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### Voltammetric Micro Level Determination of Periodate

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A simple and convenient method is presented for the determination of submicro level concentration of periodate based on differential pulse polarographic reduction of  $\text{IO}_4^-$  in sodium hypochlorite medium. Linearity of the calibration curve was achieved upto 26 ppm with a limit of determination of  $0.02 \mu\text{g/ml}$ . The possible interference of commonly present metal ions was examined and ruled out. The method has been successfully applied for the analysis of periodate in industrial waste sample.

**Keyword:** Periodate, differential pulse polarography, industrial waste analysis

#### 1. Introduction

The oxidation state specific determination of inorganic anionic species in aqueous system of vital significance in correlation of toxicity with speciation and better understanding of their influence on environment<sup>[1]</sup>. Sulfide, nitrite, chlorate and cyanide are toxic. In the present work attention was focused on periodate which is a dominant oxyiodine species. Periodate is extremely hazardous and its overexposure by inhalation may cause respiratory irritation<sup>[2]</sup>.

$\text{IO}_4^-$  is not a natural component of water, but may be formed during the ozonation (disinfection) of raw water or chemical processes in industries, as a byproduct of iodide. It may further be reduced to iodates<sup>[3]</sup>. This all has made periodate a rather concerned species to be studied and determined at micro levels.

Voltammetric methods are considered more suitable to the conventional methods of oxyhalides estimation (optical and ion-selective electrodes) because the measurement of chemical form of the concerned ion is possible due to certain selectivity of the redox potential<sup>[4]</sup>. Differential pulse polarography (DPP) and stripping voltammetry have proved useful in such determinations. However, during stripping voltammetry very positive deposition potential of oxyhalide ions causes problems in obtaining the

correct measurements for analytical purposes<sup>[5]</sup>. Therefore, the suitability of DPP is envisaged in present studies.

The electrochemistry of oxyiodine species is enriched by the fact that iodine exists in several oxidation states. Periodate is stable in its +7 state<sup>[6]</sup>. The reported polarographic studies of periodate have revealed two consecutive irreversible step electroreduction of periodate in acidic medium. Periodate is first reduced to iodate and in second step iodate reduces to give iodide<sup>[7]</sup>. The polarographic reduction of periodate has also been reported in unbuffered solutions<sup>[8]</sup>, and in alkaline medium<sup>[9]</sup>.

Torpava *et al*<sup>[10]</sup> have shown catalytic currents of iodide and periodate ions on dropping and stationary electrodes in presence of cystine.

Hajjaji *et al*<sup>[11]</sup> have described an indirect polarographic determination method of periodate based on its reaction with hydroxylamine. Temerk *et al*<sup>[12]</sup> have determined iodate, periodate and bromate in Britton-Robinson buffer with a concentration limit of  $2.4 \times 10^{-6} \text{ M}$ . Here, the determination of periodate in polarographic medium of  $\text{NH}_3\text{-NH}_4\text{Cl}$  in presence of potassium nitrate using DPP has been evaluated. The results obtained by suggested method have also been compared with UV-vis spectrophotometric method. The determination of nitrite-nitrate<sup>[13]</sup>, iodide<sup>[14]</sup>,

fluoride <sup>[15]</sup>, chlorate <sup>[16]</sup> and sulfide <sup>[17]</sup> have been reported earlier.

## 2. Methods and Procedure

### 2.1 Instrumentation

A microprocessor based pulse polarographic analyzer (Model CL-362) in combination with a drop-timer assembly, all from Elico Limited, Hyderabad, India, was used for voltammetric measurements. Current voltage curves were recorded by an Epson printer (Epson-LX-300+II). The instrumental settings for DPP were as follows: a dropping mercury electrode was used as the working electrode; pulse amplitude, 25 mV; drop time, 0.5 s; scan rate, 12 mV/sec and charging current compensation, 20%. The saturated calomel electrode (SCE) and platinum wire worked as a reference and auxiliary electrodes, respectively.

The pH measurements were made by a digital pH meter (Model- 5000) of India.

Sample analysis was also carried out by a UV-vis spectrophotometer (Model SL-160) of Elico. It has a wavelength range of 190-1100 nm. The tungsten-halogen deuterium lamp and wide range photomultiplier were also used as the light source and detector, respectively. The spectral band width of resolution was 0.5 nm.

### 2.2 Sample Preparation

Waste water samples were collected in clean polyethylene containers. These were filtered in order to separate any suspended particulate matter and were acidified with hydrochloric acid to pH 2.0 for storage. A 100 ml aliquot of the sample was treated with 1 ml of an oxidizing mixture of nitric acid and sulfuric acid to destroy the biological and organic matters <sup>[18]</sup>.

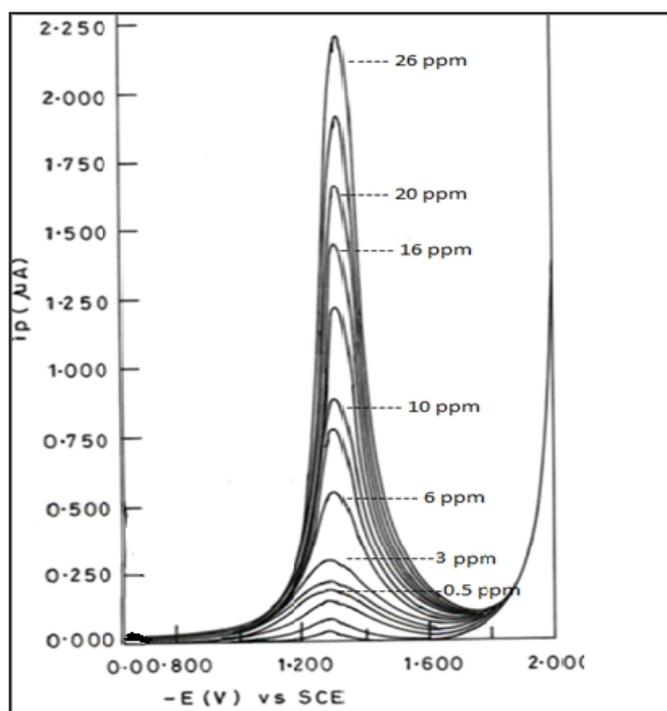
### 2.3 Chemicals

Chemicals used were of analytical grade purity. Stock solution of periodate was prepared from  $\text{KIO}_4$  of Loba Chemie, Mumbai (Batch no. 56621). Solutions were prepared in milli-Q-water (Millipore).

The measurements were made in air conditioned laboratory at  $25 \pm 1$  °C. The test solutions were deaerated by bubbling purified nitrogen for 20 mins prior to voltammetric recordings. Nitrogen was purified by passing it through a vanadous chloride scrubbing solution <sup>[19]</sup>.

## 3. Results and Discussion

$\text{IO}_4^-$  showed a sharp DP peak in 0.145 M sodium hypochlorite at -1.35V. A linearity between peak height and periodate concentration was observed between 0.02 and 26.0 ppm as shown in figure 1. The characteristics of the calibration curve were as follows: slope = 0.08; intercept = 0.06; coefficient of correlation( $r$ ) = 0.99 and standard deviation =  $\pm 0.74$ .



**Fig 1:** DP polarograms of periodate at different concentrations in presence of 0.145 M sodium perchlorate

### 3.1 Interference

DP polarograms were also recorded in presence of other common metal ions such as copper, lead, cadmium and zinc. The DP peaks of these ions were distinguishable from each other and had different peak potentials are listed in table 1, thus showed no interference.

**Table 1:** Peak potential of metal ions in 0.145M sodium hypochlorite

S. No.	Metal ion	-E <sub>p</sub> (V) vs SCE
1	Cu (II)	0.03
2	$\text{IO}_4^-$	1.35
3	Pb (II)	0.35
4	Cd (II)	0.54
5	Zn (II)	1.01

The possibility of influence of iodate present in solution was also ruled out by analyzing a synthetic sample of periodate in presence of iodate. The results obtained were quantitative in terms of periodate concentration present in sample.

### 3.2 Determination of $\text{IO}_4^-$

A measured volume of the prepared sample was taken into the polarographic cell with 0.145 M sodium hypochlorite supporting electrolyte for the determination of periodate.

A DP polarogram was recorded over the potential range of -0.8V to -1.5V. Peak current was noted after making blank correction. It was done by recording the current of the supporting electrolyte alone without any salt, and subtracting it from the peak current obtained for test solution of sample analysis.

### 3.3 Limit of determination

The limit upto which a measurable DP peak of  $\text{IO}_4^-$  was observed was taken as its limit of determination. The minimum amount which thus could be determined was found to be 0.02  $\mu\text{g/ml}$ .

## 4. Analytical application

### 4.1 Voltammetric measurements

The concentrations in all observations were determined by the standard addition method [20]. Generally two standard additions were made for the quantitation. The results of determination of periodate in samples of industrial waste water are summarized in table 2.

**Table 2:** Peak potential of metal ions in industrial waste  $\text{H}_2\text{O}$  samples

S. No.	Samples	Conc. ( $\mu\text{g/ml}$ )			S.D. ( $\pm$ )	C.V. (%)
		Min.	Max.	Ave.		
1	Paint Industry	1.47	1.52	1.50	0.02	1.33
2	Navkar Basni	2.21	2.25	2.24	0.02	0.89
3	Ronk Industry	2.58	2.65	2.61	0.03	1.14

N = 3, number of samples analysed

### 4.2 Comparison

The UV-vis spectrophotometric method was used to compare the results obtained by DPP. The observations are included in table 3.

**Table 3:** Comparison of results between DPP and UV-vis spectrophotometry

Sample	$\text{IO}_4^-$ Conc. ( $\mu\text{g/ml}$ )	
	DPP	UV-vis
Sample 1	1.50	1.48
Sample 2	2.24	2.21
Sample 3	2.61	2.60

## 5. Conclusion

It is difficult to determine microgram levels of periodate by the conventional methods of estimation of halides and oxyhalides due to either by interference of major ions or sensitivity of the technique. Ion chromatographic method has the required sensitivity, but its high cost and non-availability in common laboratories makes it inconvenient. The suggested method seems to overcome these problems as it enables the determination of trace periodate in waste waters which usually contains many other interfering ions. Furthermore, the results of determination of periodate are in good agreement with other methods including DPP.

## 6. Acknowledgement

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