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Simultaneous determination of Vanadium (V) and Iron (III) by kinetic spectrophotometric methods

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ABSTRACT

Vanadium (V) and iron (III) can be determined simultaneously at pH 2 and 298 ± 0.05 K spectrophotometrically at λ_{max} = 612 nm. A catalyst is a substance modifying the rate of a chemical reaction without affecting its equilibrium state. According to Mottola1 a more precise definition a catalyst is "a substance lowering the free activation energy without altering the equilibrium position".

The standard vanadium-iron alloy samples were procured from thermit alloys, Tirupathi. The standard solution of vanadium-iron alloy was prepared as per the procedure described in the literature. Iodate ion is a good oxidizing agent having an oxidizing potential of-1.6 as per the procedure per the following relation.

$$IO_3^- + 3\,H_2O \longrightarrow H_5IO_6 + H^+ + 2e \qquad E^0 + \ -1.6V$$
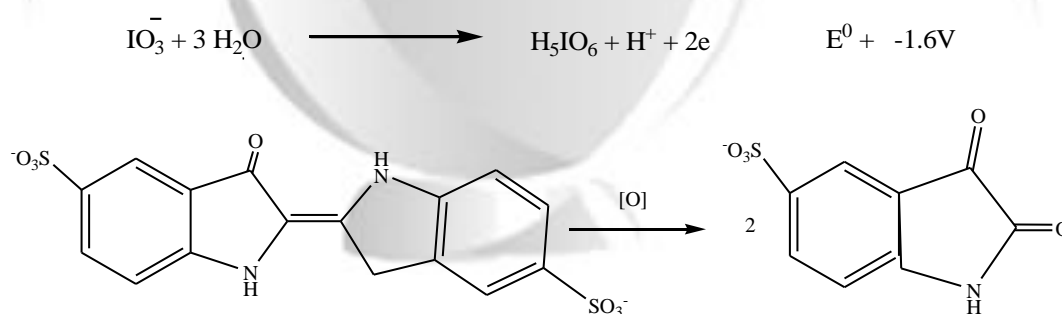
Keywords: Simultaneous determination; Vanadium (V); Iron (III); determination

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INTRODUCTION

The reaction proceeds through an induction period and then goes rapidly. The length of the induction period was found to be proportional to the concentration of iron (III) and independent of the concentration of vanadium (V). On the other hand the rate of the reaction was found to be related to the concentration of vanadium (V) and independent of the concentration of iron (III). Many differential kinetic methods have been proposed for the analysis of mixtures of closely related species without prior separation. This work presents a method which concentrates on the simultaneous determination of mixtures of Vanadium (V) and iron (III).

EXEPERIMENTAL

Apparatus are used UV-visible Shimazu amber programmed in kinetic mode was used for monitor the rate of reactiona a Systronic pH meter was used for the adjustment of pH A Shimadzu-TB-80 thermostal was used for the maintenance of constant temperature. The constant temperature was maintained by circulating water in the cell cabin of the spectrophotometer.

All reagents are AR grade and triply distilled water was used in all the experiments. A stock solution of vanadium (V) was prepared by dissolving appropriate amount (0.5985 g) of sodium metavanadate ($NaVO_3$, Rumali Hangeri) in 250ml of water and standardized potentimetrically. The concentration of vanadium in the solution was 1000 μg ml^{-1}. The dilute standard solutions needed were prepared by diluting the aliquots of stock solution.

Solution of iron (III) (1000 mg ml^{-1}) was prepared by dissolving 1.8085g of Fe $(NO_3)_3$ $9H_2O$ (s.d. fine chem. Mumbai) in 250 ml of water and standardized as per the accepted method titrimetrically. This solution was used for the preparation of working standard solutions. A stock solution of indigocarmine (IC) (1 $X10^{-4}$ m mol/ml) was prepared by dissolving 0.466 gm of AR sample (e-Merck) in 100 ml of water in a volumetric flask. The solution of potassium Iodate (0.033 M) was prepared by dissolving 1.77 g of KIO_3 (Merck India) in 250 ml of water in a volumetric flask.

Solution of ionic buffer solution of 3M sodium nitrate was prepared by dissolving appropriate amount of $NaNO_3$ (Merck India) in 100 ml of water. The stock solution (5000 μg ml^{-1}) of interfering ions were prepared by dissolving appropriate amounts of the salts in distilled water [1-20].

Fig - 3.1: Effect of indigocarmine concentration on the initial rate and induction period

$[V(V)] = 1.8 \ \mu g. \ ml^{-1}$ $[Fe(III)] = 6 \ \mu g. \ ml^{-1}$

$[IO_3^-] = 2 \times 10^{-3} \ mol.dm^{-3}$ $pH = 2 \pm 0.1$
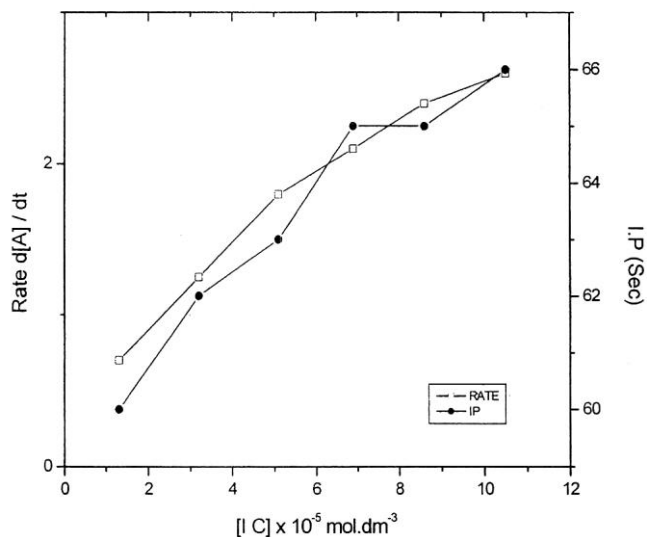

g – 3.2 : Effect of iodate concentration on the initial rate and induction period.

$[V(V)] = 2 \ \mu g.ml^{-1}$ $[Fe(III)] = 6 \ \mu g.ml^{-1}$

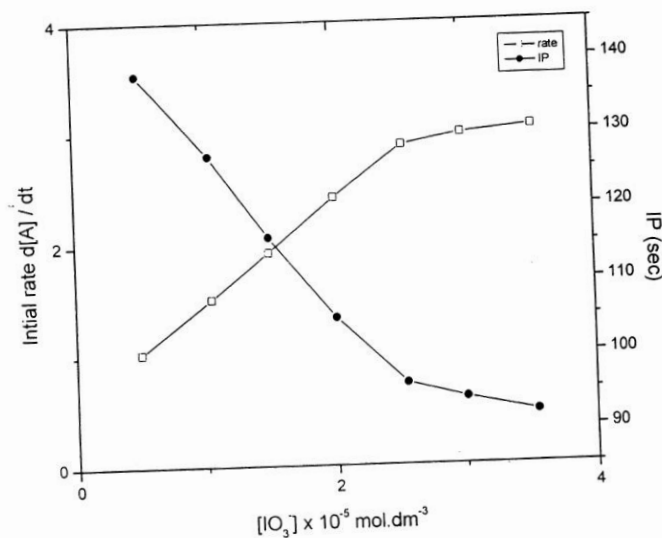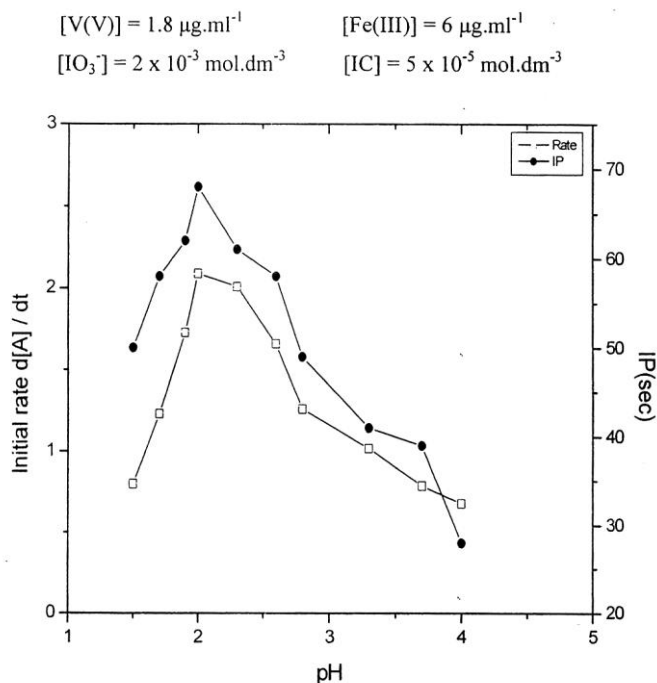$[IC] = 5 \times 10^{-5} \ mol.dm^{-3}$ $pH = 2 \pm 0.1.$

Fig – 3.3 : Effect of pH on the rate of reaction and induction period.

$[V(V)] = 1.8 \ \mu g.ml^{-1}$ $[Fe(III)] = 6 \ \mu g.ml^{-1}$

$[IO_3^-] = 2 \times 10^{-3} \ mol.dm^{-3}$ $[IC] = 5 \times 10^{-5} \ mol.dm^{-3}$

RESULTS AND DISCUSSION

The reaction was carried out under stoichiometric and non stochiometric proportions of IC and KIO$_3$. The reaction mixture consisted of IC, KIO$_3$, ionic buffer solution, HNO$_3$ for the maintenance and adjustment of pH. The experiment was repeated for the maintenance and adjustment of pH. The experiment was repeated for various concentration of IC, IO$_3$, catalyst and acid. All the solutions were placed in a thermostat at desired temperature for the establishment of thermal equilibrium.

The standard vanadium-iron alloy samples were procured from thermit alloys, Thirupathi. The standard solution of vanadium-iron alloy was prepared as per the procedure described in the literature. Iodate ion is a good oxidizing agent having an oxidizing potential of-1.6 as per the procedure per the following relation.
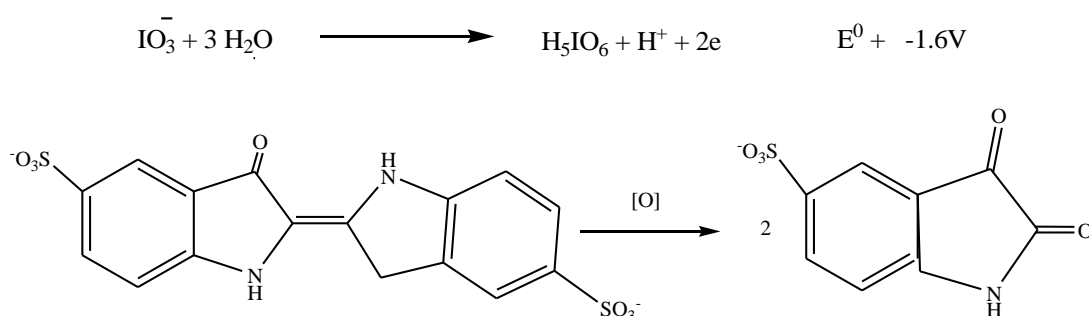
$$IO_3^- + 3\ H_2O \longrightarrow H_5IO_6 + H^+ + 2e \qquad E^0 + \ -1.6V$$

Fig – 3.4 : **Effect of temperature on the rate of reaction and induction period.**

$[V(V)] = 1.8 \ \mu g \ ml^{-1}$ $[Fe(III)] = 6 \ \mu g \ ml^{-1}$

$[IO_3^-] = 2 \times 10^{-3} mol.dm^{-3}$ $[IC] = 5 \times 10^{-3} mol.dm^{-3}$
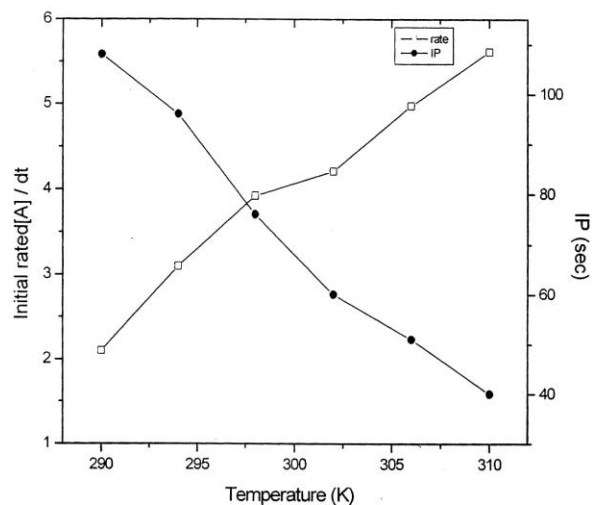
$pH = 2 \pm 0.1$

Table-1: Analysis of the synthetic mixtures of Vanadium (V) and iron (III).

Vanadium (V) In $\mu g.ml^{-1}$			Iron (III) In $\mu g.ml^{-1}$		
Range studied 0.25-2.5			Range studied 7.5-15		
Added	Found	Relative percentage Error	Added	Found	Relative Percentage Error
0.25	0.242	3.33	15.00	15.08	0.2
0.50	0.486	2.80	15.25	14028	0.3
0.74	0.736	0.54	13.55	13.60	0.3
0.98	0.978	0.20	12.40	12.45	0.4
1.25	1.260	0.80	12.00	12.09	0.75
1.48	1.510	1.30	11.10	11.14	0.36
1.76	1.790	1.70	10.00	10.08	0.8
2.02	2.080	2.90	9.10	9.12	0.3
2.25	2.290	1.77	8.50	8.45	0.5
2.50	2.530	1.20	7.50	7.4	1.3

Table-2 Analysis of V (V) and Fe (III) in Vansteel

Vanadium (V) In μg.ml^{-1}			Iron (III) In μg. ml^{-1}		
Actual	Found	Sd	Actual	Found	Sd
1.73	1.78	1.2	6.83	6.88	1.8
1.71	1.73	1.4	8.54	8.57	1.9
1.30	1.31	1.1	10.62	10.65	2.1
1.28	1.26	1.4	11.51	11.54	2.0

CONCLUSION

The new method is useful for the determination of Fe (III) and V (V) either independently or in case of mixtures. The intermediate species formed between V (V) and IC may be the main cause for increase in the velocity of the reaction, and the formation reactions seem to be promoted by the presence of Fe (III). The steps of the reaction are not clear because of the complexity of the reaction. The results obtained in the experiments are close to the expected values. Therefore this method can be employed for the analysis of real samples containing vanadium and iron.

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