

# QUANTITATIVE ANALYSIS OF NITRITES USING ENVIRONMENTALLY BENIGN PROCDURE

#### M. T. Bachute

Department of Chemistry, K. B. P. Mahavidyalaya, Pandharpur, Dist. Solapur(MS)

**Abstract** : An environmentally benign procedure has been developed for the quantitative analysis of sodium nitrite and potassium nitrite. These nitrites were analysed quantitatively by redox titration method in the presence of biocatalyst . The solution of nitrite was titrated against 0.1 N potassium permanganate solution in the presence of paste of green peas as bio catalyst and little volume of 2N sulphuric acid. The results obtained were comparable with those obtained by routine procedure.

**KEYWORDS** : Quantitative , environmentally , municipal wastes, industrial wastes.

#### Introduction

Nitrite is present at trace level in soil, natural waters and plant and animal tissues. Their presence in water can be a result of water processing or use of nitrite salts as corrosion inhibitors. To surface waters they get from the same sources as nitrates, i.e. in municipal wastes, industrial wastes, mining wastes and with water flowing in from artificially fertilized fields. Human saliva also small amount of nitrite<sup>1</sup>

In pure form nitrite was first prepared by the Swedish Chemist Scheele<sup>2</sup> by heating potassium nitrate at red heat for half an hour [2 KNO<sub>3</sub>  $\longrightarrow$  KNO<sub>2</sub> + O<sub>2</sub>].

Nitrites appear as intermediates in the nitrogen cycle. They are unstable and, depending on conditions, are transformed into nitrates or ammonia. Nitrites are commonly used in preservatives. The sources of ammonium ions in surface waters are reactions of biochemical decomposition of organic nitrogen compounds, reduction of nitrites and nitrates by hydrogen sulfide, iron (II), humus substances (or other reducing compounds) and, first of all, municipal wastes, industrial wastes and animal farm wastes. Nitrogen compounds enhance eutrophication of surface waters. Organic nitrogen compounds undergo biochemical decomposition into nitrites later oxidized to nitrates.<sup>3</sup>With realization of reaction of nitrous acid with aromatic amines to form diazonium ions, nitrites gained importance in development of organic chemistry in 19<sup>th</sup> century.<sup>4</sup>

The basic method for determination of nitrites in water samples, relies on the reaction of nitrites with sulphanilic acid giving diazo compounds, which couples with 1-naphthylamine. The reaction

gives an azo dye of intense red colour. There are other methods that are modifications of that proposed by Griess, e.g.<sup>5,6</sup> that involving the reaction with sulfanilamide and N-(1-naphthyl)-ethylenediamine).  $HPLC^7$  with normal and reversed phase columns should also be mentioned. Ion chromatography has become a standard method for determining anions and cations in water, air and solid samples. In 1984 the American Society for Testing Materials (ASTM)<sup>8</sup> approved it as the standard method for determining anions in water. There are a number of methods for determining NO2 - , NO3 - and NH4 + ions. Determination of these analytes in the sample often poses analytical problems related to low selectivity of the methods and the presence of many interfering factors.

There are reports<sup>3</sup> on therapeutic uses of inorganic nitrites as vasodilator, antitode for cynide and hydrogen sulphide poisoning, antimicrobial agents.

Therefore there is need to develop environmentally benign procedure for quantitative analysis of inorganic nitrites. In view of this literature search revealed that biocatalyst like lipoxygenase enzyme can be used as source of oxygen in redox volumetric analysis. Lipoxygenases are dioxygenating enzymes found in plants<sup>9</sup> like potato tuber, green peas, cucumber etc. and mammals like rats. They bring about hydroperoxide formation fatty acids<sup>10</sup>. Therefore we thought to use plant material containing lipoxygenaseas catalyst in quantitative analysis of sodium nitrite and potassium nitrite.

#### **PRESENT WORK**

In the present work the solution of nitirite was titrated against  $0.1NKMnO_4$  solution in the presence of paste of green peas and little volume of sulphuric acid. It was necessary to heat the mixture prior to titration

#### DETERMINATION OF AMOUNT OF NITRITE IN THE STOCK SOLUTION

Stock solution of nitrite was prepared by dissolving 1.1g of nitrite in 250cm<sup>3</sup> distilled water. <u>Titration using routine procedure<sup>11</sup></u>

To potassium permanganate solution  $(0.1N, 10cm^3)$  in a 500cm<sup>3</sup> conical flask, was added sulphuric acid solution  $(1N, 225cm^3)$ . This solution was heated to 40 to 50<sup>o</sup>C and hot solution was titrated against nitrite solution with constant stirring and slow addition till pink colour disappeared. Tip of the burette was immersed deep in the KMnO<sub>4</sub> solution. Three burette readings were recorded and CBR was recorded as X cm<sup>3</sup>(**Table III**).

1	In Burette	Nitrite solution
2	In conical flask	$10 \text{ cm}^3  0.1 \text{N KMnO}_4 + 225 \text{ cm}^3 1 \text{N H}_2 \text{SO}_4$
3	Indicator	KMnO <sub>4</sub> itself
4	End point	Pink to colourless

Table	Ш

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#### **Burette Readings**

For Sodium nitrite: Pilot Reading : 7.00 to 8.00 cm<sup>3</sup>

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Level	Burette Readings cm <sup>3</sup>			CBR	Amount of
	1	2 3	3	X cm <sup>3</sup>	NaNO <sub>2</sub> found
Final	7.60	7.60	7.60		
Initial	0.00	0.00	0.00	7.60	1.058g
Difference	7.60	7.60	7.60		
For Potassium Nitrite: Pilot reading 11.00 to 12.00cm <sup>3</sup>					

Level	Burette Readings			CBR	Amount of
	1 2	3		X cm <sup>3</sup>	KNO <sub>2</sub> found
Final	11.60	11.60	11.40		
Initial	0.00	0.00	0.00	11.60	1.099g
Difference	11.60	11.60	11.40		

Titration using potato tuber pieces

To potassium permanganate solution  $(0.1N, 10cm^3)$  in a  $100cm^3$  conical flask, was added sulphuric acid solution  $(1 N, 90cm^3)$ . To this solution potato pieces were added and the solution was slowly titrated against nitrite solution with constant stirring till pink colour disappeared. Tip of the burette was immersed deep in the KMnO<sub>4</sub> solution. Three burette readings were recorded and CBR was recorded as Y cm<sup>3</sup> (**Table IV**).

#### Table IV

1	In Burette	Nitrite solution
2	In conical flask	$10 \text{ cm}^3  0.1 \text{N KMnO}_4 + 90 \text{ cm}^3 1 \text{N H}_2 \text{SO}_4 + \text{potato pieces}( 0.5 \text{g})$
		for NaNO <sub>2</sub> and 2.00g for $KNO_2$
3	Indicator	KMnO <sub>4</sub> itself
4	End point	Pink to colourless

## Burette Readings (In the presence of potato pieces)

For sodium Nitrite(Stock solution : 1.1g/250cm<sup>3</sup>) : Pilot Reading 7.00 to 9.00cm<sup>3</sup>

Level	Burette Readings			CBR	Amount of
	1 cm <sup>3</sup>	$2 \text{ cm}^3$	3 cm <sup>3</sup>	Y cm <sup>3</sup>	NaNO <sub>2</sub> found
Final	7.90	7.90	7.80		
Initial	0.00	0.00	0.00	7.90	1.098g
Difference	7.90	7.90	7.80		

# For potassium Nitrite(Stock solution : 1.1g/250cm<sup>3</sup>): Pilot Reading 11.00 to 12.00 cm<sup>3</sup>

Level	Burette Readings			CBR	Amount	of
	$1 \text{ cm}^3$	$2 \text{ cm}^3$	$3 \text{ cm}^3$	X cm <sup>3</sup>	KNO <sub>2</sub> found	
Final	11.20	11.20	11.20			
Initial	0.00	0.00	0.00	11.20	1.1g	
Difference	11.20	11.20	11.20			

#### Calculations

A.	For sodium nitrite		
	Factor used :	$1 \text{ cm}^3$	$1N \text{ KMnO}_4 = 0.0345 \text{ g NaNO}_2$
B.	For potassium	nitrite	
	Factor used	$1 \text{ cm}^3$	$1N \text{ KMnO}_4 = 0.0445 \text{ g KNO}_2$

#### DISCUSSION

Results show that results obtained from both procedure are nearly same. The catalysis of the reaction may be probably due to the lipoxygenase enzyme present in the potato tuber. Lipoxygenase is a dioxygenating enzyme because of which there may be increase in oxygen level in the reaction mixture. Hence reaction occurs at room temperature.

#### Fuel saving:

Important thing in this procedure is that it is not necessary to heat the solution prior to titration which is required in routine procedure. This saves a lot of fuel.

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