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Spectrophotometric Determination of Iodine Species in Table Salt, Pharmaceutical Preparations and Sea Water

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Abstract

A sensitive spectrophotometric method for the determination of iodine species like iodide, iodine, iodate and periodate is described. The method involves the oxidation of hydroxylamine to nitrite with iodate under acidic condition. The formed nitrite is determined based on the diazo coupling reaction between p-nitroaniline and N-(1-naphthyl) ethylenediammine dihydrochloride [NEDA]. The method obeys Beer's law in the concentration range 1-15 µg of iodate in an overall aqueous volume of 10 mL at 545 nm and the color is stable for 3h. The relative standard deviation is 1.7 % (n=10) at 12 µg of iodate. The molar absorptivity is calculated to be 8.33 x 10^4 L $mol^{-1}cm^{-1}$ with a correlation coefficient of 0.9998.

The developed method can be applied to samples containing iodine and iodide by oxidation to iodate using bromine solution under acidic condition. Periodate is determined by prereduction to iodate using ethylene glycol under acidic condition. The optimum experimental reaction conditions are evaluated. The effect of interfering ions on the determination of iodate is described. The proposed method has been successfully applied to the determination of iodide and iodate in salt samples, iodine in pharmaceutical preparations and added periodate in sea water sample.

Keywords:

Spectrophotometry; iodate; nitrite; p-nitroanailine; NEDA; diazocoupling

1. Introduction

Iodine is an essential micronutrient in human growth and is very important for iodine supplementation metabolism. The deficiency of iodine can result in brain damage, mental retardation, and endemic goitre. Deficiency of iodine causes serious delay in neurological development. On the other hand, an excess of iodine or iodide can cause goiter and hypothyroidism [1] Table salts are iodized with iodate or iodide to serve as a source of iodine. The iodized salt is recognized as the method of choice and the most successful strategy for the prevention of iodine deficiency disorders. The recommended concentration of the iodate in salts is 40 ppm [2]. Iodine is an effective germicide [3] for a wide range of microorganisms. Iodine is often used in conjunction with complexing nonionic surfactants or polymers (iodophors) in disinfectants that are used in dairies, laboratories and food processing plants. In addition to this iodine finds applications as dietary supplements, catalysts, pharmaceutical preparations, stabilizers and in photography. Iodine is also used in the production of motor fuels, high purity metals and in cloud seeding. The varied applications of iodine have made the determination of iodine very important.

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Several methods of iodine determination have been proposed, including selective electrodes [5], X-rayfluorescence (XRF) [6], inductively coupled plasma mass spectrometry (ICP-MS)[7,8]. Trace levels of iodide have been determined by the catalytic effect usually on the cerium (IV)-arsenic (III) reaction [9,10]. Other reactions catalyzed or inhibited by iodide have also been described [11,12]. Precipitation as silver iodide, dissolution in potassium cyanide, and determination of silver in the complex by atomic absorption spectrometry has been used for the indirect determination of iodide in a flow system [13]. Sensitive extraction photometric methods based on ion-pair formation with methylene blue [14] or with brilliant green [15] are well known. Recently methods based on the oxidation of leuco xylene cyanol FF [16], thionin [17] and Varian blue [18] with iodate are reported. Several spectrophotometric methods have been reported for the determination of periodate and iodate [19-23]. El-Shahawi [24] used the ion-associate of periodate with amiloride hydrochloride for simultaneous spectrophotometric determination of periodate and iodate by liquid-liquid extraction. Most of the reported methods are not sensitive enough or require complicated and expensive instruments. The need for a sensitive, simple and reliable method for the determination of iodine species is recognized.

This paper describes a spectrophotometric method for the determination of iodine species like iodide, iodate and periodate. The method involves the oxidation of hydroxylamine to nitrite with a known amount of iodate under acidic condition. The formed nitrite is determined based on the diazo coupling reaction between p-nitroaniline and N-(1-naphthyl) ethylenediammine dihydrochloride [NEDA]. The method obeys Beer's law in the concentration range 0-15µg of iodate in an overall aqueous volume of 10 mL at 545 nm.

The developed method can be applied directly to samples containing iodate, iodide and iodine after oxidation to iodate with a known excess of bromine solution and periodate after prereduction to iodate with ethylene glycol under acidic condition. The proposed method has been successfully applied to the determination of iodide and iodate in salt samples and iodine in pharmaceutical preparations.

2. Experimental

2.1Apparatus

All absorbance measurements were made using Elico SL 177 scanning spectrophotometer with 1 cm glass cells.

2.2 Reagents

All reagents were of analytical reagent grade and distilled water was used for preparing all solutions. A stock solution of iodate (1000 µg mL^{-1}) was prepared by dissolving 0.1223 g of potassium iodate in 100 mL of distilled water. A working solution of 3 µg mL^{-1} was prepared by suitable dilution. *p*-nitroaniline (0.05 %) was prepared by dissolving 0.05 g of *p*-nitroaniline in 2 mol L^{-1} sulphuric acid. N-(1-naphthyl) ethylenediammine dihydro-chloride NEDA (0.1%) was prepared by dissolving 0.1 g of NEDA in 100 mL of distilled water. Hydroxylamine solution (500 µg mL^{-1}) was prepared by dissolving 0.1053 g of hydroxylammonium chloride in 100 mL of distilled water. Sulphuric acid (2 mol L^{-1}) was prepared by adding 27.8 mL of concentrated sulphuric acid (Sp.Gravity 1.8) to 150 mL of water and diluting to 250 mL with water. Bromine solution (84 µg mL^{-1}) was prepared Dissolve 0.1 g of potassium bromate and 1.0 g of potassium bromide and diluting to 500 mL with distilled water. Dilute 30 mL of the made up stock solution to 100 mL with water. Transfer 50 mL of this made up solution into a 100 mL calibrated flask containing 40 mL of 4.25 mol L^{-1} sulphuric acid and diluting to 100 mL with water to obtain 84 µg mL^{-1} of bromine solution. Prepare it on the day of use. Hydrazine solution (400 µg mL^{-1}) was

prepared by dissolving 0.1626 g of hydrazinium sulphate in 100 mL of water. Formaldehyde solution (7600 µg mL^{-1}) was prepared by diluting 2 mL of formaldehyde (38 %) to 100 mL with water. Suitable aliquot of this solution was diluted to obtain 1000 µg mL^{-1} solution. *p*-phenylenediamine solution (0.5%) was prepared by dissolving 0.50 g of *p*-phenylenediamine in 5 mL of 6 M sulphuric acid and diluting to 100 mL wth distilled water. Bromine solution (600 µg mL^{-1}) was prepared by dissolving 0.2143 g of potassium bromate and 2.143 g of potassium bromide and diluting to 1 L with water. Fifty mL of this stock solution was transferred into a 100 mL calibrated flask containing 40 mL of 4.25 mol L^{-1} sulphuric acid and diluting to 100 mL with water. (300 µg mL^{-1}) This solution was prepared on the day of use. Sulphuric acid (4.25 mol L^{-1}) was prepared by adding 59 mL of concentrated sulphuric acid (Sp.Gravity 1.84) to 150 mL of water, cooled and then diluting to 250 mL. Sulphuric acid (6 M) was prepared by adding 166.7 mL of concentrated sulphuric acid (Sp.Gravity 1.84) to 200 mL of distilled water, cooled and diluted to 500 mL. Formic acid (24 %) was prepared by adding 28 mL of formic acid (85 %) to 50 mL of water and then diluting to 100 mL.

2.3. Procedure

2.3.1. Calibration graph

To 5 mL aliquot of the sample containing 1-15 µg of iodate, 1 mL of 0.05 % *p*-nitroaniline and 1 mL of 500 ppm hydroxylamine were added. The contents were mixed well and kept aside for 10 minutes. 1 mL of 0.1 % N-(1-naphthyl) ethylenediammine dihydrochloride (NEDA) was added before diluting to 10 mL with distilled water. The absorbance of the solution was measured at 545 nm against reagent blank. The plot of absorbance versus concentration of iodate is a straight line with positive slope.

2.3.2. Determination of Iodate

Five mL of the sample containing not more than 15 µg of iodate is treated with 1 mL of 0.05 % *p*-nitroaniline and 1 mL of 500 ppm hydroxylamine. The contents were mixed well and kept aside for 10 minutes. 1 mL of 0.1 % N-(1-naphthyl) ethylenediammine dihydrochloride (NEDA) was added before diluting to 10 mL with distilled water. The absorbance of the solution was measured at 545 nm against reagent blank. The plot of absorbance versus concentration of iodate is a straight line with positive slope.

2.3.3. Determination of Periodate

In a 10 mL calibrated flask, 3 mL of sample containing not more than 16 µg of periodate and 1 mL of 0.5 N sulphuric acid is treated with 1 mL of 500 ppm ethylene glycol. The contents were mixed well and kept aside for 5 minutes. To this solution 1 mL of 0.05 % *p*-nitroaniline and 1 mL of 500 ppm hydroxylamine were added. The contents were mixed well and kept aside for 5 minutes. 1 mL of 0.1 % N-(1-naphthyl) ethylenediammine dihydrochloride (NEDA) was added before diluting to 10 mL with distilled water. The absorbance of the solution was measured at 545 nm against reagent blank. The concentration of iodate is established from the calibration graph.

Concentration of Periodate = Concentration of iodate x 1.09

2.3.4. Determination of Iodide or Iodine

In a 10 mL calibrated flask, 3 mL of sample containing not more than 10 µg of iodide or iodine is treated with 1 mL of 84 ppm of bromine solution. The contents were mixed well and kept aside for 5 minutes. Excess bromine is destroyed with 1 mL of 24 % formic acid. To this solution 1 mL of 0.05 % *p*-nitroaniline and 1 mL of 500 ppm hydroxylamine were added. The contents were mixed well and kept aside for 5 minutes. 1 mL of 0.1 % N-(1-naphthyl)

ethylenediammine dihydrochloride (NEDA) was added before diluting to 10 mL with distilled water. The absorbance of the solution was measured at 545 nm against reagent blank. The concentration of iodate is established from the calibration graph.

3. Results and Discussion

Strong oxidizing agents like Ce^{4+}, MnO_4^- and BrO_3^- oxidize hydroxylamine to nitrate. In some cases nitrite is formed or as an intermediate. Methods based on the oxidation of hydroxylamine to nitrite are reported [25,26]. The generated nitrite was determined by diazo coupling reaction with *p*-nitroaniline and NEDA. The present investigation is based on the oxidation of hydroxylamine to nitrite with a known amount of iodate under acidic condition and the determination of nitrite by diazo coupling reaction between *p*-nitroaniline and NEDA. The formed azodye, 4-(4-nitrophenylazo)-N-(1-naphthyl) ethylenediammine dihydrochloride, has an absorption maximum at 545 nm (Fig.1). The developed colour was stable for 3 h. Iodate and periodate are well known oxidizing agents. They oxidize many inorganic and organic compounds. Most of the oxidizable organic compounds have groups, amino, imino, carboxyl or diol on adjacent carbons. The reaction between periodate and the above mentioned groups is well known as the Malaprade reaction, and was used for the indirect determination of 1,2-diols and related compounds [27-29].

Experimental conditions were optimized for the oxidation of hydroxylamine to nitrite with 12 μg of iodate. The effect of variation in acidity for an effective oxidation of hydroxylamine with a known amount of iodate was studied and it was established that the oxidation was found to be effective in the acidity range 0.1 –1.0 N with respect to sulphuric acid. Thus for all subsequent studies, a reaction acidity of 0.57 N was maintained. The effect of variation of *p*-nitroaniline and NEDA were studied and it was established that 1 mL of 0.05 % *p*-nitroaniline and 1 mL of 0.1% NEDA were sufficient to provide maximum absorbance by forming the dye. Variation study of hydroxylamine showed that 1 mL of 300 μg mL^{-1} hydroxylamine was sufficient for generation of nitrite to form the dye to give maximum absorbance. Further addition upto 1000 μg mL^{-1} of hydroxylamine showed no change in the absorbance value. Hence 1 mL of 500 μg mL^{-1} hydroxylamine is recommended for further investigations. Order of addition of reagents plays a very important role in the reaction. It was established that the addition of *p*-nitroaniline before the addition of hydroxylamine resulted in a maximum absorbance (0.577) for 12 μg of iodate. Whereas for the same concentration of iodate, when the amine was added after the addition of hydroxylamine, a decrease in the absorbance value (0.384) was observed. This observation indicated that there is no loss of nitrite due to competing diazotization reaction with the amine and hence the amine was added before the addition of hydroxylamine for further investigations.

Time required for oxidation of hydroxylamine to nitrite was studied and it was found that a minimum of 2 minutes was sufficient for the oxidation reaction to be completed. Hence a period of 5 minutes was recommended for the oxidation reaction. Samples containing periodate require reduction to iodate. This was achieved by using 1 mL of 500 μg mL^{-1} ethylene glycol under acidic condition. Samples containing iodide and iodine are oxidized to iodate with a known excess of bromine solution prior to determination by the proposed method. Excess bromine was destroyed with formic acid. Under these conditions the system obeys Beer's law in the concentration range 0-15 μg of iodate in a sample volume of 10 mL with a detection limit of 0.216 μg. The molar absorptivity of the system was found to be 8.33 x 10^4 L mol^{-1} cm^{-1}. A calibration graph was obtained with a positive slope and the equation being $Y = 0.1342\ X + 0.5718$ where Y is the absorbance and X (μg) is the concentration of iodate. The correlation coefficient was 0.9998 and the relative standard deviation was 1.7 % (n=10) for 12 μg of iodate.

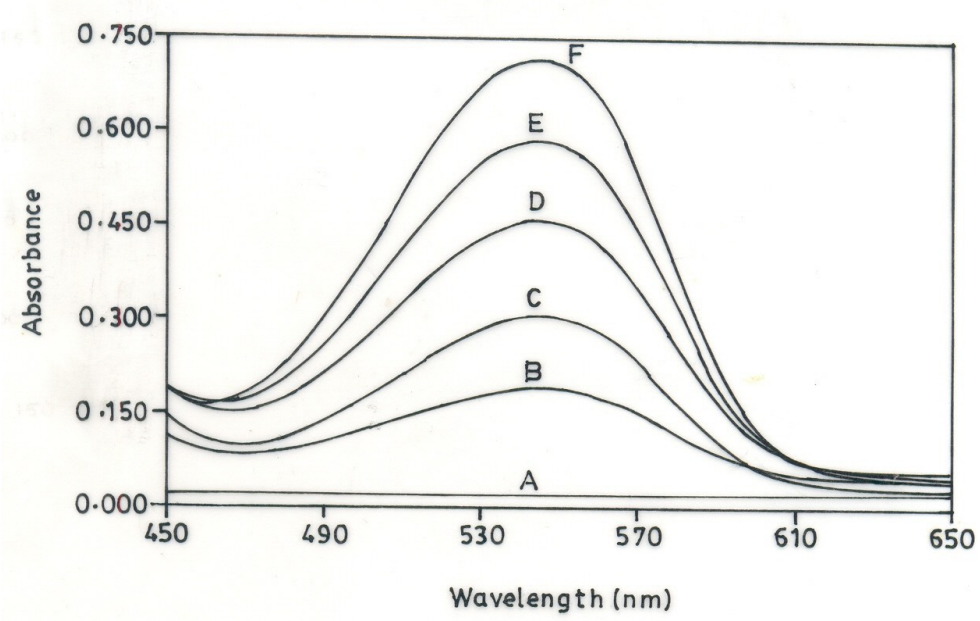

Fig.1. Absorption spectra recorded against water A- blank; B- 3 µg iodate; C- 6 µg iodate; D- 9 µg iodate; E- 12 µg of iodate ; F - 15 µg iodate.

3.1 Interferences

The interfering effect of anions and cations, which may co-exist with iodate were studied. Any deviation in the absorbance of ±0.01 to that obtained in the absence of other interfering ions in iodate determination was taken as a sign of interference. Varying concentration of interfering species was introduced along with 12 µg of iodate and the absorbance values were compared to that in the absence of interference. Tolerance limit of various ions studied in iodate determination are summarized in Table 1. Hydrazine is tolerated upto 400 µg mL^{-1} by the addition of 1 mL of 1000 µg mL^{-1} HCHO prior to the addition of hydroxylamine.

Table 1. Effect of some interfering species in the iodate determination (Iodate = 12 µg)

Species	Amount tolerated (µg)
Phosphate, Oxalate, Citrate, Tartrate, Borate, Carbonate, Bicarbonate, Bromide, Fluoride, Oxalate, Sulphate	1500
Ba (II), Pb (II), Mg (II), Cd (II), Bi (III), Ni (II), Li (I), Sr (II), Cr (III),NH_4^+	1000
Co(II), Mn(II)	100
NO_3^-	100
SO_3^{2-} ,$S_2O_3^{2-}$	1
Ethylene Glycol	1000
Hydrazine	0.1
Hydrazine[a]	100
$Fe(CN)_6^{4-}$	15
HCHO	00
Fe (II)	10
Fe (III)	2
Hg (II), Cu (II)	50
Glucose, acetone	50

[a] Treated with 2 mL of 300 µg mL^{-1} bromine solution, followed by 1 mL of 85 % formic acid prior to the addition of amine and hydroxylamine.

3.2 Applications

In order to evaluate the analytical applicability of the proposed method samples of salt containing iodate or iodide and pharmaceutical preparations containing iodine were analysed. The salt samples from the local market, USA, UK and Germany were analyzed.

p-phenylenediamine method[2] was used as reference method to compare results. The method is based on the oxidation of *p*-phenylenediamine with iodate in acid medium. Table 2 shows the results obtained for the analysis of iodate in iodized salt from India and Germany by both the proposed, reference [2] and BIS (Bureau of Indian Standards) method [30].

Table 2. Determination of iodate in iodized salt

Salt Sample (Manufacturer)	Manufacturer's claim	Amount of iodate present (µg/g) Proposed method[a]	Amount of iodate present (µg/g) *p*-phenylene diamine method[b]	Amount of iodate present (µg/g) BIS method[c]
Annapurna (Hindustan Lever Ltd.) Mumbai INDIA	Minimum 30 ppm of iodine ≡ Iodate 41.34 ppm	39.40* ± 0.28	40.00* ± 1.46	39.66* ± 0.19
Aashirvaad (ITC Ltd.)Kolkata INDIA	Miniumum 30 ppm of iodine ≡ Iodate 41.34 ppm	39.40* ± 0.28	41.07* ± 0.75	40.37* ± 0.50
Udhayam (GHCL Ltd.)Thirupporor,Tamilnadu INDIA	Miniumum 30 ppm of iodine ≡ Iodate 41.34 ppm	40.6* ± 0.28	40.53* ± 0.62	39.66* ± 0.87
Tata iodized Salt (Tata Chemicals Ltd.)Mumbai INDIA	Greater than 15 ppm of iodine ≡ iodate greater than 20.67 ppm	40.60* ± 0.28	39.47* ± 0.75	40.72* ± 0.62
i-shakti Crystal (Tata Chemicals Ltd.),Mumbai INDIA	Greater than 15 ppm of iodine ≡ iodate greater than 20.67 ppm	40.60* ± 0.28	40.00* ± 1.0	41.25* ± 0.75
Marken Jodsalz[d] Reines Alpensalz aus Natursole +Fluorid + Folsaure (Sudsalz Gmbh, Ridlerstr. 75, 80339 Munchen.)	Iodate 20.4µg ≡ Potassium Iodate 0.0025%	20.25* ± .61 0.00248 %	20.28* ± 0.40 0.00248 %	20.59* ± 0.40 0.00252 %
Altlander[e] Jodsalz mit Fluor (Akzo Nobel Salt bv, Den Niederlanden)	Iodate 20.4-32.5 µg ≡ Potassium Iodate 0.0025% -0.0042 %	21.50* ± 0.40 0.00263 %	21.95* ± 0.40 0.00268 %	21.37* ± 0.60 0.00261 %

* Average of three determinations
[a] Volume of sample used : 1 mL of 25 % salt solution.
[b] Volume of sample used : 10 mL of 25 % salt solution..
[c] 20 g of the salt sample was dissolved in 100 mL. To this solution 10 mL of 10 % KI and 5 mL of 2 N sulphuric acid were added. The contents were titrated against 0.05 N sodium thiosulphate solution using starch as indicator.
[d] Marken Jodsalz contain ingredients such as calcium carbonate, magnesium carbonate, sodium fluoride 0.047 %- 0.064 % as additives in addition to iodate.
[e] Altlander Jodsalz contain ingredients such as potassium fluoride 0.065%-0.087%, sodium hexacyanoferrate(II)[E535] and silicon dioxide[E551] as additives in addition to iodate

In the reference method, a 10 mL aliquot of AR sodium chloride solution (25 %), 0-5 mL of 80 µg mL^{-1} iodate, 4 mL of 0.5% *p*-phenylenediamine(ppda) in sulphuric acid were added. The contents were mixed well and the volume made upto 25 mL with water. The colour developed was measured at 525 nm. A calibration graph was obtained by plotting absorbance versus amount of iodate. Salt samples were analysed by the *p*-phenylenediamine (ppda) method by taking 10 mL of salt solution (25%), 6 mL of water, 4 mL of 0.5% *p*-phenylenediamine (ppda) solution in sulphuric acid in a 25 mL calibrated flask. The contents were mixed well and the volume was made upto 25 mL. The colour developed was measured at 525 nm. In the BIS method, 20 g of salt sample is dissolved in 100 mL of water, to this 10 mL of 10 % potassium iodide solution was added, followed by the addition of 5 mL of 1 mol L^{-1} sulphuric acid. The liberated iodine was titrated against 0.025 mol L^{-1} sodium thiosulphate solution, adding 1 mL of 1% starch solution near the end of titration method.

The amount of iodate present in the salt sample was established from the calibration graph. Samples containing iodide or iodine were converted to iodate using bromine solution and removing excess bromine with formic acid [31]. The formed iodate was determined by p-phenylenediamine (ppda) method. Table 3 shows the results obtained for the analysis of iodide in iodized salt from UK and USA. There is a good agreement between the results obtained by the proposed and *p*-phenylenediamine (ppda) method.

Iodophore is a weak complex of iodine and carrier polymer. It has a prolonged microbial action and used as an antiseptic and disinfectant. It is mainly used for cleaning the contaminated wounds, preoperative skin and disinfection of equipments. Pharmaceutical preparations were purchased from the local market and analyzed for iodine content after oxidation to iodate with a known excess of bromine solution. Excess bromine was destroyed with formic acid. The formed iodate was determined by the proposed method and the results compared with the *p*-phenylenediamine (ppda) method. Table 4 shows the results obtained for the analyses of iodine in pharmaceutical preparations.

Table 3. Determination of iodide in iodized salt

Salt Sample (Manufacturer)	Manufacturer's claim	Amount of iodide present (µg/g) Proposed method [a]	Amount of iodide present (µg/g) *p*-phenylenediamine method [b]
Cerebos Extra Fine Iodized Table Salt[c] (Cerebos Middlewich Chechire, England)	Iodide 8.8 µg/g ≡ Potassium Iodide 1150µg/100 g	8.868* ± 0.11 1159 µg/100g	8.815* ± 0.13 1152 µg/100g
Diamond Crystal Iodized salt[d] (Cargill Incorporaed Minneapolis MN-55440)	Iodide 49µg/g ≡ Potassium Iodide 0.0064%	49.667* ± 1.0 0.00649 %	49.543* ± 0.55 0.00648 %

*Average of three determinations.

[a]Volume of sample used: Cerebos Iodized salt: Three mL of 15 % salt solution, Diamond Crystal salt : Three mL of 5 % salt solution;

[b]Volume of sample used : Cerebos Iodized salt: Twelve mL of 30 % salt solution, Diamond Crystal Iodized salt : Ten mL of 25 % salt solution.

[c]Cerebos Iodized salt contain ingredients such as magnesium carbonate, sodium hexacyanoferrate(II) as anticaking agents in addition to iodide.

[d]Diamond Crystal Iodized salt contain ingredients such as silicon dioxide, tricalcium phosphate, dextrose and sodium bicarbonate as additives in addition to iodide

Table 4. Determination of iodine in Pharmaceuticals

Sample (Manufacturer)	Manufacturer's claim	Proposed method [a]	*p*-Phenylenediamine method [b]
		% of iodine in sample	% of iodine in sample
Wokadine Ointment Wockhardt Ltd, New Delhi, India	Available iodine 0.5 % w/w	0.508 ± 0.13	0.498* ± 0.01
Betadine solution G.S.Pharmbutor Pvt.Ltd, Uttarakhand, India	Available iodine 0.5 % w/v	0.508* ± 0.01	0.503* ± 0.01
Betadine Ointment G.S.Pharmbutor Pvt.Ltd,, Rajasthan, India	Available iodine 0.5 % w/w	0.502* ± 0.07	0.498* ± 0.01
Collosol Iodine Oral solution Solvay Pharma India Limited, Ahmedabad, India	Available iodine 8 mg/5 mL	8.104 mg/5mL* ± 0.10	8.063 mg/5mL* ± 0.11

*Average of three determinations

[a]Wokadine and Betadine ointment (0.5 g of ointment dissolved in 50 mL water, 1 mL of this solution was diluted to 50 mL, three mL of this solution was used), Betadine solution (0.5 mL dissolved in 50 mL, one mL of this solution diluted to 50 mL, three mL of this solution was used), Collosol Iodine Oral solution.(2.5 mL dissolved in 100 mL of water, 1 mL of this solution was diluted to 50 mL, three mL of this solution was used.)

[b]Wokadine and Betadine ointment(0.5 g of ointment dissolved in 250 mL water, 10 mL of this solution was used),Betadine solution(1 mL dissolved in 250 mL water,5 mL of this solution was used), Collosol Iodine Oral solution. (2.5 mL dissolved in 250 mL of water, 6 mL of this solution was used).

Table 5. Determination of periodate in sea water[a].

Samples	Periodate added (µg)	Proposed method	*p*-phenylenediamine method[c]
		Amount of periodate present (µg)	Amount of periodate present (µg)
Sample 1	500	496.0* ± 0.20	491.1* ± 0.20
Sample 2	1000	1008.0* ± 0.15	1007.0* ± 0.20
Sample 3	1200	1205.0* ± 0.18	1200.4* ± 0.20

*Average of three determinations.

[a] Sample from Bay of Bengal,Chennai Coast,India

[b] Volume of sample used : One mL .

[c] Volume of sample used : Ten mL.

4. Conclusion

This method described is simple, precise, sensitive and selective for the determination of iodine species iodide, iodate and periodate. The system obeys Beer's law in the concentration range of 0-15 µg of iodate in an overall volume of 10 mL. The developed method is more sensitive compared to the existing spectrophotometric methods. The 'ε' L mol^{-1} cm^{-1} values of spectrophotometric methods based on oxidation of Leuco Xylene CyanolFF [16], Thionin [17] and Varian Blue [18] with iodate are 2.02 x 10^4, 2.7 x $10^4$and 2.19x10^4 L mol^{-1} cm^{-1} respectively. The molar absorptivity of the system is found to be 8.33 x 10^4 L mol^{-1} cm^{-1}. The application of proposed method to the determination of iodide and

iodate in salt samples and iodine in pharmaceutical preparations demonstrate the utility of the method to serve as alternate to the existing methods.

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References

1. Gilman A G, Goodman L S, Rad T W and Murad F (1985) The Pharmacological Basis of Therapeutics, 7th edition, Macmillan, New York.
2. Trivedi R H, Mehta S H, Bhatt S D and Choudhri B P (1987**)** Colorimetric Determination of Iodate in Iodated Salt Using P-Phenylenediamine. Ind J Technol 25: 43.
3. Rodeheaver G, Bellamy W, Kody M, Spatafora G, Fitton L, Leyden K and Edlich R (1982) Bactericidal Activity and Toxicity of Iodine-Containing Solutions in Wounds. Arch Surg 117(2): 181.
4. Edmonds J S and Morita M (1998) The Determination of Iodine Species in Environmental and Biological Samples. Pure Appl Chem 70: 1567.
5. Alegret S, Florido A, Lima JLFC and Machado AASC (1989) Flow-Through Tubular Iodide and Bromide Selective Electrodes Based on Epoxy Resin Heterogeneous Membranes. Talanta 8: 825.
6. Crecelius EA (1975) Determination of Total Iodine in Milk by X-Ray Fluorescence Spectrometry and Iodide Electrode. Anal Chem 47: 2034.
7. Vanhoe H F, van Allemeersch, Versieck J and Dams R (1993**)** Effect of Solvent Type on the Determination of Total Iodine in Milk Powder and Human Serum by Inductively Coupled Plasma Mass Spectrometry. Analyst 118: 1015.
8. Schrame P and Hasse S (1994) Iodine Determination in Biological Materials by ICP-MS. Microchim Acta 116: 205.
9. Rubio S and Perez-Bendito D (1989) Micellar Media in Kinetic Determinations. Anal Chim Acta 224: 185.
10. Kennedy R O, Bator J, Reading C (1989) A Microassay for The Determination of Iodide and Its Application to The Measurement of The Iodination of Proteins and The Catalytic Activities of Iodo Compounds, Anal. Biochem 179:139.
11. Garcia M.S, Sanchez-Pedreno C, Albero M.I and Sanchez C (1991) Kinetic Determination of Iodide in Pharmaceutical and Food Samples. Analyst 116: 653.
12. Yonehava N, Yamana T, Tomiyasu T and Sakamoto H (1989) Simultaneous Spectrophotometric Determination of Traces of Bromide and Iodide Based on Their Catalytic Effects on Pyrocatechol Violet-Hydrogen Peroxide Reaction. Anal Sci 5:175.
13. Esmadi FT,.Kharoaf MA and Attiyat M S (1991) Sequential Atomic Absorption Spectrometric Determination of Chloride and Iodide in A Flow System Using an On-Line Preconcentration Technique. Analyst 116: 353.

14. Koh T, Ono M and Makino I (1989) Spectrophotometric Determination of Iodide at the 10^{-6} mol L^{-1} Level by Solvent Extraction with Methylene Blue. Analyst 13:945-948.

15. Niazi S B, Mozammil M (1991**)** Spectrophotometric Determination of Traces of Iodide by Liquid-Liquid Extraction of Brilliant Green-Iodide Ion Pair. Anal Chim Acta 252: 115.

16. Chand P, Narayana B (2006) A Highly Sensitive Spectrophotometric Method for the Determination of Iodate Using Leuco Xylene Cyanol FF. Acta Chim Slov 53: 77.

17. Narayana B, Cherian T (2005) A Facile Spectrophotometric Method for The Determination of Iodate in Table Salt using New Chromogenic Reagents, South African J Chem 60: 8-10.

18. Cherian T, Narayana B (2005) Rapid Spectrophotometric Determination of Trace Amounts of Chromium Using Variamine Blue As a Chromogenic Reagent. J Braz Chem Soc 16(2): 197-201.

19. Bhattacharyya SN and Chetia PK (1967**)** Spectrophotometric Determination of Periodate in Presence of Iodate Using Aluminum Hydroxide. Anal Chem 19: 369.

20. Kamburova M (1992) Triphenyltetrazolium chloride for determination of iodate and Periodate, Talanta 39:987

21. El-Shahawi M.S and Al-Hashimi FA (1996) Spectrophotometric Determination of Periodate or Iodate Ions by Liquid-Liquid Extraction as an Ion-Pair Using Tetramethylammonium Iodide. Talanta 43: 2037.

22. Harez A and Bashir W (1985) p-Phenylenediamine as a Chromogen for Iodate and Periodate in Aqueous Solution. Microchem J 31: 375.

23. Callejon M and Munoz J (1986) Spectrophotometric Determination of Periodate with the Bisthiosemicarbazones of Phthalimide and 1, 3-Indandione. Microchem J 34: 83.

24. El-Shahawi M.S (1997) Extraction Equilibrium of The Ion-Associate of Periodate with Amiloride Hydrochloride and Simultaneous Spectrophotometric Determination of Periodate and Iodate by Liquid-Liquid Extraction. Anal Chim Acta 356: 85

25. 25. Balasubramanian N and Maheswari V (1996) Indirect spectrophotometric determination of chromium, Journal of AOAC International 79(4)

26. Maheswari V and Balasubramanian N (1997) Chemical Amplification Method for the Determination of Chromium at Trace Levels. Analusis 25: 2.

27. Haree ZAK (1985) p-Phenylenediamine as A Chromogen for Iodate and Periodate In Aqueous Solution. Microchem J 31: 375.

28. Rammler DH, Bilton R, Haugland R and Parkinson C (1973) Hydrolytic Enzyme Substrates. II. A New Method for Measuring Periodate, Anal. Biochem 52:198.

29. 29. Verma KK, Gupta DS Sangh SK and Jain A (1987) Spectrophotometric Determination of Periodate With Amodiaquine Dihydrochloride and Its Application to the Indirect Determination of Some Organic Compounds via the Malaprade Reaction. Analyst 112: 1519.

30. Indian Standard Specification for Iodized Salt IS 7224:1985 (Bureau of Indian Standards, New Delhi, 1986).

31. Bhatty MK and Townshend A (1971) Spectrophotometric Determination of Small Amounts of Nitrate and Nitrite by Conversion to Nitrotoluene and Extraction Into Toluene. Anal Chim Acta 56: 55.