

Research Article

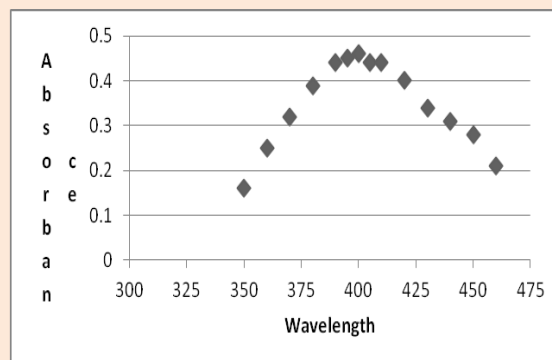
Spectrophotometric determination of anion periodate (IO_4^-) in synthetic mixtures and environmental water samples using tannic acid a reagent

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Simple, accurate spectrophotometric method has been proposed for determination of periodate anion with tannic acid as a reagent. This method was based on the formation of yellow colored complex between the tannic acid and anion. The optimum conditions for the determination were established. The method obeyed Beer's law in the range of 10-30 ppm. The molar absorptivity and Sandell's sensitivity values were found to be $4.65 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-2}$ and $2.0688 \mu\text{g cm}^{-2}$ respectively. The Co relation coefficient is found to be 0.998. The method had been successfully employed for the determination of periodate in the synthetic mixtures and environmental water samples.

Keywords: Periodate, Tannic acid, Synthetic mixtures environmental water samples, spectrophotometer.

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Introduction

Periodate (IO_4^-) is important oxidants [1, 2] which can oxidise a number of compounds. It has been used for the oxidation of many organic substrates. Periodate have been reported to play an important role in biochemical studies [3, 4]. It forms an integral part of many biological and biochemical reactions which take place in plants as well as animals.

The periodate anion (IO_4^-) in the form of various inorganic salts of periodate, attacks vicinal diols to cleave the carbon-carbon bond by oxidation reaction, leading to the formation of aldehydes. The relative quantities of periodate consumed in this process or aldehydes produced can be useful for quantitative determination as well as in determining the structure of organic compounds, especially polysaccharides. The quantitative determination of vicinal diols by periodate was first introduced by Malaprade who discovered that periodic acid can readily cleave and oxidize the α -glycol groups on mannitol [5]. Periodate oxidation has been widely used as a routine method for elucidation of structures in carbohydrates [6], quantitative determination of sugars in polysaccharides [7] and oxidation of organic [8] as well as inorganic ions [9]. Periodate-oxidised substrates also have biomedical [10] applications.

Periodate ion also exhibits anti-carcinogenic and anti-tumour activity due to its role as an oxidising agent. In vitro treatment of human peripheral blood lymphocytes (a form of white blood cells) with periodate has increased their cytotoxicity against leukaemia cells [11] while patients treated sodium periodate (IO_4^-) experienced more than 50 percent decrease in their tumour mass [12]. It has been observed that human blood cells when activated with periodate, express cytolytic activity for tumour cells [13]. Periodate anion in the form of periodic acid in Periodic acid-Schiff's (PAS) stain is commonly used to assist in the diagnosis of several medical conditions including different types of cancers [14] like leukaemia [15].

When coupled with other methods of analysis IO_4^- ion in the form of sodium periodate can be used for the determination of micro amounts of biologically interesting substances like reducing sugars which do not possess intensive spectroscopic groups in their molecules [16]. Industrial and commercial applications of periodate ion includes its use as a disinfectant [17]. Fungal and termite resistance of wood has been improved when it was reacted with periodic acid or sodium periodate [18].

A variety of methods have been developed for determination of IO_4^- which include chemiluminescence, fluorometry [19,20], infrared spectroscopy [21], flow injection determination by spectrofluorometric detection [22], flow-injection voltammetry [23] among others. Spectrophotometric determination of IO_4^- has been studied by employing different reagents like benzhydrazide [24], methylene green in the presence of iodide [25], salicyl aldehyde guanylhyazone [26], ferrocyanide [27], p-phenylenediamine, Azure B [28].

Experimental

Materials and Reagents

Apparatus: All glasswares used for the experimental purpose were made up of Pyrex or Borosil glass. The burette, pipette and standard flasks were calibrated by the method described by Vogel [29]. A single pan digital analytical balance of series CA-223, supplied by Contech, having sensitivity of 0.001 g was used for weighing chemicals, reagents and samples.

Instruments

pH meter: A digital pH meter model EQ-610, supplied by Equiptronics, an accuracy of ± 0.02 pH and resolution of 0.01 pH was used to measure the pH of the solutions. The pH meter was calibrated with standard buffer solutions of pH 7.0, 4.0 and 9.2.

Spectrophotometer: The absorption measurements were carried out on a spectrophotometer, model EQ-822, supplied by Equiptronics, Powai using 1 cm matched glass cells. The spectrophotometer was calibrated by measuring the absorption spectra of potassium chromate in potassium hydroxide solution and that of potassium permanganate in sulphuric acid solution [30].

Soxhlet extractor: Soxhlet extraction technique was employed for the extraction of real samples. For this purpose a soxhlet extractor.

Reagents

Periodate solution: The stock solution of periodate (1000 ppm) was prepared by first dissolving 0.3013 g of potassium periodate [KIO_3] in distilled water in the presence of concentrated sulphuric acid. The resulting clear solution was diluted to 250 cm^3 with distilled water in a standard volumetric flask. This stock solution was standardized titrimetrically by the procedure mentioned in the literature [31]. Appropriate dilution of stock solution was carried out to obtain solutions of desired concentration of periodate.

Reagent solution: 3% Tannic acid solution was prepared by dissolving weighed amount in distilled water and then diluting it up to the mark.

Buffer solution (pH 6.0): The buffer solution of pH 6 was prepared by dissolving 20 g of ammonium acetate in 0.8 cm^3 of glacial acetic acid and diluting to 100 cm^3 with distilled water.

Preparation of foreign ion solution: The solutions of various anions were prepared by dissolving their A.R. grade sodium, potassium or ammonium salts in distilled water. The solutions of cations were prepared by dissolving their A.R. grade salts in distilled water or dilute acids as required.

Procedure

To a series of 10 cm³ standard volumetric flasks containing varying amounts of periodate solution, 1 cm³ of 3% tannic acid solution was added. The pH of the solution was between 5.5- 6.5. The yellow colored complex was diluted upto the mark with distilled water and the absorbance for periodate was measured at 400 nm.

Spectrophotometric determination of periodate as per developed procedure

An aliquot of solution containing 10-30 µg/cm³ of periodate (IO₄⁻) was transferred into a 10 cm³ standard volumetric flask and 1 ml of 3% tannic acid was added at pH6. The absorbance for periodate was measured at 400nm.

Reference Method

To a series of 10 cm³ standard volumetric flasks containing 1-6 µg of standard periodate solution, 2 cm³ of citrate buffer solution of pH 6 and 1 cm³ of 0.15 M iodide solution were added. The solution was diluted upto the mark with distilled water and the absorbance for periodate was taken at 352 nm, during first three minutes after initiation of the reaction as per the procedure mentioned in the literature [30].

Effect of foreign ions

The effect of foreign ions on the sequential spectrophotometric determination of periodate was studied by adding a definite amount of the desired foreign ions to 5 µg/cm³ of periodate and treating the mixture as per the developed procedure. The tolerance limit was taken as the amount of ion causing an error of not more than ±2 % in the absorbance values.

Spectrophotometric determination of periodate in synthetic mixtures

A number of synthetic mixtures containing periodates with other commonly associated anions were prepared and treated as per the developed procedure. The treatment was carried out covering concentrations within the dynamic ranges of the species, using different concentrations of these anions.

Sequential spectrophotometric determination of periodate in environmental water samples

Water samples were collected from Koparkhairane lake in Koparkhairane city, India and Thane creek near B.N.Bandodkar College of Science, Thane, India. Each 50 cm³ filtered environmental water sample was boiled and reduced to 20 cm³. It was treated with 2 cm³ of conc. HNO₃, neutralised with dilute ammonia and again heated for 5 minutes. The resulting solution was cooled, filtered with Whatman filter paper no. 41 and diluted to 25 cm³ with distilled water in a standard volumetric flask. An aliquot of each sample was analysed for periodate. They tested negative. To appropriate aliquots of these samples, known amounts of IO₄⁻ was added and then analysed by the proposed procedure.

Results and discussion**Calibration curve**

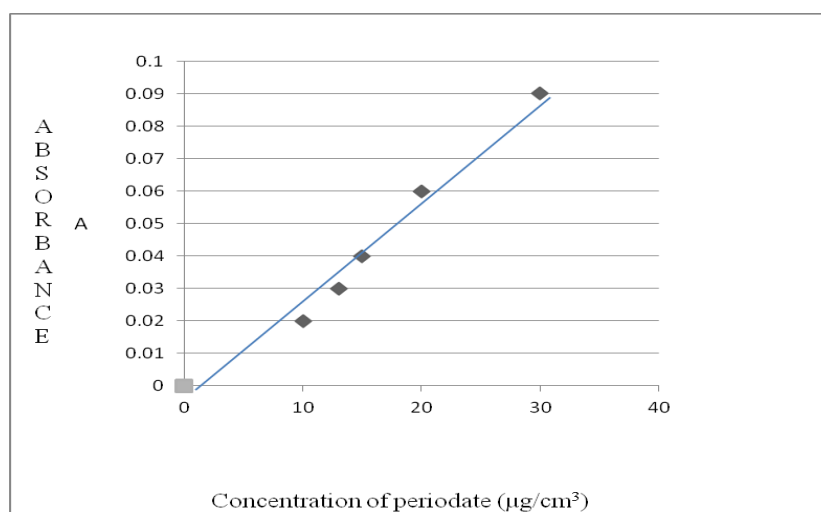
The adherence to Beer's law was studied by measuring the absorbance value of the series of solutions containing different concentrations of IO₄⁻. A linear calibration graph drawn between absorbance and periodate concentration indicates that IO₄⁻ may be determined in the range 10.0-30.0 µg/cm³ (Figure. 5.1). The Beer's law limit, molar absorptivity, Sandell's sensitivity, correlation coefficient, of the results are given in **Tables 1** and **2** and **Figure 1**.

Table 1 Calibration curve

Concentration of IO ₄ ⁻ (µg/cm ³)	Absorbance
10	0.02
13	0.03
15	0.04
20	0.06
30	0.09

Table 2 Analytical parameters pertaining to the proposed method

Parameter	Values
Absorption maxima	400 nm
Beer's range	10-30 ppm
Molar absorptivity	$4.65 \times 10^{-4} \text{ L mol}^{-1} \text{ cm}^{-2}$
Sandell's sensitivity	$2.0688 \mu\text{g cm}^{-2}$
Regression equation	$Y=0.0035X +0.0136$
Slope	0.0035
Intercept	0.0136
Co relation coefficient	0.998

**Figure 1** Calibration curve for periodate using Tannic acid as a reagent**Effect of foreign ions:**

In order to assess the possible applications of the proposed method, the effect of foreign ions on $5 \mu\text{g/cm}^3$ of periodate. The tolerance limit was considered to be the amount that caused a $\pm 2\%$ deviation in the absorbance value. The results reveal that various foreign ions can be tolerated at satisfactory levels.

Table 3 Effect of foreign ions on the determination of periodate($5 \mu\text{g/cm}^3$)

Anions	Tolerance Limit ($\mu\text{g/cm}^3$)	Cations	Tolerance Limit ($\mu\text{g/cm}^3$)
NO_3^-	5000	Cu^{3+}	500
NO_2^-	5000	Ni^{2+}	500
CH_3COO^-	2000	Ca^{2+}	100
SO_3^-	2000	Hg^{2+}	100
Br^-	5000	Th^{4+}	100
Cl^-	5000	Mg^{2+}	100
I^-	2000	Ba^{2+}	100
SO_4^{2-}	2000	Cd^{2+}	100
SCN^-	2000	Sn^{2+}	100
BrO_3^-	5000	$^c\text{Fe}^{2+}$	100
$\text{C}_2\text{O}_4^{2-}$	1000	$^d\text{Ce}^{2+}$	Below 100
WO_4^{2-}	1000	$^d\text{Sr}^{3+}$	Below 100

EDTA	500	Al ³⁺	Below 100
IO ₃ ⁻	1000	^c Zn ²⁺	Below 100
Borax	1000	Pb ²⁺	Below 100
S ₂ O ₃ ²⁻	1000	^d Zr ⁴⁺	Below 100
Citrate	100	^d Mn ²⁺	Below 100
Tartarate	100	Fe ³⁺	Below 100
^a S ₂ O ₈ ²⁻	100		
^b PO ₄ ³⁻	100		
IO ₄ ⁻	Below 100		

a- Masked by 5000 µg/cm³ CH₃COO⁻, b- Removed gravimetrically, c- Masked by 0.5% thiourea, d- Masked by 5000 µg/cm³ C₂O₄²⁻, e- Removed gravimetrically

Applications

The proposed method under the already established optimum conditions was satisfactorily applied for the determination of IO₄⁻ in synthetic mixtures and environmental water samples. The results are highly reproducible and the recoveries for the different added concentrations of IO₄⁻ are in the range of 93-105%. The results show that the proposed method is suitable for determination of trace amounts of IO₄⁻ in synthetic mixtures and environmental water samples **Tables 4** and **5**.

Table 4 Determination of IO₄⁻ in synthetic mixtures

Synthetic Mixture	Proposed Method (IO ₄ ⁻)			Reference Method (IO ₄ ⁻)[32]	
	IO ₄ ⁻ added (µg/cm ³)	^a IO ₄ ⁻ found (µg/cm ³)	% Recovery	^a IO ₄ ⁻ found (µg/cm ³)	% Recovery
^b 1.	4	4.1± 0.058	102.50	4.07± 0.012	101.75
^c 2.	2	2.1± 0.058	105.00	2.23± 0.115	111.5
^d 3.	6	5.9± 0.12	98.33	5.87± 0.073	97.8

a - Mean± standard deviation (n=3), b - IO₄⁻ (4 µg) + C₂O₄²⁻ (5 µg) + SO₃²⁻ (10 µg), c - IO₄⁻ (2 µg) + WO₄²⁻ (5 µg) + SCN⁻ (10 µg), d - IO₄⁻ (6 µg) + I⁻ (5 µg) + SO₄²⁻ (6 µg)

Table 5 Determination of IO₄⁻ and MoO₄²⁻ in environmental water samples

^b Water sample	Proposed Method (IO ₄ ⁻)			Reference Method (IO ₄ ⁻)	
	IO ₄ ⁻ added (µg/cm ³)	^a IO ₄ ⁻ found (µg/cm ³)	% Recovery	^a IO ₄ ⁻ found (µg/cm ³)	% Recovery
^c 1.	3	3.23± 0.152	107.6	2.86±0.115	95.33
^d 2.	2	2.1± 0.058	105.0	2.13±0.115	106.50

a - Mean± standard deviation (n=3), b - Gave no test for periodate(IO₄²⁻), c - Koparkhairane lake water sample, d - Thane creek water sample

Conclusion

This method describes a simple, rapid, sensitive spectrophotometric procedure for the sequential determination of periodate in water samples. It provides a good reproducibility. The proposed method has the advantage of enabling a wide range of determination without the need for extraction or heating, the reagents used in the proposed method are readily available in most laboratories. The results of this study clearly show the potential and versatility of this method, which could be applied to monitoring of periodate spectrophotometrically in water samples.

Acknowledgements

The author is thanking to the University grants commission for financial assistance. The support and facilities provided by Vidya Prasark Mandal, Thane is gratefully acknowledged. My sincere thanks to the principal, Dr. (Mrs.) M.K. Pejaver, B.N. Bhandodkar College of science, for encouragement, support and for providing all facilities to conduct work. I am thankful to the Principal, V.E.S. college of Arts, Science and college for her kind support.

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Publication History

Received 15th May 2015
Revised 24th May 2015
Accepted 10th Jun 2015
Online 30th Jun 2015