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NOVEL COLORIMETRIC METHOD 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 trifluoperazine. The proposed method reports the reaction of iodate ion with trifluoperazine in acidic medium to form a red colored product with an absorption maximum at 500 nm. Beer's Law was obeyed in the range of 0.2-2.8µg/ml with molar absorptivity $4.08x10^4$ $L.mol.^{-1}.$ cm^{-1}. The relative standard deviation of the method was less than 2% and accuracy (average recovery) was 100±1.3%. The optimum conditions for all color development are described and the proposed method has been successfully applied for the determination of iodate ion in table salt samples.

KEYWORDS: Iodate, Spectrophotometric, table salt samples.

INTRODUCTION

Sodium chloride salt is expected to be one of the basic and important food materials and is an important trace element in biology and medicine. Therefore, it is recommended to examine the iodate content of the salt.[1] 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).[2] The recommended concentration of iodine in the salt is range from 5–100 µg/g (ppm) of iodine in salt depending on the country of manufacture and the storage conditions.[3-6] 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.[4] Iodine deficiency can cause goitre, cretinism, reduced of intelligence, mental retardation, brain damage, deaf-mutism, and cause miscarriage in pregnant women. On the other hand, an excess of iodine or iodide can cause goiter and hypothyroidism.[5,6] Therefore, determination of iodine in table salt is very important for successful iodine supplementation. Several analytical methods, such as spectrophotometry[7–11], indirect iodometry[12], spectrofluorometry[13,15], high performance liquid chromatography[16–17], chemiluminescence[18], gas chromatography-mass spectrometry[19-20], capillary zone electrophoresis[21-23], electro analytical methods[24–26], and inductively coupled plasma mass spectrometry[27] 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[Aldrich] 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.[28] Standard solution of iodate (10ppm). This solution was prepared by diluting 10 ml of the stock solution to 100 ml by distilled water in a volumetric flask.

Trifluoperazine hydrochloride solution 0.1% was prepared by dissolving 0.1g of trifluoperazine hydrochloride in 100ml distilled water in a volumetric flask.

Sulfuric acid 5M, this solution was prepared by diluting 36 ml of 18M H_2SO_4 solution to 100 ml by distilled water in a volumetric flask.

Recommended procedure

Aliquots of standard solution of iodate (5-70µg) were transferred into a series of 25 ml calibrated flasks, added 1 ml of 5M sulfuric acid solution and 5 ml of 0.1% trifluoperazine

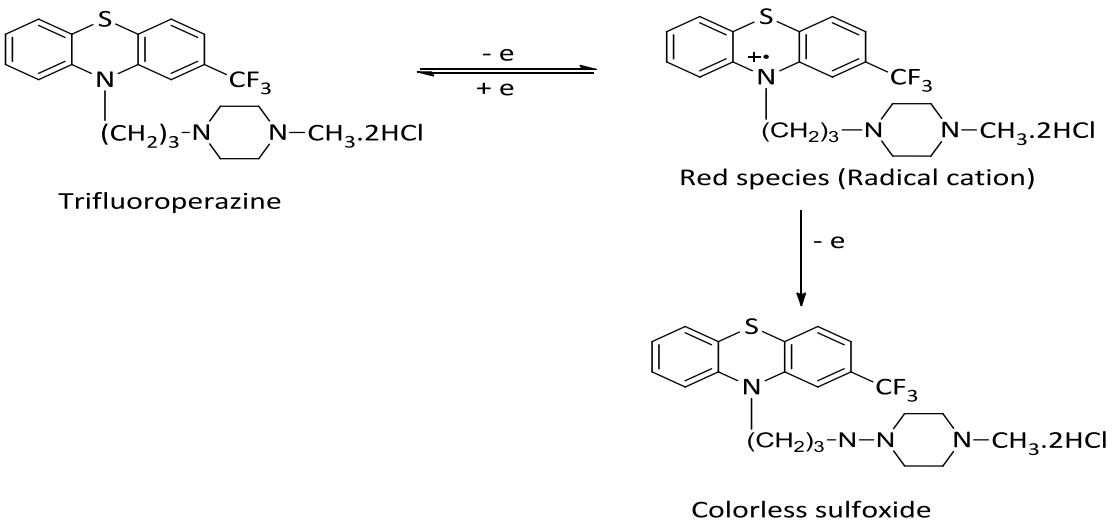
solution, dilute the solution to the mark with distilled water. The absorbance of the red-colored product was measured at 500 nm against a reagent blank.

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

Trifluoperazine 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 cation, and is irreversibly oxidized to a colorless sulfoxide with loss of electron[29], as shown below.



The red –colored radical cat ion shows maximum absorbance at 500 nm, where the blank does not absorb appreciably, as shown in (figure 1).

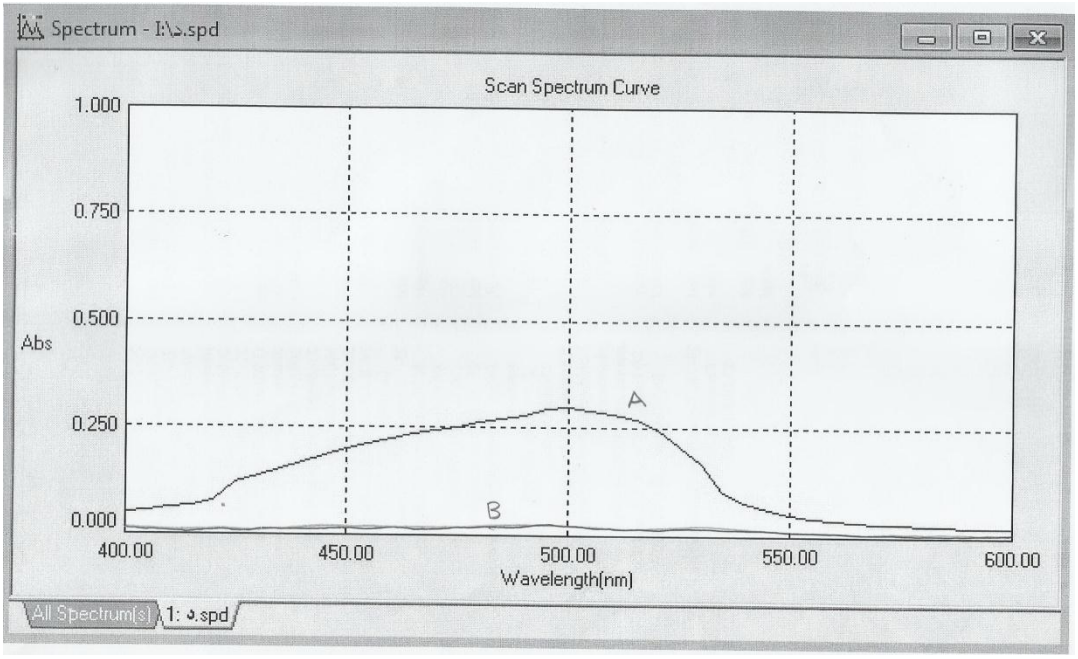


Fig. (1): Absorption spectra of A- 2 µg/ ml of iodate -trifluoperazine product against blank. B-blank against distilled water.

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 0.5-3 ml of 5 M sulfuric acid solution give high sensitivity and 1ml of this solution has been used for subsequent experiments. The effect of the amount of trifluoperazine on the absorbance was investigated. A maximum and constant absorbance was found with 3 to 6 ml of 0.1% trifluoperazine solution and 5 ml has been used for subsequent experiments. The color reaction occurred at room temperature immediately and remained stable for at least 2 hour, 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 µg /ml (figure 2), with correlation coefficient of 0.999, intercept of 0.038 and slope of 0.13.

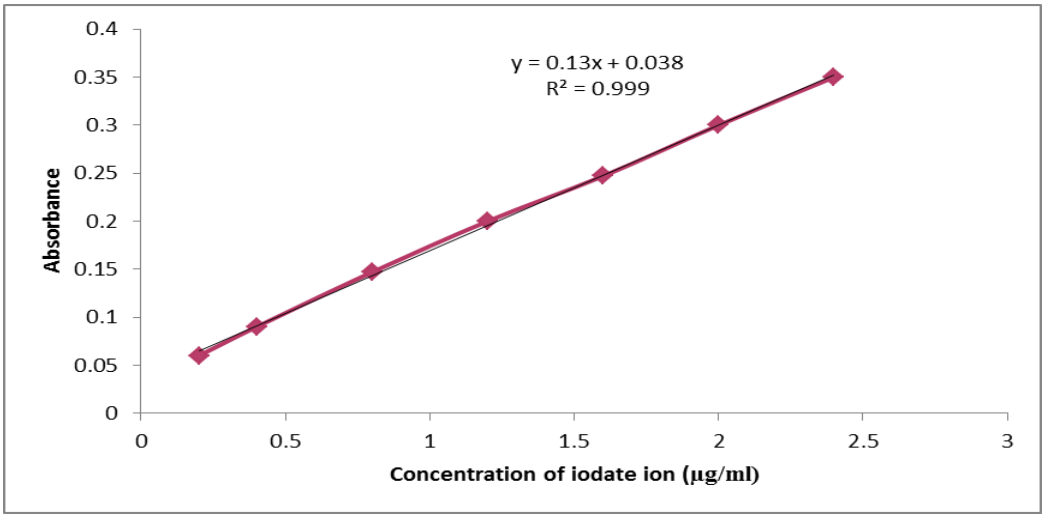


Fig. (2):- Calibration graph of iodate.

Molar absorptivity of the product formed and sandell's sensitivity were found to be 4.08×10^4 $L.mol^{-1}.\ cm^{-1}$ and 7.696 ng/cm^2 respectively. 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 ± 1.3 revealing high accuracy of the method. Relative standard deviation (RSD), which is an indicator of precision is better than 2%. 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)	500
Beer´s law limit (µg. ml^{-1})	0.2-2.8
Molar absorptivity ($l.mol^{-1}.\ cm^{-1}$)	4.08×10^4
Correlation coefficient (r^2)	0.999
Regression equation(Y= a × + b)	
Slope (a)	0.13
Intercept (b)	0.038
Recovery %	100±1.3
Relative standard deviation (%)	< 2%

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 ± 2% error in the recovery of iodate at 1 µg. 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.[30]

Table 2: Effects of diverse ions in the determination of (1.0 µg.mL^{-1}) of iodate.

Foreign ion	Tolerance limit in µg.mL^{-1}
Mg^{2+}, Ca^{2+}, Br^-, Cl^-	3500
NO_3^-, Mn^{2+}, Zn^{2+}	3000
SO_4, PO_4^{3-},	2500
Al^{3+}, SCN^-	1500
$^\star Fe^{3+}$, $^\star Ce^{4+}$, $^\star VO_3^-$	≤ 4

* Masked with masking agent (NaF).

Application to real samples

In order to evaluate the analytical applicability of the proposed method, samples of table salt containing iodate(provided from different companies as cited in table 3) were analyzed. The salt samples of different brands were purchased from local markets. The results were also compared statistically by student t-test with those obtained by standard method[31] 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	Amount of iodate(µg/ml) * t-value Proposed method P-aminophenol standard method
Magic time salt (Miami TL3122 co.USA	**6.25mg/kgm 6.3 1.45**
Salt Duhok(Duhok-Iraq)	**26.7 27 1.65**
Al-Rashed(Mosul-Iraq)	**19.8 20 0.98**
Bil Bak(Turkey) [25-40ppm]	**27.6 27.9 1.01**
ZER (Turkey) [25-40ppm]	**29.8 29.00 0.84**
Zidnee (K.S.A)[70-100 mg/kgm]	**24.9 26.00 0.74**
	31.114 31.15 1.02

*Average of ten determinations.

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

CONCLUSIONS

For the first time, trifluoperazine 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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