



## ASSESSMENT OF IODINE IN DIFFERENT SAMPLES USING 2, 8-DIMETHYL-3, 7-DIAMINOPHENAZINE FOR AN ENVIRONMENTAL FAVORABLE APPROACH

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**Abstract:** An environmental favorable spectrophotometric approach is proposed for the analysis of iodine. The tactic includes oxidation of iodine to iodate,  $\text{IO}_3^-$  with bromine water and therefore the liberation of free iodine from the iodate by addition of iodide in acidic medium  $\text{pH } 4 \pm 0.2$ . The liberated iodine bleaches the pinkish red color of 2,8-dimethyl-3,7-diaminophenazine. The decrease in the intensity of the colour of the dye was measured at 530 nm. Beer's law is obeyed in the concentration range of 2-13  $\mu\text{g}$  of iodine. The molar absorptivity and Sandell's sensitivity of the colour system in  $1.90 \times 10^5 \text{ l mol}^{-1} \text{ cm}^{-1}$  and  $66.78 \times 10^{-5} \mu\text{g cm}^{-2}$  respectively. The analytical parameters were optimized and the method has been applied for the determination of iodine in tap water and soil.

**Keywords:** Environment, Iodine, Spectrophotometric, Sustainable approach.

### INTRODUCTION

Iodine is an essential non-metal part in nutrition. Iodine occurs naturally not solely but as iodide, however, may occur additionally as iodate within the kind of minerals such as lautarite and dietzeite (Gillman *et al.*, 1985). Iodine can be found naturally in air, water and soil and there are some iodine-containing minerals. The chronic intoxication including irritation and nervousness develop from the ingestion of excessive amount of iodides. The ingestion may cause fatal poisoning due to nitrogen retention. Inhalation of iodine vapour acts as an irritant causing a pulmonary edema (Institute of Medicine, 2006; Zimmermann and Boelaert, 2015; Vieja and Santisteban, 2018).

Iodination of common salt is finest approach to get rid of iodine deficiency disorders (IDDs) and associated thyroid diseases (Sawalha *et al.*,

2019). Iodine is an essential dietary element for mammals with the diet being the major source of exposure to iodine for the general human population (Yadav, 2006). Iodine insufficiency during impregnation rise miscarriage rates, lower parturition, and increases infant death rate (Zimmermann, 2011; Pearce *et al.*, 2016). Common salt is iodized by iodate as a source of iodine, in order to prevent iodine deficiency (Vieja and Santisteban, 2018). Further studies suggest that the reduction of iodate to iodide may be microbial enzymatically mediated in soils and water. Its industrial exposure shows lachrymation, burning sensation in eyes. Iodine absorption through various portions of the respiratory tract may result in systematic poisoning in humans, blepharitis, rhinitis, catarrhal stomatitis and chronic pharyngitis. The threshold concentration for iodine is 0.1 ppm or 1

mg/m<sup>3</sup> in the air as adopted by the American Conference of Governmental Industrial Hygienists.

The bioavailability of iodine from food and water is high and absorbed iodine is rapidly distributed. The thyroid gland is the main storage organ and target of iodine toxicity, with exposure to excess iodine leading to hypothyroidism (with or without goitre), hyperthyroidism and changes in the incidence and types of thyroid malignancies. Levels of iodine found in drinking water are generally low. Although higher levels of exposure may occur in specific instances when iodine is used as a drinking water disinfectant, extended periods of exposure are considered unlikely, and a guideline value for iodine is not recommended at this time. Nowadays, pesticide applications containing different chemicals including iodine are a threat to ecosystems (Yadav, 2010). Nanotechnology can also play extremely important role in water pollution management studies (Yadav and Sharma, 2013). Guidance levels for iodine intake differ with age, gender and pregnancy/breast-feeding; social status and occupational health must be taken into consideration (Yadav and Sengupta, 2009).

The molecular iodine is rapidly converted into iodide following ingestion and is efficiently absorbed throughout the gastrointestinal tract. Muscles and eyes also contain high iodide concentrations. Iodine is an essential element in the synthesis of thyroid hormones: thyroxine (T<sub>4</sub>) and triiodothyronine (T<sub>3</sub>) through the precursor protein thyroglobulin and the action of the enzyme peroxidase (Verma, 2017). The highest concentration of iodine in the human body is found in the thyroid, which contains 70-80% of the total iodine content (15-20 mg). Molecular iodine vapour is converted into iodide before absorption. Iodide is excreted primarily by the kidneys and is partially reabsorbed from the tubules following glomerular filtration. Smaller amounts of iodine are excreted in saliva, sweat, bile, and milk. Environmental iodine has relatively close association with pollution. Ecological risk assessment for soil toxicity opens ways for soil management (Yadav and Mishra,

2013) that will ultimately lead to environmental health management to frame better policy for sustainable approach because ecological balance is necessary for the biodiversity, sustainable development and human survival (Ashok, 2017, 2018; Verma, 2019, 2021).

Water and soil samples were collected from Hapur district of Uttar Pradesh during summer season of 2019-2020 *i.e.*, April to July. The samples were collected in clean polythene bottles without any air bubbles as per requirements. The bottles were rinsed before sampling and tightly sealed after collection. All this experimental work was done in the laboratory of Department of Chemistry, SSV College Hapur (C.C.S. University, Meerut). The present work is simple, sensitive and non-catalytic method. Most of the methods are either not sensitive enough, or require complicated and expensive instruments, or time consuming.

#### MATERIALS AND METHODS

A Systronics 106 digital spectrophotometer apparatus was used to measure the absorbance, a Systronics 335 digital pH meter was used for the measurement of pH, and Remi centrifuge for centrifugation. In reagents, all the chemicals used were of International standards grade or the most effective quality on the market. Double distilled deionised water was used throughout the experiment. For stock solution, iodine of 1 mg (E. Merck) was prepared in 30% ethanol. The working standard solution was prepared by the appropriate dilution of stock. For bromine water, a saturated aqueous solution of bromine was prepared daily. The formic acid (50% aqueous solution), potassium iodide (E. Merck) with 0.1 % aqueous solution, 2,8-dimethyl-3,7-diaminophenazine (Schmid and Co., W Germany) with 0.02% aqueous solution and acetate buffer solution (pH 4) were used in the analysis.

#### Analysis Procedure

Aliquots of sample solutions containing 2-13 µg of iodine in a 25 ml calibrated test tube, 0.5 ml of bromine water was added and shaken for two min. The excess of bromine was removed by the drop wise addition of formic acid. To this 1 ml of 0.1 %

potassium iodide was added, followed by addition of 1 ml of 2 M HCl. The mixture was gently shaken for the liberation of iodine. Then, 1 ml of 0.02% 2,8-dimethyl-3,7-diaminophenazine was added, followed by the addition of 2 ml of buffer solution. The content obtained was marked with distilled water and mixed thoroughly. The absorbance of the colored species was measured at 530 nm. The results are summarized in table 3.

### Determination of Iodine

Water samples from different sources and soil samples through random sampling were collected

for analysis. In the iodine test with tap water and soil sample, it didn't show any changes hence a known amount of iodine was added to the samples. These samples were analysed by the proposed and reported method (Pearson, 1976). The recoveries were 97-99% (table 1). 5g of these samples were dissolved in water, shaken thoroughly and filtered. The filtrate was subjected to centrifugation for about 10 min. The supernatant liquid was taken and to it 1 ml of 5% EDTA solution was added. Aliquots were taken and analysed by the proposed and reported method (Pearson, 1976). The results obtained are shown in table1.

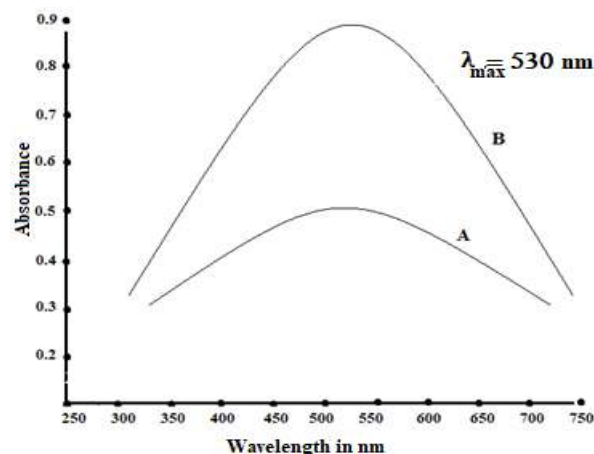
**Table 1: Determination of iodine in tap water and soil.**

Sample	Amount of iodide ( $\mu\text{g}$ )		Total iodide found ( $\mu\text{g}$ )		% of recovery	
	Originally found	Added	Present method	Reported method	Present method	Reported method
Tap water (2ml)						
A	-	4	3.9	3.87	97.5	97
B	-	6	5.9	5.86	98.4	97.88
C	-	8	7.92	7.85	99	98.2
Soil (5g)						
A	-	5	5	4.88	100	99.79
B	-	7	6.99	6.8	99.88	99.12
C	-	9	9	8.88	100	98.23

## RESULTS AND DISCUSSION

### Spectral characteristics

The absorbance of the 2,8-dimethyl-3,7-diaminophenazine with various concentration of iodine against a reference solution showed maximum absorbance at 530nm (Fig. 1). Authors noticed an adherence to the Beer's Law, molar absorptivity and Sandell's sensitivity. The colour reaction was found to obey Beer's law over a concentration range of 2-13  $\mu\text{g}$  of iodine per 25ml of the solution (0.08-0.52ppm) (Fig. 2). The molar absorptivity and Sandell's sensitivity were found to  $1.90 \times 10^5 \text{ l mol}^{-1} \text{ cm}^{-1}$  and  $66.78 \times 10^{-5} \mu\text{g cm}^{-2}$  respectively.



**Fig. 1: Absorption spectra of the dye** (A=Concentration of iodine in  $\mu\text{g}/25\text{ml}$ , B=Reagent blank).

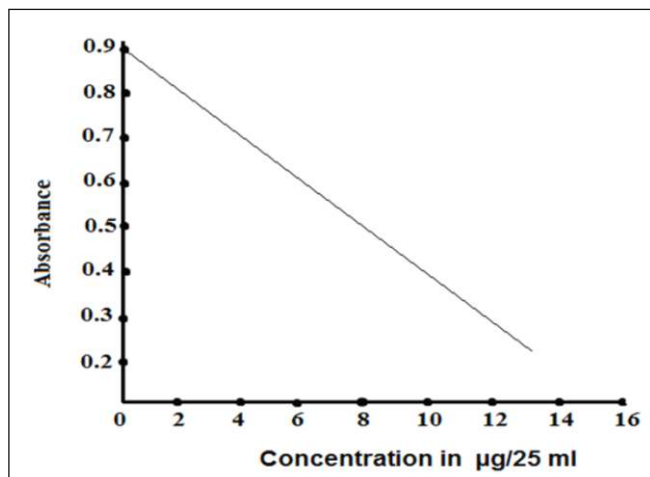


Fig. 2: Calibration curve for the determination of iodine.

#### Effect of reagent concentration

The constant and maximum absorbance was obtained when 0.5ml of bromine water, 1 ml of 0.1 % potassium iodide and 1 ml of 0.05% 2,8-dimethyl-3,7-diaminophenazine was added in the described order. For the removal of excess bromine 1-2 drops of formic acid were sufficient. It was found that on increasing the amount of formic acid, the sensitivity decreased. If the quantity of 2,8-dimethyl-3,7-diaminophenazine and potassium iodide was increased, the absorbance of the solution remained constant.

#### Effect of time, temperature and pH

Normal room temperature was sufficient for this reaction and also 10 min time was needed for completion of the reaction after dilution to 25 ml. Constant and maximum absorbance value was obtained at the pH  $4 \pm 0.2$ . Hence the pH of the reaction system was maintained at  $4 \pm 0.2$  throughout the study. This could be achieved by the addition of 2 ml of acetate buffer solution in a total volume of 25 ml. Under the optimum reaction conditions, the bleached reaction system was stable for a period of more than 4 hours.

#### Precision

The repeatability of the method was checked by analyzing  $6\mu\text{g}$  of iodine in 25ml for a period of seven days. The standard deviation and relative standard deviation were found to be  $\pm 0.0090$  and 1.75% respectively.

#### Effect of foreign species

The validity of the method was assessed by investigating the effect of foreign species in a solution containing  $1\mu\text{g}$  per ml of iodine. The results were given in table 2. Most of the cations like  $\text{Fe}^{+3}$ ,  $\text{Cd}^{+2}$ ,  $\text{Ca}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Zn}^{+2}$ , etc. and anions like  $\text{SO}_4^{-2}$ ,  $\text{PO}_4^{-3}$ ,  $\text{NO}_3^{-}$ , did not interfere under the optimum conditions.

Table 2: Effect of diverse ions in the determination of iodine.

Foreign ions	Tolerance limit in ( $\mu\text{g ml}^{-1}$ )
$\text{Ca}^{2+}$ , $\text{Br}^-$ , $\text{Cl}^-$	2000
$\text{Mn}^{2+}$ , $\text{Mg}^{2+}$ , $\text{Zn}^{2+}$	1500
$\text{Gd}^{3+}$ , $\text{PO}_4^{3-}$ , $\text{Yb}^{3+}$ , $\text{Sm}^{3+}$ , $\text{Eu}^{3+}$	1000
$\text{Cr}^{3+}$ , $\text{NO}_2^-$ , $\text{La}^{3+}$ , $\text{Al}^{3+}$ , $\text{SCN}^-$	500
* $\text{Cr}_2\text{O}_7^{2-}$ , * $\text{Fe}^{3+}$ , * $\text{Ce}^{4+}$ , oxalate, citrate, tartarate	< 1
$\text{MoO}_4^{2-}$ , $\text{AsO}_4^{3-}$ , $\text{Co}^{2+}$ , $\text{WO}_4^{2-}$	< 100

The proposed method has been satisfactorily applied to the determination of iodine in tap water and soil samples. The results obtained are presented in table 1, 2 and 3 are in agreement with those obtained with reported method (Pearson, 1976).

#### CONCLUSION

The proposed method is simple, sensitive and selective. This method is a good alternative for some of the reported costly, sophisticated instrumental methods (Table 3). The developed method needs neither heating for the complete

**Table 3: Comparison of Spectrophotometric method for the determination of iodine with proposed method.**

S.No.	Reagent	Medium /pH	$\lambda$ max. (nm)	Beer's law range (ppm)	Remarks
1.	Starch	Acidic 1N H <sub>2</sub> SO <sub>4</sub>	575	0.2 -0.6	Many oxidants and reductants interfere.
2.	Ceric sulphate and arsenious acid	Acidic 2.5N H <sub>2</sub> SO <sub>4</sub>	420	0.4-1.0	Nitrites, perchlorates, persulphates interfere.
3.	Brilliant green	Acidic 6N-HCl	610	0.1-1.0	Extractive and tri-iodides salts interfere.
4.	Lueco crystal violet (LCV)	Acidic 4.0-4.5 pH	591	0.02 - 0.4	Sensitive, interference of some cations and anions costly reagents used.
5.	Rhodamine-B	Acidic 1.5-2.5 pH	555	0.04-0.4	Sensitive, free from common interference.
6.	2,8-dimethyl-3,7-diaminophenazine (Proposed method).	4.0-4.5	530	0.08-0.52	Extractive, more sensitive and no interference from common cations and anions.

development nor extraction into any organic phase. The method is applicable to the determination in tap water and soil samples.

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