

Technical paper

Determination of Nitrite and Nitrogen Dioxide by Spectrophotometry After Solid Phase Extraction

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Abstract

A simple method for the determination of nitrogen dioxide using alkaline Ferron as an absorbing medium as well as coupling agent is proposed. Nitrite ion diazotizes p-aminoacetophenone in the absorbing solution and the diazotised p-aminoacetophenone is subsequently coupled with ferron. The orange-red dye was extracted in an amino cartridge, which shows a maximum absorbance at 485 nm, obeys Beer's law in the range of 0.04 to 0.36 $\mu\text{g mL}^{-1}$ of nitrogen dioxide and has a molar absorptivity of $9.40 \cdot 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$. Optimum reaction conditions for diazotization, full colour development and the effect of variables like temperature, time and pH have been studied. Detailed studies to check the collection efficiency and $\text{NO}_2 : \text{NO}_2^-$ stoichiometric ratio has been carried out. The method has been successfully applied for the analysis of nitrogen dioxide in cigarette smoke, scooter exhaust, and workroom air. The method has also been applied for the determination of nitrite in water, soil etc.

Keywords: Spectrophotometer, p-aminoacetophenone, ferron, solid phase extraction, nitrogen dioxide.

1. Introduction

Nitrogen dioxide is one of the most hazardous primary air pollutants present in the environment with considerable toxic effects. There are several natural and anthropogenic sources like lightning, coal combustion in a pulverized fuel combustion plant, steel making, transportation etc., which contribute to higher levels of nitrogen dioxide in the environment. Traffic is estimated to contribute as much as half of the total nitrogen dioxide emission.^{1,2} It is also formed during the reduction of nitric acid, decomposition of nitrite and combustion of nitrogenous materials.³ Cigarette smoke and auto exhaust is also reported to contain significant amount of nitrogen dioxide.⁴ Inhalation of nitrogen dioxide mainly affects the respiratory system, as affinity of hemoglobin for nitrogen dioxide is 300,000 higher than that for oxygen. This affinity drastically reduces the oxygen carrying capacity of the blood.⁵ Short-term industrial exposure to nitrogen dioxide may cause nausea, vomiting, irritation to eyes, nose, respiratory tract, cyanosis, cardiac dilatation and collapse.⁶ In-

door nitrogen dioxide which is produced by gas or other fuels used for heating and cooking was found to exhibit increased respiratory symptoms, decreased pulmonary lung function, respiratory illness, increased incidences of chronic cough, conjunctivitis, bronchitis, and asthma exacerbation.⁷⁻⁸

The significance of nitrogen dioxide as a pollutant has culminated in the development of several analytical methods for its determination. These include GC,⁹ Liquid Chromatography,¹⁰ Laser induced fluorescence,¹¹ Field modulation laser spectroscopy,¹² Tunable diode laser, Droplet method,¹³ amperometric,¹⁴ chromatomembrane cell,¹⁵ fiber optic spectroscopy,¹⁶ piezo-dosimeter,¹⁷ and passive sampling devices¹⁸ etc. The collection efficiency has also been improved by modifying absorbing solution using sodium arsenite²³, triethanolamine,²⁴ and guaiacol.²⁵

A large number of spectrophotometric methods are also reported in the literature for the detection and determination of nitrogen dioxide.¹⁹⁻²² The method is based on the interaction of electromagnetic radiations with matter. When a beam of light is allowed to pass through a transpa-

of the dye was measured at 485 nm. Amount of nitrogen dioxide was deduced from the calibration graph prepared for 0.8 to 8 μg of nitrite after correction with stoichiometric factor i.e. 0.72.

Same method can be used for the determination of nitrite. Known amount of nitrite was taken in 25 mL volumetric flask to it was added 1 mL of p-aminoacetophenone and the solution was kept for 2 minutes with occasional shaking to ensure complete diazotization. 1 mL of EDTA solution was added and then 1 mL of ferron solution was added for coupling and made alkaline with sodium hydroxide. The absorbance of the dye was measured at 485 nm.

Many reagent systems have been studied for the determination of nitrogen dioxide and nitrite such as p-aminoacetanilide, p-nitro aniline, p-aminoacetophenone, 4-nitro 1-naphthyl amine, anthranilic acid, sulphanic acid but p-aminoacetophenone was found to be the most appropriate reagent for coupling with ferron.

2. 3. Procedure for Solid Phase Extraction

The orange-red dye was extracted by passing 25 mL of the dye through an amino cartridge that was pre-conditioned by passing in sequence, 3 mL each of methanol and water. Passing 2 mL methanol eluted the adsorbed dye, which was measured at 485 nm against a reagent blank, which gives negligible absorbance at this wavelength.

3. Results and Discussion

3. 1. Analytical Characteristic

The colour system shows maximum absorbance at 485 nm. (fig. 1) Beer's law was obeyed in the range 0.04 to 0.36 $\mu\text{g mL}^{-1}$; molar absorptivity and Sandell's Sensitivity were found to be $9.40 \cdot 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ and 0.00050

Table 1. Spectral characteristics, precision and accuracy of the presented method.

Parameter	Results.
Stability of colour (hours)	~ 72
λ_{max} (nm)	485
Limit of Beer's law ($\mu\text{g mL}^{-1}$)	0.04 to 0.36
Molar absorptivity ($\text{L mol}^{-1} \text{ cm}^{-1}$)	$9.40 \cdot 10^4$
Limit of detection ($\mu\text{g mL}^{-1}$)	0.010
Limit of quantification ($\mu\text{g mL}^{-1}$)	0.030
Sandell's sensitivity ($\mu\text{g mL}^{-2}$)	0.0005
Standard deviation(\pm)	0.0056
Relative standard deviation (%)	1.29
Error	0.0016
Regression equation ($Y = bx + a$) [*]	
Slope b	2.1328
Intercept a	0.0074
Correlation coefficient r^{**}	0.9959

* Where x is the concentration in $\mu\text{g mL}^{-1}$ ** n = 5

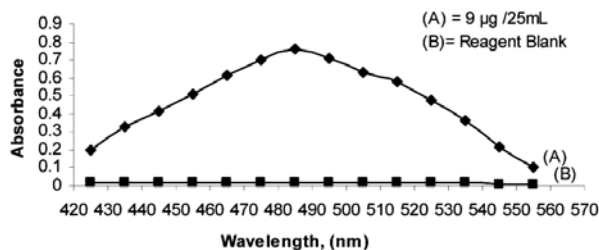


Fig 1: Absorption spectra of the dye

$\mu\text{g cm}^{-2}$ respectively. Six replicate analysis of a 5 $\mu\text{g}/25 \text{ mL}$ nitrite solution following the proposed procedure gave a standard deviation and relative standard deviation of ± 0.0056 and 1.29% respectively. Limit of quantification (LOQ) is evaluated by the relation $10 \sigma/s$ and the limit of detection is $dy 3 \sigma/s$, where σ is standard deviation of the blank with respect to water and s is the slope of the calibration curve. The limit of detection is well below the lower limit of Beer's law. The slope, the intercept, and the correlation coefficient evaluated by least-squares regression analysis are also included. (Table 1) The calibration data for nitrogen dioxide prepared from standard nitrite solution (after correcting with stoichiometric factor) is given in table 2, fig. 2.

3. 2. $\text{NO}_2 : \text{NO}_2^-$ Stoichiometric Ratio

The colour produced by absorbing a given amount of nitrogen dioxide can also be compared with that produced by adding an equimolar amount of nitrite. The ratio of their absorbance value is known as 'stoichiometric factor'. The $\text{NO}_2 : \text{NO}_2^-$ stoichiometric factor for the proposed method has been evaluated and found to be 0.72. (table 2, fig. 2) The stoichiometric factor varied and was lowered if glycol was not used.

The following reaction takes place



Table 2. Evaluation of $\text{NO}_2 : \text{NO}_2^-$ stoichiometric factor Flow rate = 0.75 Lit / min.

No. of analysis	Absorbance due to NO_2 at 485 nm	Absorbance due to NO_2^- at 485 nm	Stoichiometric factor
1	0.093	0.129	0.720
2	0.176	0.244	0.721
3	0.260	0.360	0.722
4	0.350	0.479	0.730
5	0.431	0.599	0.719
6	0.520	0.715	0.727
7	0.611	0.839	0.728
8	0.690	0.949	0.721
9	0.775	1.064	0.728

Mean 72.46 %

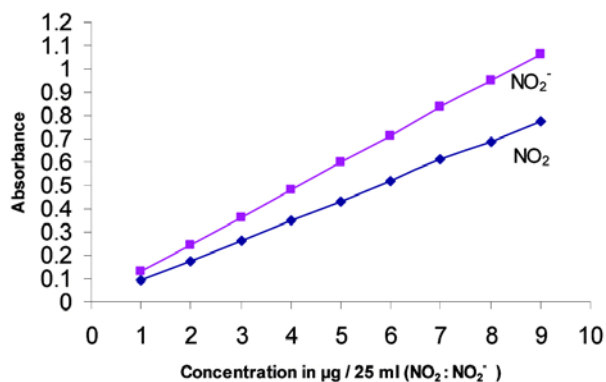


Fig 2: Stoichiometric factor $\text{NO}_2 : \text{NO}_2^-$ Calibration curve for the determination of nitrogen dioxide (extractive system)

Every substance competing with NO_2 for the OH radical will increase the $\text{NO}_2 : \text{NO}_2^-$ stoichiometric factor higher than 0.5.³⁸ In the present investigation alkaline glycol is used for the absorption of the NO_2 .

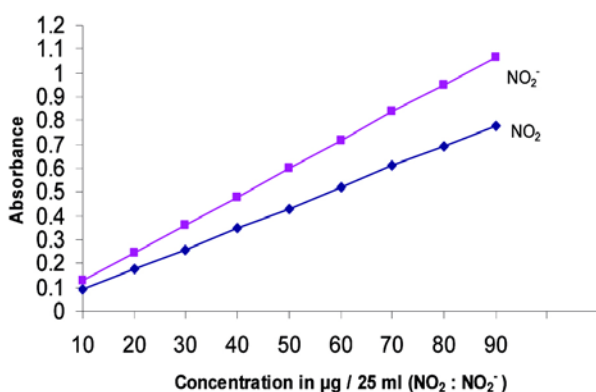


Fig 3: Calibration curve for the determination of nitrogen dioxide (non extractive system)

3. 3. Collection Efficiency

The collection efficiency of the absorbing solution was determined by drawing air containing nitrogen dioxide through two impingers containing absorbing solution, connected in series. After sampling, the NO_2 content of each impinger was analysed separately by the recommended procedure. It was observed that the first impinger has a collection efficiency of 98–99% at a flow rate of $0.75 \text{ lit min}^{-1}$ whereas the second impinger showed negligible absorbance.

3. 4. Effect of pH

The effect of pH on the absorbance of the dye was studied over the range 7.0–11.0. While complete diazotization required 0.1 M hydrochloric acid, the colour started appearing at pH 8. The dye had maximum and constant ab-

sorbance across the pH range of 10–11. Below pH 7 no colour appeared and above pH 11.0 the colour was not stable.

3. 5. Effect of Time and Temperature

Diazotization and coupling required 2 minutes for completion. The resulting dye was stable for ~72 hours which makes the method versatile and useful for field measurement. Maximum absorption was obtained by maintaining the absorbing solution at 4°C .

3. 6. Effect of Foreign Species

The effect of diverse ions commonly found with nitrogen dioxide was studied by adding known amount of diverse ions into the absorbing solution before sampling and then nitrogen dioxide was analysed as given in the procedure. Interferences from sulphur dioxide and heavy metals were masked with H_2O_2 and 10% EDTA respectively. Carbon monoxide, carbon dioxide, ammonia, formaldehyde and benzene which are present in air with nitrogen dioxide had no effect on colour development.

3. 7. Applications

To check the validity of the method it was applied for analysis of nitrogen dioxide and data were compared to those obtained by a reported method. In the reported method absorbing solution containing 8-hydroxyquinoline and sodium hydroxide is proposed for the collection of nitrogen dioxide. The nitrite formed is diazotized with p-nitroaniline in acidic medium which is subsequently coupled with 8-hydroxyquinoline to give purple azoxine dye in alkaline medium.²⁶

3. 7. 1. Determination of Nitrogen Dioxide in Cigarette Smoke

Absorbing system consisting of glass wool (to remove the particulate matter), followed by three midjet impingers, the first two containing acidic potassium permanganate (2.5% KMnO_4 (w/v) in 2.5% H_2SO_4) for oxidation of nitric oxide to nitrogen dioxide and third impinger containing 10 mL of absorbing solution for collection of cigarette smoke was set up. A cigarettes marked to a required length was fixed in a glass holder placed in the tip of suction hole of the first impinger. It was lighted and the air was sucked at a rate of 0.75 L min^{-1} till it burned to the marked length. The collected gas was analyzed for nitrogen dioxide as recommended in the procedure. (table 3)

3. 7. 2. Auto Exhaust

Auto exhaust gases from automobile was drawn through two midjet impingers each containing 5 mL of absorbing solution attached to a suction pump. The gas

was analysed for nitrogen dioxide by the proposed method and the results are shown in table 3.

3. 7. 3. Determination of Nitrogen Dioxide in Work Room Air

Air samples were drawn through two 35 mL calibrated midget impinger containing 5 mL of absorbing solution connected in series, at a flow rate of 0.75 L min⁻¹. Amount of nitrogen dioxide was determined by the proposed method. (table 3)

3. 8. Applications for Determination of Nitrite

3. 8. 1. In Water Sample

River water, pond, and tap water samples were collected from the different regions. Samples were preserved by treating with 2 ml of mercuric chloride (4 µg /100 mL) and stored at 0 °C. They were filtered through whatman filter paper No. 41 before analysis. In order to check the applicability of the proposed method for the determination of nitrite in water, nitrite free tap water was fortified by adding known amount of nitrite. The sample were then

Table 3. Results of analysis of air

Source of sample	Volume of sample taken (Lit) ^b	mg/m ³ present method ^c	Nitrogen dioxide found ^a		F-test	± t-test	Error ^d
			Present method (µg mL ⁻¹)	Reported method ²⁶ (µg mL ⁻¹)			
Cigarette smoke (with filter) ^c	10	0.73	0.36(± 0.026)	0.35(±0.029)	1.24	0.63	0.005
	–	0.71	0.35(± 0.017)	0.35(±0.013)	1.71	0.00	0.000
	–	0.61	0.30(± 0.021)	0.31(±0.019)	1.80	0.87	0.004
Cigarette smoke (without filter)	10	1.4	0.68(± 0.022)	0.67(±0.021)	1.10	0.81	0.004
	–	1.2	0.60(± 0.010)	0.62(±0.008)	1.56	3.82	0.001
	–	1.3	0.65(± 0.022)	0.67(±0.017)	1.67	1.79	0.003
Auto exhaust	10	0.87	0.43(± 0.015)	0.44(±0.014)	1.12	1.20	0.002
	–	0.79	0.39(± 0.019)	0.39(±0.017)	1.24	0.00	0.003
	–	0.91	0.45(± 0.022)	0.46(±0.017)	1.67	0.89	0.003
Work room air (Domestic environment)	10	0.20	0.10(± 0.020)	0.11(±0.018)	1.25	1.91	0.003
	–	0.59	0.29(± 0.011)	0.30(±0.010)	1.21	1.65	0.002
	–	0.30	0.15(± 0.012)	0.12(±0.009)	1.78	4.96	0.002
Work room air (Laboratory)	10	0.20	0.10(± 0.015)	0.11(±0.014)	1.12	1.21	0.002
	–	0.2	0.09(± 0.021)	0.08(±0.020)	1.10	0.85	0.004
	–	0.2	0.09(± 0.020)	0.09(±0.016)	1.56	0.00	0.003

^aMean of three replicate analyses, ^bVolume of air sample taken - 10 Liters, ^cSmoke of one cigarette analyzed, ^dThe F and t value refer to comparison of the proposed method with the reported method. Theoretical value at 95% confidence level t = 2.776. ^eConversion factor ppm to mg/m³ mg/m³ = 46.01(Mol.wt of NO₂) / 22.4 (at NTP) x C [ppm]

Table 4. Determination of nitrite in water and soil

Sample	Nitrite added	Proposed method		Reported method ²⁷	
		*Nitrite found (%)	Recovery (%)	^a Nitrite found (%)	Recovery (%)
^b Riverwater (5 mL)	–	2.5(± 0.006)	–	2.7	–
	–	2.3(± 0.004)	–	2.2	–
	–	2.0(± 0.001)	–	1.9	–
Pond water (5mL)	–	1.7(± 0.026)	–	1.4	–
	–	1.5(± 0.015)	–	1.6	–
	–	1.2(± 0.011)	–	1.0	–
^c Soil	–	0.8(± 0.001)	–	1.0	–
	–	1.5(± 0.010)	–	1.2	–
	–	2.0(± 0.015)	–	2.3	–
Tap water	4	3.90(± 0.026)	97.5	3.91	97.75
	6	5.70(± 0.021)	95.00	5.90	98.33
	8	7.94(± 0.020)	99.25	7.89	98.62

^a Mean of three replicate analyses, ^b Samples were collected from different region of Shvsnath River, which receives effluent from various industries, ^c 5 g of soil sample were taken.

Table 5. Comparison table of nitrogen dioxide

S. No	Reagents/Ref.	Range ($\mu\text{g mL}^{-1}$)	λ_{max} (nm)	Remarks
1	Sulphanilic acid + naphthylamine ²⁸	0.05–1.20	520	Cu^{2+} , Fe^{3+} and strong oxidants
2	8-Hydroxyquinoline PNA ¹⁹	0.07–0.49	570	Extractive, Cu^{2+} , Fe_{3+} are interfere
3	o-Nitroaniline + 1-amino-naphthalene-2-sulphonic acid ²⁹	0.08–0.64	545	Extractive, less sensitive
4	p-Aminoacetophenone + NEDA + oxalic acid ²⁰	0.12–0.96	548	Heavy metals and SO_2 are interfering
5	Potassium iodide + HCl + LCV ³⁰	0.004–0.04	590	Reagents costly
6	p-Aminoacetophenone + Ferron (proposed method)	0.04–0.36	485	Simple, Sensitive, reagents cheap, common ions do not interfere.

Table 6. Comparison table of nitrite

S. No	Reagents/Ref.	Range ($\mu\text{g mL}^{-1}$)	λ_{max} (nm)	Remarks
1	p-rosaniline + NEDA ³¹	0.08–0.72	565	Fe^{3+} , Cr^{6+} and s severely interfere
2	p-Aminophenyl mercaptoacetic acid ³²	0.1–1.6	495	S and Sb^{3+} interfere
3	Sulfathiazole + NEDA ³³	0.054–816	546	Less sensitive
4	p-Aminoacetophenone + NEDA ³⁴	1–0.8	545	Less sensitive
5	o-Nitroaniline + 1-aminonaphthalene-2-sulphonic acid ³⁵	0.08–0.68	545	Less sensitive
6	p-Nitroaniline + phloroglucinol ³⁶	0.004–0.04	420	Cu^{2+} and Fe^{3+} interfere above 75
7	Leucocrystal violet ³⁷	0.004–0.04	590	Reagents costly
8	(proposed work) p-aminoacetophenone + Ferron	0.03–0.4	485	Simple, Sensitive, reagents cheap, common ions do not interfere.

analysed by the proposed and reported method.²⁷ (table 4)

3. 8. 2. In Soil

Soil samples from farmland and roadside were taken, and dried at 55 °C in an oven for 12–16 hours. The dried sample was passed through a 2 mm mesh sieve. Sufficient water (containing 1 or 2 drops of concentrated sulphuric acid) was poured to soak the soil completely. After a few minutes it was filtered, and leached with water. The filtrate was made up to 100 mL and the amount of nitrite was analyzed by the proposed and reported methods. table 4.

3. 7. 4. Comparison With Other Reported Methods

The method has been compared with other reported methods. The advantages of the present method over other methods are summarized in table 5 and 6.

4. Conclusions

In the proposed work a simple, selective and inexpensive solid phase extraction method coupled with spectrophotometry has been developed for the determination

of nitrite and nitrogen dioxide. Although a number of sophisticated techniques like HPLC, GC, CE are available for determination of these pollutants at trace levels, factors such as low cost of instrument, easy handling and almost no maintenance have caused spectrophotometry to be popular technique, particularly in laboratories of developing countries. The sensitivities of various reagents used for the spectrophotometric determination of nitrite and nitrogen dioxide are compared (table 5 and 6) and the method was found to be quite sensitive. Use of solid phase extraction further lead to a ten folds increase in sensitivity.

5. Acknowledgement

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Povzetek

Predlagana je enostavna metoda za določevanje dušikovega dioksida z uporabo Ferrona kot absorpcijskega sredstva in reagenta. Nitritni ion reagira s p-aminoacetofenonom v alkalni absorpcijski raztopini, tako diazotirani p-aminoacetofenon pa nadalje reagira s Ferronom. Nastalo oranžno-rdeče barvilo z maksimumom absorbance pri 485 nm in molarim ekstinkcijskim koeficientom $9,4 \cdot 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ skoncentriramo z ekstrakcijo na trdnem nosilcu. Odvisnost absorbance od koncentracije dušikovega dioksida je linearna v območju 0,04 do 0,32 $\mu\text{g mL}^{-1}$.

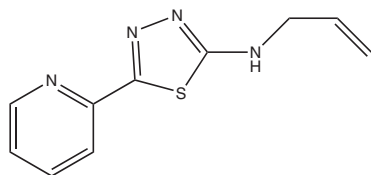
Optimizirali smo reakcijske pogoje diazotiranja in proučili vpliv spremenljivk kot so temperatura, reakcijski čas in pH. Podrobno smo raziskali učinkovitost absorpcije in stehiometrično razmerje pretvorbe dušikovega dioksida v nitrit. Metoda smo uspešno uporabili za analizo dušikovega dioksida v cigaretnem dimu, izpušnih plinih motorjev in delovnih prostorih ter za določevanje nitrita v vodah, vzorcih tal itd.

ERRATA

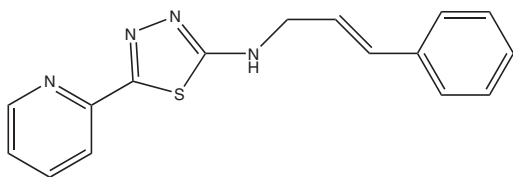
ACSi 2007, 54, 325-335, and 2007, 54, 868-881

In two papers published in the previous issues of Acta Chimica Slovenica, written by Leokadia Strzemecka in *Acta Chim. Slov.* 2007, 54, 325-335, and 2007, 54, 868-881, the correct name of studied compounds in the title and throughout the text should be: *N*-allyl-5-(pyridin-2-yl)-1,3,4-thiadiazol-2-amine for compound **1**, and *N*-cinnamyl-5-(pyridin-2-yl)-1,3,4-thiadiazol-2-amine for compound **2** (see Figure 1).

Figure 1



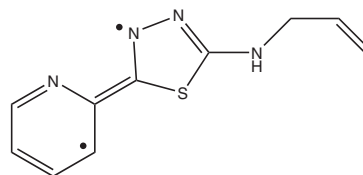
N-allyl-5-(pyridin-2-yl)-1,3,4-thiadiazol-2-amine (**1**)



N-cinnamyl-5-(pyridin-2-yl)-1,3,4-thiadiazol-2-amine (**2**)

In all Figures where the tautomeric and resonance structure of compounds **1** and **2** with one electron (radical) appears, the biradical structure should be read (see Figure 2 for example), and single arrow (\rightarrow) linking different tautomeric structures should be replaced with two reverse arrows (\rightleftharpoons).

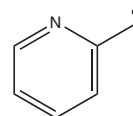
Figure 2



biradical of *N*-allyl-5-(pyridin-2-yl)-1,3,4-thiadiazol-2-amine (**1**)

Corrections of type indicated in Figure 3 (see Figure 6 in *Acta Chim. Slov.* 2007, 54, 868-88, for example) and of similar types should be carried out in all other schemes as well.

Figure 3



2-methylpyridyl radical