

Research Article

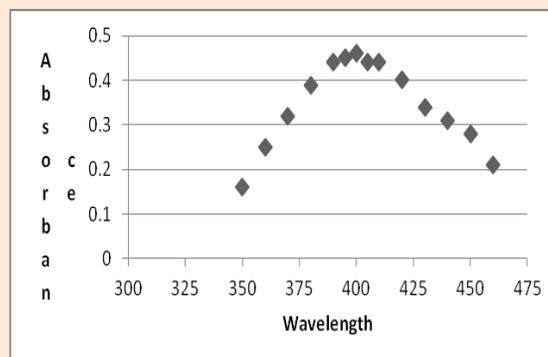
Spectrophotometric determination of anion periodate (IO_4^-) in synthetic mixtures and environmental water samples using tannic acid a reagent

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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 Correlation 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**

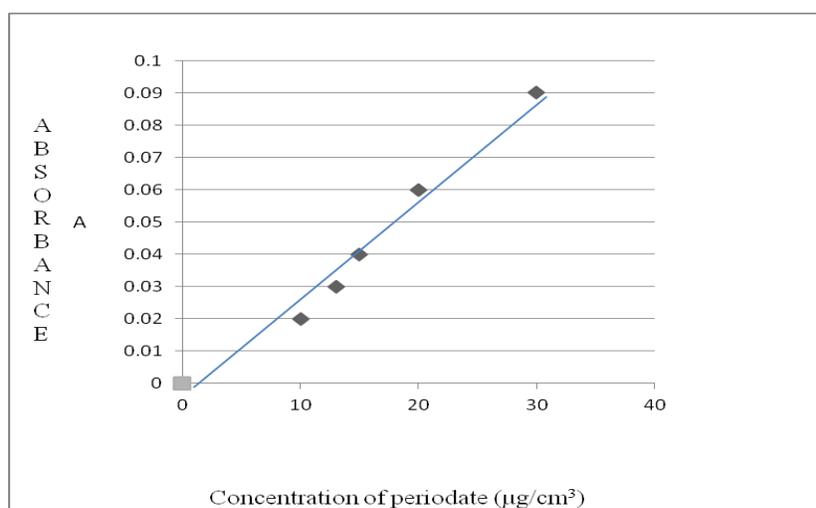
The adherence to Beer's law was studied by measuring the absorbance value of the series of solutions containing different concentrations of IO₄⁻. A linear calibration graph drawn between absorbance and periodate concentration indicates that IO₄⁻ may be determined in the range 10.0-30.0 µg/cm³ (Figure. 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.

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References

- [1] Gaikwad A, Silva M, Pérez-Bendito D. Sensitive determination of periodate and tartaric acid by stopped-flow chemiluminescence spectrometry. *Analyst* **1994**; 119:1819-1824.
- [2] Ribeiro PRS, Pezza L, Pezza HR. A simple spectrophotometric method for the determination of captopril in pharmaceutical preparations using ammonium molybdate. *Eclat Quím* **2010**; 35(3):179-88.
- [3] Kobayashi M, Ichishima E. Application of Periodate Oxidized Glucans to Biochemical Reactions. *J Carbohydr Chem* **1991**;10(4):635-44.
- [4] Williams RJP, Fraústo da Silva JJR. The Involvement of Molybdenum in Life. *Biochem Biophys Res Commun* **2002**;292(2):293-9.
- [5] Malaprade L. Oxidation of some polyalcohols by periodic acid-applications. *Compt Rend* **1928**; 186:382-4.
- [6] Hamilton JK, Smith F. Reduction of the products of periodate oxidation of carbohydrates. III. The constitution of Amylopectin. *J Am Chem Soc* **1956**;78(22):5910-12.
- [7] Bahl OP, Hullar TL, Smith F. Reduction of the products of periodate oxidation of carbohydrates. XIII. Determination of sugars in polysaccharides oxidized by periodate. *J Org Chem* **1964**; 29(5):1076-78.
- [8] Halsall TG, Hirst EL, Jones JKN. Oxidation of carbohydrates by the periodate ion. *J Chem Soc* **1947**; 1427-32.
- [9] Hiremath SC, Tuwar SM, Nandibewoor ST. Oxidation of chromium (III) by periodate in aqueous alkaline medium - A kinetic study. *Indian J Chem Sec A* **1999**;38A(1):61-4.
- [10] Balakrishnan B, Lesieur S, Labarre D, Jayakrishnan A. Periodate oxidation of sodium alginate in water and in ethanol-water mixture: a comparative study. *Carbohydr Res* **2005**;340(7):1425-9
- [11] Noda I, Fujieda S, Saito H, Saito T, Otsubo T, Yagita M. Enhancement of cytolytic activity of human peripheral blood lymphocytes by sodium periodate (IO₄) possible involvement of protein kinase C. *Int J Immunopharmacol* **1998**;20(1-3):15-27.
- [12] Wang J, Walle A, Gordon B, Novogrodsky A, Suthanthiran M, Rubin AL, Morrison H, Silver RT, Stenzel KH. Adoptive immunotherapy for stage IV renal cell carcinoma: a novel protocol utilizing periodate and interleukin-2-activated autologous leukocytes and continuous infusions of low-dose interleukin-2. *Am J Med* **1987**;83(6):1016-23.
- [13] Wang J, Suthanthiran M, Walle A, Lagman M, Schwartz R, Murthi V, Novogrodsky A, Stenzel KH. Anti-tumor properties of lymphocytes activated by the oxidizing mitogens. *J Immunol* **1986**;136(12):4735-9.
- [14] Lee AF, Rees H, Owen DA, Huntsman DG. Periodic Acid-Schiff is superior to hematoxylin and eosin for screening prophylactic gastrectomies from CDH1 mutation carriers. *Am J Surg Pathol* **2010**;34(7):1007-13.
- [15] Pattari S, Varma N, Garewal G. Combined staining approach with myeloperoxidase and Periodic acid-Schiff's on routine bone marrow aspiration smears. *Internet J Hematol* **2002**;1:1.
- [16] Maeda M, Tsuji A. Chemiluminescence flow injection analysis of biological compounds based on reaction with lucigenin. *Anal Sci* **1986**;2:183-6.

- [17] Okochi M, Yokokawa H, Lim TK, Taguchi T, Takahashi H, Yokouchi H, Kaiho T, Sakuma A, Matsunaga T. Disinfection of microorganisms by use of electrochemically regenerated periodate. *Appl Environ Microbiol* **2005**;71(10):6410-3.
- [18] Chen GC, Rowell RM. Fungal and termite resistance of wood reacted with periodic acid or sodium periodate. *Wood Fiber Sci* **1989**;21(2):163-8
- [19] Jie N, Yang D, Zhang Q, Yang J, Song Z. Fluorometric determination of periodate with thiamine and its application to the determination of ethylene glycol and glycerol. *Anal Chim Acta* **1998**;359(1-2):87-92.
- [20] Jie N, Sun X, Yang J, Zhang Q, Liu Z, Song Z. A study on the reaction of periodate with paracetamol and its application. *Anal Lett* **1997**;30(13):2441-50.
- [21] Al-Kayssi M, Magee RJ. The determination of periodate by infrared spectroscopy. *Anal Chim Acta* **1963**;28:176-8.
- [22] Ensafi AA, Dehahgi GB. Spectrofluorimetric flow injection determination of trace amounts of periodate. *Spectrochim Acta A Mol Biomol Spectrosc* **2001**;57(9):1739-43.
- [23] Gökçel HI, Nişli G. Static and flow-injection voltammetric determination of periodate by reduction at a rotating platinum wire electrode. *Anal Chim Acta* **1994**;292(1-2):99-105.
- [24] Escarrilla AM, Maloney PF, Maloney PM. Colorimetric determination of periodate with benzhydrazide. *Anal Chim Acta* **1969**;45(1):199-201.
- [25] Bagherian G, Chamjangali MA, Goudarzi N, Namazi N. Selective spectrophotometric determination of periodate based on its reaction with methylene green and its application to indirect determination of ethylene glycol and glycerol. *Spectrochim Acta A* **2010**;76:29-32.
- [26] Nevado JJB, González PV. Spectrophotometric determination of periodate with salicylaldehyde guanylhydrazone. Indirect determination of some organic compounds using the malaprade reaction. *Analyst* **1989**;114:243-4.
- [27] Rahim SA, Bashir WA. Spectrophotometric determination of iodate, periodate, and persulfate in aqueous solution. *Microchem J* **1984**;29:87-91
- [28] Hareez AK, Bashir WA. p-Phenylenediamine as a chromogen for iodate and periodate in aqueous solution. *Microchem J* **1985**;31:375-7
- [29] Vogel AI. *Textbook of Quantitative Inorganic analysis*. 3rd ed. ELBS and Longman Press; 1978.
- [30] Sandell EB. *Colorimetric Determination of Traces of metals*. 3rd ed. New York (NY): Inter science Publishers; 1965.
- [31] Narayana B, Cherian T. A facile spectrophotometric method for the determination of periodate using azure B. *J Braz Chem Soc* **2005**;16,5:978-81
- [32] Afkhami A, Madrakian T, Zarei AR. Spectrophotometric determination of periodate, iodate and bromated mixtures based on their reactions with iodide. *Anal Sci* **2001**;17:1199-202

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