model 2380 AAS are used in the present study. All reagents used are of analytical grade and the solutions are prepared in double distilled water. The reagent, antipyrine, is obtained from Aldrich Chemicals, USA.

Effect of supporting electrolyte: The effect of supporting electrolyte on

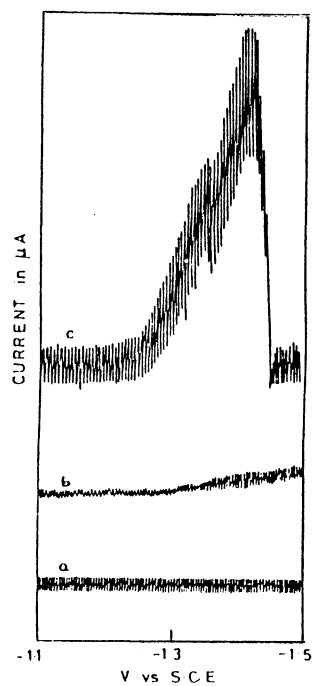

Fig. 1. Current-voltage curves of
(a) KCl (0.05 M) + antipyrine (0.6 M)
(b) KCl (0.05 M) + iron(II)(2.234 ppm)
(c) KCl (0.05 M) + iron(II)(2.234 ppm) + antipyrine (0.6 M)

catalytic current of iron(II) is studied by fixing the concentration of the metal ion and the reagent at 5.6 ppm and 0.6 M respectively and varying the concentration of potassium chloride supporting electrolyte from 0.025 to 0.25 M. The wave height increased with increase in the concentration of potassium chloride up to 0.05 M and with further increase, the height of the wave started decreasing. The shape and peak potential did not change at all concentrations of potassium chloride.

Effect of reagent concentration: At a fixed analytical concentration of iron(II) (5.6 ppm) and potoassium chloride (0.05 M) the effect of antipyrine on the peak current is studied. It is seen that the peak is not well manifested below 0.3 M and above 0.7 M concentration of antipyrine. Therefore 0.6 M (where the current is maximum) is maintained in all other studies.

Effect of height of the mercury column: The wave height of 5.6 ppm iron and 0.6 M antipyrine in 0.05 M potassium chloride remained constant with increase in mercury pressure confirming the catalytic nature of the wave.

Effect of surface active material: The concentration of Triton X-100 is varied from 0.001 to 0.004% keeping the concentrations of Fe(II) at 5.6 ppm, antipyrine at 0.6 M and potassium chloride at 0.05 M. It is seen from the nature of the catalytic waves that the maximum suppressor has no effect on the waves.

Effect of temperature: The wave height for the system iron(II) (5.6 ppm), antipyrine (0.6 M)in potassium chloride (0.05 M) increases with rise in temperature and the temperature coefficient is found to be more than 3% suggesting the catalytic nature.

Effect of metal ion concentration: The peak current increased proportionally with iron(II) in the concentration range 0.5 ppm to 7.0 ppm in the presence of 0.6 M antipyrine in 0.05 M potassium chloride medium (Fig. 2).

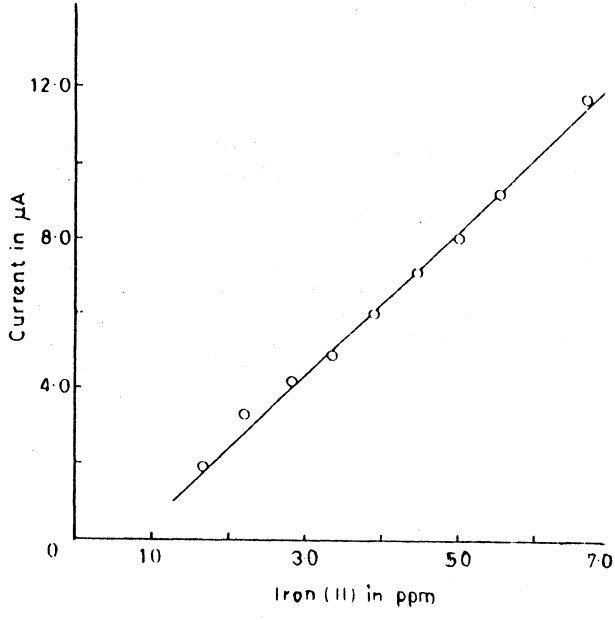

Fig. 2. Effect of iron(II) concentration on peak current

Effect of foreign ions on the polarographic behaviour of iron(II)-antipyrine system: The effect of various metal ions which are known to be commonly associated with iron, have been studied on the catalytic wave of iron(II)-antipyrine system in potassium chloride medium. The interference due to Co(II) and Cr(III) is masked by 2% aqueous sodium fluoride and Mn(II) and Ni(II) interfere even if present in small concentrations.

Applications: The method developed is applied for the determination of Fe(II) in drinking water and in leafy vegetables. Standard addition method is used for analysis.

(a) *Drinking water samples*: One litre of water samples collected from Kalyani Dam of Triupti town, Chittoor Distt. is preconcentrated to 100 mL. 5 mL of this is taken for analysis.

(b) *Leafy vegetables*: 5 g of leafy vegetables grown in nearby village of Tirupati town, Chittoor Distt. is collected, digested by dry ash method and brought into solution. 0.5 mL of this is taken for analysis.

Aliquots of the above solution are taken, polarographed using conditions developed as already mentioned. The results are given in Tables 1 and 2. The results obtained by this method are further supported by atomic absorption spectrophotometric method.

TABLE-1
DETERMINATION OF IRON(II) IN WATER SAMPLES, KALYANI DAM, TIRUPATI

Potassium chloride 0.05 M; Antipyrine 0.6 M

Sample	Fe(II), ppm		Fe(III) in the sample, μg/L	
	Added	Total found	Catalytic method	AAS method
a	0.5	0.90	8.0	
b	0.5	0.85	7.0	7.68
c	0.5	0.89	7.8	

TABLE-2
DETERMINATION OF IRON(II) IN LEAFY VEGETABLES, *AMARANTHUS VIRIDIS*

Potassium chloride 0.05 M; Antipyrine 0.6 M

Sample	Fe(II), ppm		Fe(II) ih the sample, μg/ g	
	Added	Total found	Catalytic method	AAS method
a	0.5	1.382	176.4	
b	0.5	1.402	180.4	179.6
c	0.5	1.366	173.2	

The method reported here for iron(II) estimation in trace levels is simple, sensitive and may be applied successfully for water samples and leafy vegtables.

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