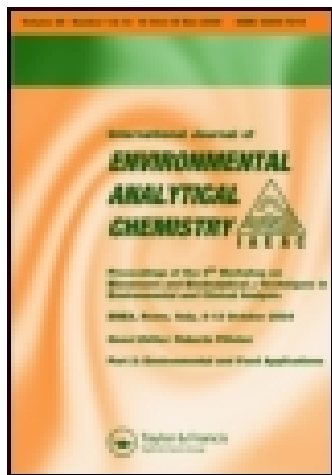


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SPECTROPHOTOMETRIC DETERMINATION OF NIATRITE IN ENVIRONMENTAL SAMPLES USING A NEW REAGENT SYSTEM

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A sensitive and rapid spectrophotometric method has been investigated for the trace determination of nitrite in potable and polluted water. The method is based on the Griess reaction in which nitrite is diazotised with sulphadiazine in acid medium and then coupled with *N*-(1-naphthyl)-ethylenediamine dihydrochloride to give a purple coloured azo dye, having λ_{\max} at 545 nm and a molar absorptivity of $6.9 \times 10^4 \text{ l. mol}^{-1} \text{ cm}^{-1}$. The proposed reagent system has also been used as spot reaction for the qualitative determination of nitrite in effluents (limit of identification, 0.025 μg).

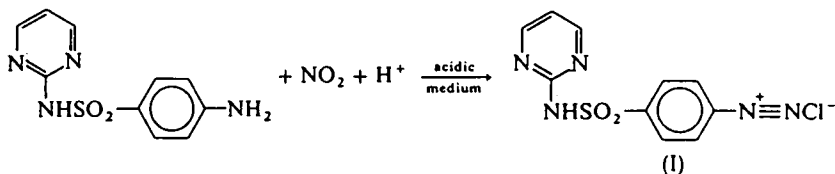
KEY WORDS: Nitrite, water, spectrophotometry.

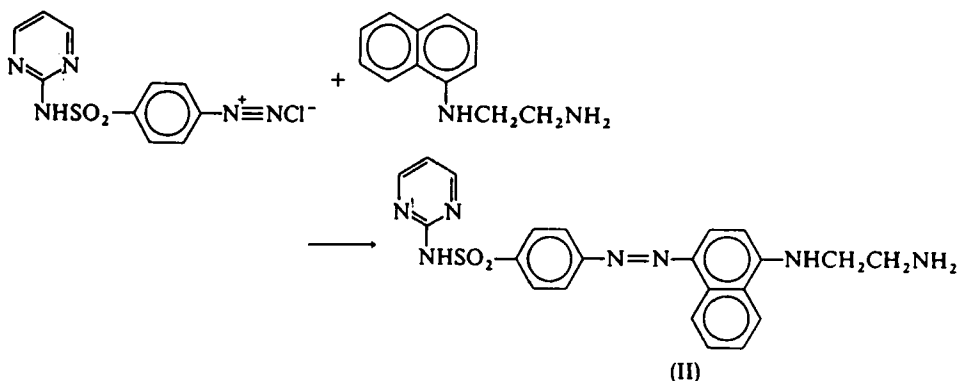
INTRODUCTION

Nitrite in the environment originates from biological denitrification, acid rain and industrial waste. Nitrite induces methemoglobinemia and carcinogenic nitrosoamines can be formed in the body^{1,2}. The maximum permissible limit specified by the U.S. Public Health Service is $0.06 \mu\text{g ml}^{-1}$. Many analytical methods have been reported in the literature; most of these are based on the Griess reaction³⁻⁸.

In this communication a new reagent system using sulphadiazine and *N*-(1-naphthyl)-ethylenediamine dihydrochloride (NEDA) has been used for the spectrophotometric and spot-test determination of trace levels of nitrite. The method is based on diazotisation of sulphadiazine by nitrite in acid medium and subsequent coupling with NEDA to give a purple coloured azo dye. The method is highly sensitive with an $\epsilon_{\max} = 6.9 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$ at $\lambda_{\max} = 545 \text{ nm}$. Two steps are involved in the colour reaction. In the first step sulphadiazine reacts with nitrite in acid medium to form a diazonium ion (I) and in the second step this ion is coupled with NEDA to give a purple coloured azo dye (II):

First Step



Second Step**EXPERIMENTAL**

Apparatus Spectrophotometry was carried out using a Shimadzu UV-Visible recording UV-240 agraphicord, with 1-cm matched silica cells. Calibrated glassware was used for volumetric measurements.

Reagents and solutions All chemicals used were of Analar grade unless stated otherwise.

Standard nitrite solution A stock solution of nitrite was prepared by dissolving 0.15 g of pre-dried sodium nitrite in 100 ml of demineralised water. This solution contains 1 mg NO_2^- /ml. A little chloroform was added as a stabilizer⁹. Working standards were prepared daily by appropriate dilution of the stock solution.

Sulphadiazine solution A 1×10^{-3} M reagent solution was prepared in 2 M hydrochloric acid.

NEDA A 0.02% (w/v) solution of the reagent was prepared in demineralised water.

Hydrochloric acid solution A 5 M hydrochloric acid solution was prepared.

Solutions of diverse ions Solutions of diverse ions were prepared by the method of West¹⁰.

Procedure A sample aliquot containing 2–12 μg of nitrite (0.08–0.48 ppm) was taken in a 25 ml volumetric flask. To this 1 ml of sulphadiazine solution was added and the acidity was adjusted to ca 1 M with hydrochloric acid; 1 ml of EDTA solution was added for masking metal ions. The solution was kept for 2 min at 25°C for complete diazotization. After this, 1 ml of NEDA solution was added for coupling. After 5 min the volume was made up to the mark with 5 M hydrochloric acid. After 10 min the absorbance of the coloured dye was measured at 545 nm against a reagent blank.

RESULTS AND DISCUSSION

Reaction conditions

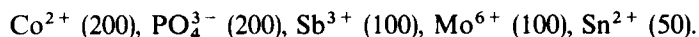
At least 0.1 M hydrochloric acid was found necessary for complete diazotization. Constant absorbance values were obtained over the range of 0.5–3.0 M hydrochloric acid.

The effect of varying the molar ratio of sulphadiazine and NEDA was also studied. For sulphadiazine and nitrite the absorbance was constant for a molar ratio of 1:1. The maximum absorbance was obtained at a nitrite:NEDA ratio of at least 1:10; higher concentrations of NEDA did not affect the absorbance. The colour of the purple dye was more stable, if the final volume was made up to the mark with 5 M hydrochloric acid.

Beer's law was obeyed in the tested range of 0.08–0.40 $\mu\text{g ml}^{-1}$ nitrite. The molar absorptivity for the present system was $6.9 \times 10^4 \text{ l.mole}^{-1} \text{ cm}^{-1}$. The coefficient of variation for the determination of $6\mu\text{g}$ nitrite per 25 ml over a period of 7 days was 0.36%.

Effect of diverse ions

Since the system was developed for the analysis of polluted waters, the interference by foreign ions commonly present in water was studied by adding known amounts of diverse ions to a solution containing $6 \mu\text{g}$ nitrite per 25 ml. The tolerance limits (amount of interferent causing an error of $\pm 2\%$ or less; in ppm) of the diverse ions were found to be as follows:



The interference from sulphites can be masked by adding 1 ml of 10% tetrachloromercurate solution. Cu^{2+} and Fe^{2+} do not interfere.

Application

In order to assess the validity of the method, nitrite was determined in polluted water and tap water. Various amounts of nitrite were added to potable tap water and the method was compared with the standard method using sulphanilamide-NEDA¹¹. The data given in Table 1 shows that the proposed method gives results comparable with the standard method.

Polluted water was collected from the Zuari river (in Goa) and preserved by the addition of mercuric chloride and stored at 0°C. Samples were analysed by the proposed method as well as by the standard method (Table 2); the results are fully satisfactory.

Table 1 Determination of nitrate in tap water ($\mu\text{g}/25$ ml).

Sample No.	Nitrate added	Nitrite found	
		Present method	Standard method
1	1.5	1.48	1.50
2	3.0	3.10	2.90
3	5.0	5.00	5.10
4	7.0	7.05	6.95

Qualitative nitrite test

The proposed reagent system has also been used as spot reaction for the qualitative determination of nitrite in effluents. The test has been carried out on filter paper or in a micro test tube.

With the filter paper, equal volumes of sulphadiazine (1×10^{-3} M) and NEDA (0.02%, w/v) solutions were taken in a dish and strips of filter paper were soaked in the mixed reagent. The papers were allowed to remain in the solution for 30 min with occasional stirring. Next, the strips were dried by exposing them to a stream of warm air issuing from a drying apparatus. After that a drop of the test solution was placed on the dried impregnated paper. A purple colour indicates the presence of nitrite in the sample. The limit of detection is $0.025 \mu\text{g}$.

Alternatively, a drop of test solution was taken in a micro test tube. One drop of sulphadiazine reagent followed by one drop of NEDA reagent were added. A purple colour indicates the presence of nitrite in the sample. The limit of detection is $0.025 \mu\text{g}$, which compares well with reported methods^{1,2}.

CONCLUSION

The sensitivity of the proposed spectrophotometric method is satisfactory. The method is more sensitive than the standard method and other reported methods for

Table 2 Determination of nitrite in polluted water*

Sample No.	Concentration of nitrite (ppm)	
	Present method*	Standard method**
1	0.18	0.19
2	0.23	0.23
3	0.27	0.25
4	0.39	0.38

* Samples were collected from the Zuari river in Goa.

** Mean of three replicate analyses.

Table 3 Comparison of spectrophotometric methods for trace-level nitrite determination.

<i>Reagents</i>	λ_{\max} (<i>nm</i>)	ϵ_{\max} ($l.mol^{-1} cm^{-1}$)	<i>Remarks</i>	<i>Reference</i>
<i>p</i> -Nitroaniline + azuline	515	5.2×10^4	Cu(II) and Fe(II) interfere; acidity should be maintained rigidly, colour is stable for 1 h	13
Sulphanylamide + 1-amino-3-pyridinol	470–485	2.8×10^4	Cu(II), Zn(II), Mg(II), Co(II) and Bi(III) interfere	14
Sulphanylamide + 2-Chloro-3-pyridinol	460	2.1×10^4	<i>Idem</i>	14
<i>p</i> -Amino acetophenone + <i>m</i> -phenylenediamine	450	2.3×10^4	Long standing time an rigorous pH control is necessary; Cu(II) interferes	15
<i>p</i> -Nitroaniline + 8-quinolinol	550	3.8×10^4	Colour stable for 1 day; Fe(II), Cu(II) and sulphide interfere	16
4-Aminosalicylic acid + α -naphthol	520	1.47×10^4	Many metal ions interfere and method is not sensitive	6
<i>p</i> -Aminobenzoic acid + 8-quinolinol	499	3.2×10^4	Cu(II) does not interfere	17
<i>p</i> -Nitrianiline + α -naphthol	610	5.2×10^4	Colour is stable for 1 day	3
<i>o</i> -Nitroaniline + NEDA	645	6.04×10^4	Method is sensitive but Cu(II) and Fe(II) interfere	5
<i>o</i> -Nitroaniline + 8-quinolinol	570	5.85×10^4	Fe(II) and Cu(II) do not interfere	18
Sulphanilamide + NEDA	540	4.6×10^4		11
<i>p</i> -Nitroaniline + guaicol	540	6.2×10^4	Fe(II) and Cu(II) do not interfere	7
<i>p</i> -Amino acetophenone + NEDA	545	4.6×10^4	Fe(II) and Cu(II) do not interfere	8
Sulphadiazine + NEDA	545	6.9×10^4	Method rapid and highly sensitive; Cu(II) and Fe(II) do not interfere	<i>Present method</i>

the spectrophotometric determination of nitrite^{3,5,7,8,11,13–18}. This is shown by the comparison made in Table 3.

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