Determination of Periodate in the Presence of Iodate

S.A. AL-TAMRAH
Chemistry Department, College of Science, King Saud University, Riyadh, Saudi Arabia.

(Received 13th March, 1982)

Summary: A method for the determination of periodate in the presence of iodate based on its oxidation of phthalophenone (reduced phenolphthalien) is described. As small as 1×10^{-4} M of the periodate can be determined by measuring the absorbance of the pink colour at 553 nm.

Introduction

Analytical methods for periodate are almost based on its oxidation of many organic and inorganic compounds which are usually simple if the periodate is present alone. In the presence of other oxidants the situation becomes more difficult.

Many methods are now available for the spectrophotometric determination of periodate based on its oxidation of many organic compounds[1-3]. A rapid and selective method has recently been developed based on the oxidation of soluble starch by periodate using phenol-4-aminoantipyrine[4]. The absorbance was measured at 500 nm. The limit of detection was 2 uM of periodate. Honda et al.[5] determined periodate using phenolphthalein and sodium bisulphite. The method was applied to the determination of unconsumed periodate after oxidation of some organic compounds.

Experimental

Apparatus

A Zeiss DMR 11, Recording Spectrophotometer, was used throughout this work.

Chemicals

Potassium periodate (KIO_4), potassium iodate (KIO_3) phenolphthalein ($C_{20}H_{14}O_4$), sodium hydroxide (NaOH) and zinc granules. These chemicals were all supplied by the British Drug House (BDH). The phthalophenone reagent was prepared by mixing 2 g of phenolphthalein, 10 g of sodium hydroxide, 5 g of zinc powder and 20 ml of distilled water. The mixture was refluxed for 2 hours until the pink colour disappeared completely and then filtered. The colourless solution was completed to 50 ml with distilled water and kept in a dark place (It is recommended to add some zinc granules in the phthalophenone solution to avoid oxidation). The reagent is diluted 10 times before use.

It was found that periodate reacts with phthalophenone in alkaline solution to give phenolphthalein which gives a pink colour, depending on the periodate concentration. Conditions affecting this reaction were studied which are:

1.Effect of time

The reaction between periodate and phthalophenone was studied at room temperature (25 °C) by measuring the absorbance of 1.2×10^{-4} M KIO_4 at 553 nm and found to be complete after 60 minutes as shown in Fig.(1).

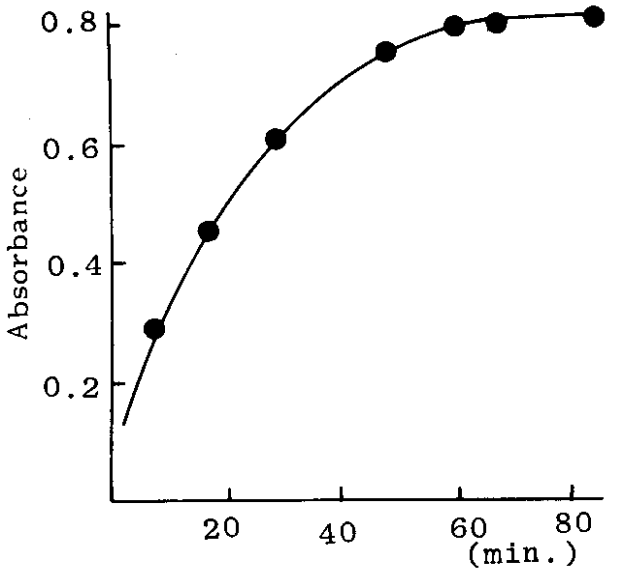

Fig.1.Effect of time on the reaction of periodate with the reagent at 25°C.

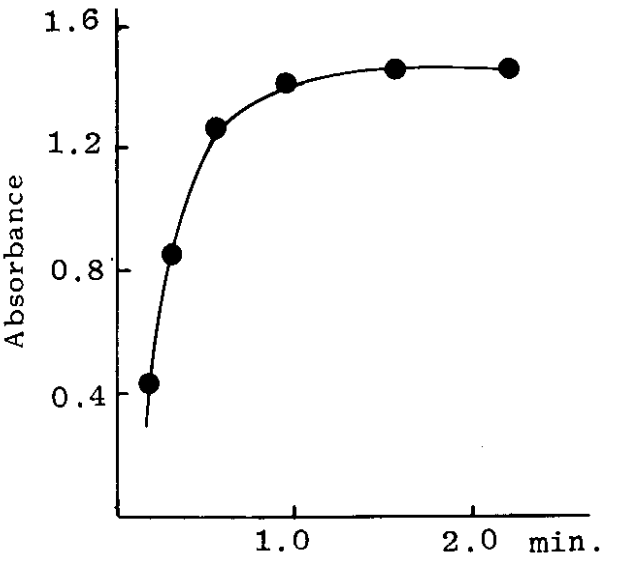

Fig.2.Effect of time on the reaction of periodate with the reagent at 100°C.

2.Effect of Temperature

As mentioned earlier, the reaction needs about one hour to complete at room temperature. In order to enhance the reaction which is advantageous to the analytical purpose, the effect of temperature was studied and one minute was found to be enough to complete the reaction, at 100°C,Fig.(2).

3.Effect of reagent

The concentration of reagent taken was found to be important. The colour produced depends to some extent on the amount of phthalophenone. This was studied by measuring the absorbance of 1.2×10^{-4} M KIO_4 using different amounts of phthalophenone. 0.4 ml of the reagent was found to be enough Fig.(3).

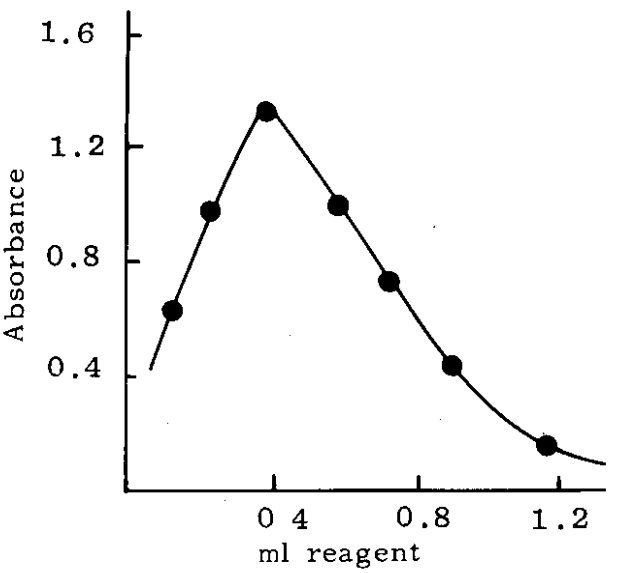

Fig.3.Effect of the reagent on the absorbance of 1.2×10^{-4} M KIO_4

General Procedure

In the direct procedure, 1 ml of the periodate sample is mixed with 0.4 ml of the reagent and made up to 50 ml with distilled water. The mixture is then immersed in boiling water for 1 minute and the absorbance measured at 553 nm.

Reaction of iodate with the reagent

No colour appears even after 24 hours when 5 ml of potassium iodate solution (1×10^{-3} M) was mixed with the reagent at room temperature. After heating faint colour appeared which was insensitive for analytical purposes. This observation made it possible to determine periodate in the presence of iodate at room temperature without any chemical separation. The effect of iodate on the absorbance of periodate was investigated by adding different amounts of potassium iodate from 0.01 - 0.09 M to the periodate solution (1.2×10^{-4} M) and reading the absorbance after one hour. No significant change in the absorbance was observed, Fig. (4).

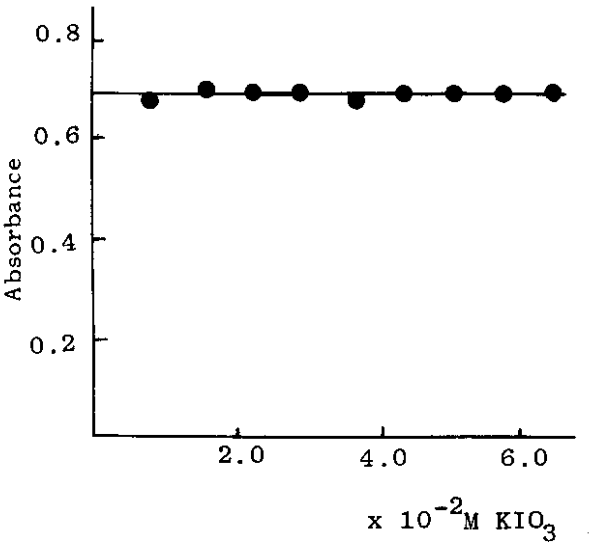

Fig.4.Effect of iodate on the absorbance of 1.2×10^{-4}M KIO_4

Results and Discussion

The oxidation of phthalophenone by periodate at room temperature (25°C) in alkaline solution yields a pink colour which can be measured for the spectrophotometric determination of periodate. The zinc ions formed after the reduction of phenolphthalein, i.e., the preparation of the reagent, react with periodate precipitating zinc periodate[6]. Therefore, no absorbance can be measured at the lower concentrations of periodate and the calibration graph did not pass through the origin. At higher temperature, better sensitivity was achieved, Fig. (5) becuase the high temperature increases the solubility of zinc periodate. However, the addition of 0.5 g EDTA to the reagent prior to the addition of periodate gave a straight calibration graph passing through the origin Fig. (5), because EDTA forms a complex with zinc leaving periodate to react with the reagent.

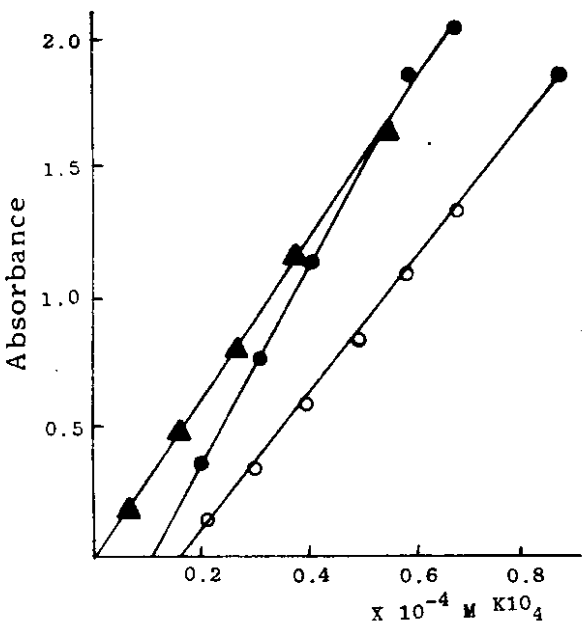

Fig.5.Calibration graph for potassium periodate

Iodate when tested under the same conditions did not oxidize the reagent which enabled the determination of periodate in the presence of iodate without

Table 1

Sample	Amount of KIO_3 added (M)	Amount of Taken	KIO_4 (M) Found	Accuracy
1	6×10^{-3}	1.4×10^{-4}	1.4×1^{-4}	100%
2	1.4×10^{-3}	1.0×10^{-4}	0.974×10^{-4}	97.5%
3	2.0×10^{-3}	0.8×10^{-4}	0.73×10^{-4}	91%
4	1.0×10^{-3}	0.6×10^{-4}	0.58×104	96.6%
5	0.5×10^{-3}	0.4×10^{-4}	0.39×10^{-4}	97.5%
6	3×10^{-3}	0.2×10^{-4}	0.195×10^{-4}	97.5%

any chemical separation.

The method was applied to the determination of periodate in the presence of iodate. Different samples were analysed and gave the results shown in Table 1.

The method is simple and rapid with reasonable sensitivity. For the determination of periodate alone only one minute is needed for each measurement.

Acknowledgement

The author thanks Prof. M. Afzal for his help and Mr. Y. Asha for technical assistance.

References

1. R.Fields and H.B.F.Dixon, *Biochem.J.*, ***108***(5), *883 (1968).*
2. E.M.Escarilla, P.F.Maloney and P.M.Maloney, *Anal.Chim.Acta*, ***45*** *199 (1969).*
3. G.Mahuzier, B.S.Kirkacharian and C.Harfouche Obeika, *Anal.Chim.Acta*, ***76*** *79 (1975).*
4. H.Gallati, *Anal.Chim.Acta*, ***96***, *311 (1978).*
5. S.Honda, K.Adachi, K.Kakehi, H.Yuki and K.Takiura, *Anal.Chim.Acta*, ***78*** *492 (1975).*
6. E.Kahane, *Bull.Soc.Chim., France, 15, 70 (1948).*