

Research Journal of Pharmaceutical, Biological and Chemical Sciences

Promethazine As Novel Reagent For Estimation Of Iodate Ion In Table Salt Samples.

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ABSTRACT

A simple, rapid and accurate spectrophotometric method has been developed for the determination of iodate ion using promethazine. The proposed method reports the reaction of iodate ion with promethazine in acidic medium to form a red colored product with an absorption maximum at 515 nm. Beer's Law was obeyed in the range of 0.2-2.8 µg/ml with molar absorptivity of $7.63 \times 10^4 \text{ L.mol}^{-1}.\text{cm}^{-1}$. The relative standard deviation of the method was less than 1.5% and accuracy (average recovery) was $100 \pm 1.1\%$. The proposed method has been successfully applied for the determination of iodate ion in table salt samples.

Keywords: Iodate, promethazine, spectrophotometric, table salt samples.

<https://doi.org/10.33887/rjpbcs/2020.11.1.12>

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INTRODUCTION

For correcting iodine deficiency. Table salts are iodized with potassium iodate instead of potassium iodide to avoid the possible photochemical or chemical oxidation of iodide to iodine with atmospheric oxygen (iodate is more stable than iodide)[1]. The recommended concentration of iodine in the salt is range from 5–100 $\mu\text{g/g}$ (ppm) of iodine in salt depending on the country of manufacture and the storage conditions[2-5]. Iodine is an essential part of the thyroid hormones that play an important role in the development of brain function and cell growth. Iodine deficiency is one of the most commonly occurring nutritional problems. Iodine deficiency can be caused by consumption of iodine less salt or less iodine containing salt below the minimum[3]. Iodine deficiency can cause goiter, cretinism, reduced of intelligence, mental retardation, brain damage, deaf-mutes, and cause miscarriage in pregnant women. On the other hand, an excess of iodine or iodide can cause goiter and hypothyroidism [4,5]. Therefore, determination of iodine in table salt is very important for successful iodine supplementation. Several analytical methods, such as spectrophotometry [6–8], indirect iodometry[9], spectrofluorometry [10], high performance liquid chromatography [11], chemiluminescence[12], gas chromatography-mass spectrometry [13], capillary zone electrophoresis [14], electro analytical methods [15], and inductively coupled plasma mass spectrometry[16] have been reported for the determination of iodate. In this work, we intend to develop a spectrophotometric procedure for the determination of iodate in different Table Salt samples.

EXPERIMENTAL

Apparatus

Spectra UV / Visible dual beam spectrophotometer [UVS-2700, Labomed, INC] with 1.0 cm quartz cells were used .

Reagents

All chemicals used were of analytical purity grade and all solutions were prepared in distilled water. A stock solution(100 ppm) of iodate was prepared by dissolving 0.122 g of potassium iodate in 100 mL of water and standardized using sodium thiosulphate solution Standard solution of iodate (10 ppm) was prepared by diluting 10 ml of the stock solution to 100 ml by distilled water in a volumetric flask. Promethazine hydrochloride solution 0.1% was prepared by dissolving 0.1g of Promethazine hydrochloride in 100ml distilled water in a volumetric flask .Sulfuric acid 1M, this solution was prepared by diluting 14 ml of 18 M H_2SO_4 solution to 250 ml by distilled water in a volumetric flask.

Recommended procedure

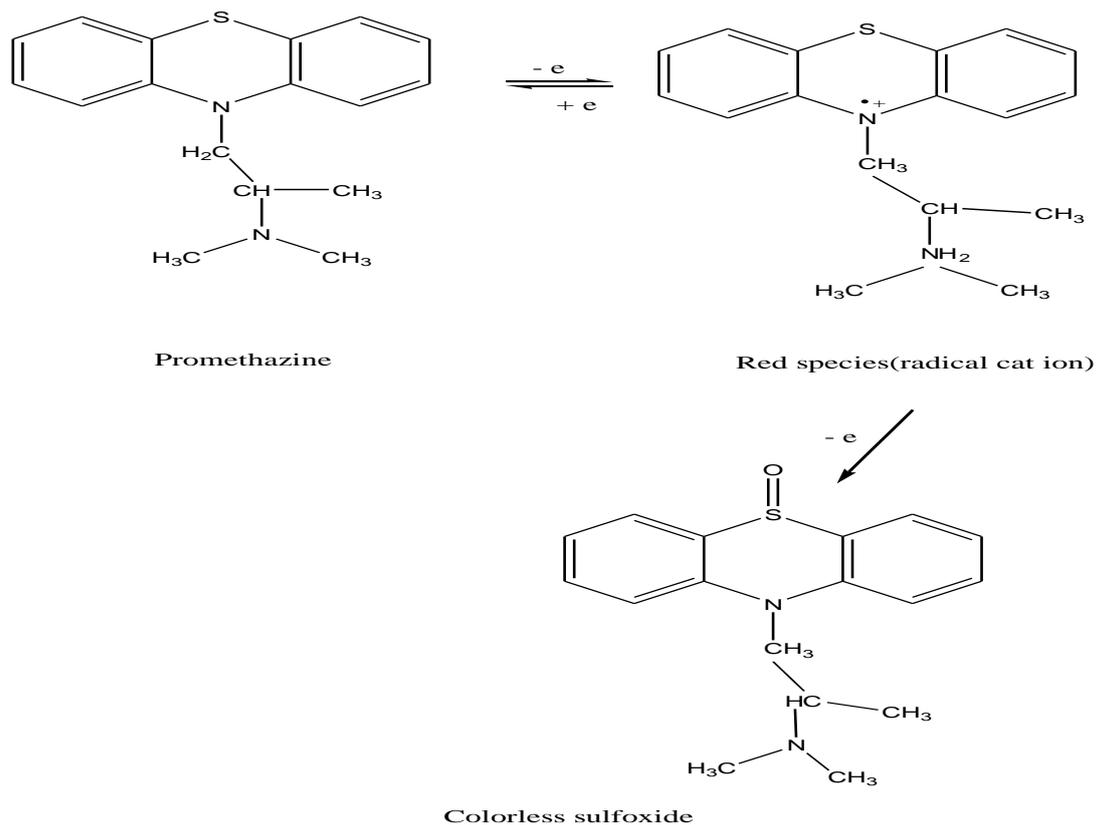
Aliquots of standard solution of iodate (5-70 μg) were transferred into a series of 25 ml calibrated flasks, added 5 ml of 1 M sulfuric acid solution and 5 ml of 0.1% Promethazine solution , dilute the solution to the mark with distilled water . The absorbance of the red-colored product was measured at 515 nm against a reagent blank.

Sampling Preparation

The table salt samples of different brands were purchased from local markets. 20 g of table salt sample was transferred into a 50mL volumetric flask, and after dissolving in de ionized water, the solution was diluted to the mark with high-purity de ionized water. and the iodate ion content was determined as mentioned under recommended procedure.

RESULTS AND DISCUSSION

Promethazine hydrochloride is oxidized in sulfuric acid medium with iodate ion solution instantaneously at room temperature to form a red-colored product which is believed to be a radical cat ion ,and is irreversibly oxidized to a colorless sulfoxide with loss of electron[17], as shown below.



The red –colored radical cat ion shows maximum absorbance at 515 nm , where the blank does not absorb appreciably, as shown in Fig [1].

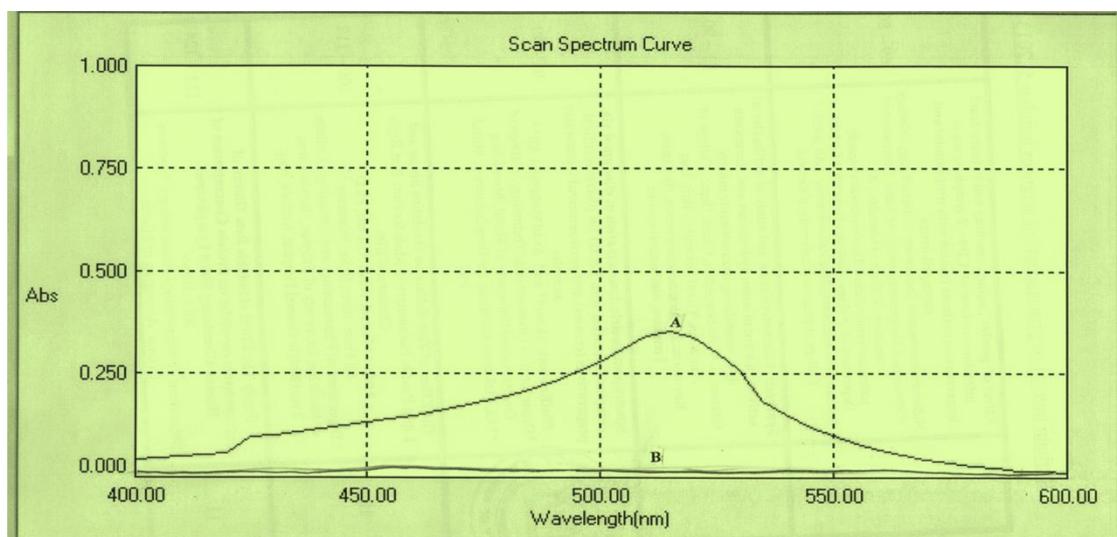


Fig 1: Absorption spectra of A- 1.6µg/ ml of iodate -promethazine product against blank. B-blank against distilled water.

The reaction variables were optimized by varying each variable while keeping others constant for obtaining maximum absorbance. The oxidation reaction was found to be quantitative in sulfuric acid medium .It was found that 3-7 ml of 1 M sulfuric acid solution give high sensitivity and 5 ml of this solution has been used for subsequent experiments . The effect of the amount of promethazine on the absorbance was investigated . A maximum and constant absorbance was found with 3 to 6 ml of 0.1% promethazine solution and 5 ml has been used for subsequent experiments . The color reaction occurred at room temperature immediately and remained stable for at least 6 hours. and a reaction time of 5 min was selected for reproducible results. under the experimental

conditions described, Beer's law is obeyed over the concentration range 0.2-2.8 $\mu\text{g/ml}$ Fig [2], with correlation coefficient of 0.999, intercept of -0.011 and slope of 0.243.

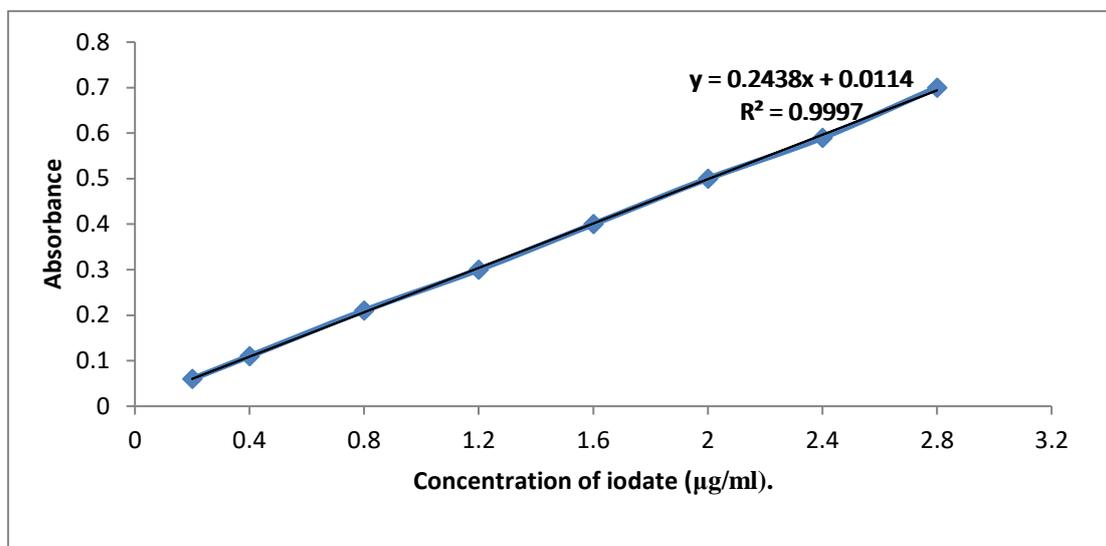


Fig 2: Calibration graph of iodate.

The conditional molar absorptivity of the product formed and sandell's sensitivity were found to be $7.63 \times 10^4 \text{ L.mol}^{-1}.\text{cm}^{-1}$ and 4.115 ng/cm^2 respectively. The accuracy and precision of the method was established by analyzing the standard iodate solution at three different levels. The average recovery which is a measure of accuracy is 100 ± 1.1 revealing high accuracy of the method. The relative standard deviation (RSD), which is an indicator of precision is better than 1.5%. The results are compiled in Table[1].

Table 1: Optical characteristics and statistical data for regression equation of the proposed method

Parameters	Value
λ max (nm)	515
Beer's law limit ($\mu\text{g} .\text{ml}^{-1}$)	0.2-2.8
Molar absorptivity ($\text{l.mol}^{-1}.\text{cm}^{-1}$)	7.63×10^4
Correlation coefficient (r^2)	0.9997
Regression equation ($Y = a \times x + b$)	
Slope (a)	0.243
Intercept (b)	0.0114
Recovery %	100 ± 1.1
Relative standard deviation (%)	< 1.5%

Effect of diverse ions

The effect of various diverse ions on the determination of iodate ion by the proposed procedure was examined. The tolerance limits of interfering species were established at the concentration required to cause not more than $\pm 2\%$ error in the recovery of iodate at $1 \mu\text{g} .\text{mL}^{-1}$. The tolerance limits of diverse ions are listed in Table 2. The oxidizing agents such as Fe(III), Ce (IV), and V(V), interfere severely with the determination of iodate. The tolerance limit of iron, cerium and vanadium can be increased by the addition of appropriate (1 mL of 2% NaF) amount of sodium fluoride [18].

Table 2: Effects of diverse ions in the determination of ($1.0 \mu\text{g.mL}^{-1}$) of iodate

Foreign ion	Tolerance limit in $\mu\text{g.mL}^{-1}$
Mg^{2+} , Ca^{2+} , Br^- , Na^+ , Cl^-	> 6000
NO_3^- , Mn^{2+} , Zn^{2+}	2000
SO_4 , PO_4^{3-} ,	1500
Al^{3+} , SCN^-	1000
* Fe^{3+} , * Ce^{4+} , * VO_3^-	≤ 3

* Masked with masking agents.

Application to real samples

In order to evaluate the applicability of the method to real sample, it was applied to the determination of iodate in table salt samples of different brands which purchased from local markets. The results were also compared statistically by student t-test with those obtained by reference method at 95% confidence level. The calculated t- values did not exceed the theoretical values indicating that there was no significant differences between the precision of the proposed and literature method as cited in Table(3) .

Table 3: Determination of iodate ion in table salt samples.

Sample and label claim	Amount of iodate($\mu\text{g/gm}$) *		t-value
	<u>Proposed method</u>	<u>(p-aminophenol) standard method</u>	
1-Magic time salt Miami TL3122 co.USA.	7.72	7.78	0.77
2- ZER (Turkey) [25-40ppm]	32.893	33.00	1.06
3- Salt Duhok(Duhok-Iraq)[70 mg/kgm]	27.616	27.92	1.01
4- Al- Rashed(Mosul-Iraq)[18-30mg/kgm]	19.9	20.00	0.84
5-Aissaray(Turkey)[20-40mg/100kgm]	25.970	26.00	0.74
6- Zidnee (K.S.A)[70-100 mg/kgm]	31.114	31.15	1.02
7- Bil Bak(Turkey) [25-40ppm]	29.06	29.31	1.12

*Average of ten determinations.

t values (n=10, at 95% confidence level tabulated value 2.101).

CONCLUTIONS

For the first time, promethazine has been used as a chromogenic reagent for the spectrophotometric determination of iodate ion . The proposed method, which is sensitive , accurate, simple and rapid, offers the advantages of sensitivity and wide range of determinations without the need for extraction or heating. The method does not involve any stringent reaction conditions and can be compared favorably with the other methods. The proposed method has been successfully applied to the determination of iodate ion in various table salt samples.

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