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SPECTROPHOTOMETRIC DETERMINATION OF NITROGEN OXIDES IN THE AIR WITH 2-N-ETHYL-5-NAPHTHOL-7-SULFONIC ACID

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For the determination of nitrogen oxides in the air, the structure of diazo and coupling compounds was studied and tested by experiments. The conditions and methods of diazo and coupling reactions were investigated. Furthermore, a spectrophotometric method using sulfanilamide as a diazo compound and 2-N-ethyl-5-naphthol-7-sulfonic acid (N-ethyl J acid) as a coupling compound was proposed. The maximum absorption wavelength of Sulfanilamide-N-ethyl J acid azo compound was at 478 nm. The molar absorptivity was 4.31×10^4 L/(mol · cm) with a recovery of 98.7–100.9% and RSD of 1.85%. For nitrogen oxides, the determinate limit of this measurement was 0.015 mg/m^3 and the determinate range 0.024–2.0 mg/m^3. Moreover, a high degree of correlation was observed between the results obtained by the proposed method and the standard methods. The proposed method can be easily applied to determine nitrogen oxides in the air.

Keywords: *nitrogen oxides, sulfanilamide, 2-N-ethyl-5-naphthol-7-sulfonic acid (N-ethyl J acid), diazo reaction, coupling reaction, spectrophotometry.*

СПЕКТРОФОТОМЕТРИЧЕСКОЕ ОПРЕДЕЛЕНИЕ ОКСИДОВ АЗОТА В ВОЗДУХЕ С 2-N-ЭТИЛ-5-НАФТОЛ-7-СУЛЬФОНОВОЙ КИСЛОТОЙ

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Для определения оксидов азота в воздухе структура диазосоединений отбиралась и тестировалась экспериментально. Исследованы условия и методы реакций диазосоединений. Для определения оксидов азота в воздухе предложен спектрофотометрический метод, в котором сульфаниламид использован в качестве диазосоединения, а 2-N-этил-5-нафтол-7-сульфоновая кислота (N-этил-J-кислота) — в качестве связующего соединения. Максимальная длина волны поглощения азосоединения сульфаниламид-N-этил-J-кислоты 478 нм. Молярная поглощательная способность составляла 4.31×10^4 л/(моль · см) при воспроизводимости 98.7–100.9% и погрешности 1.85%. Предел измерения оксидов азота 0.015 мг/м^3, диапазон измерения 0.024–2.0 мг/м^3. Наблюдалась высокая степень корреляции между результатами, полученными предлагаемым и стандартными методами. Предлагаемый метод может быть легко применен для определения оксидов азота в воздухе.

Ключевые слова: *оксиды азота, сульфаниламид, 2-N-этил-5-нафтол-7-сульфоновая кислота (N-этил-J-кислота), диазореакция, реакция сочетания, спектрофотометрия.*

Introduction. Nitrogen oxides, mainly nitrogen dioxide and nitrogen oxide, are major atmospheric pollutants which pose a potential threat to human health and the environment [1]. Nitrogen oxides come from industrial waste, fuel oil, vehicle exhaust, etc. [2–5]. They contribute to the depletion of the ozone layer [6, 7]. Moreover, nitrogen oxides, in particular nitrogen dioxide, are extremely harmful to human health [8, 9]. Long-term exposure to high concentrations of nitrogen dioxide causes severe respiratory diseases [10–12].

Therefore, measuring the concentration of nitrogen oxides in the air accurately, sensitively, and conveniently is essential for atmospheric environment quality assessment.

There are a number of methods available for the determination of nitrogen oxides: spectrophotometry, chemiluminescence, laser-induced fluorescence, and the electrochemical method. Chemiluminescence methods [13–15] have been widely applied in recent decades. They are based either on the catalytic or photolytic reduction of NO_2 to NO and a subsequent gas-phase reaction with ozone or on the chemiluminescence reaction of NO_2 with an alkaline solution of luminal. However, some of these methods encounter unwanted interference from such species as SO_2, H_2S, CO_2, and O_3. In addition, complex and costly tools are required. Laser-induced fluorescence [16–18] is a direct spectroscopic technique to measure NO_x. Molecules of NO or NO_2 are selectively excited through one of their molecular transitions with the use of a narrow-band laser. The fluorescence is detected with a photomultiplier using optical filters to reject non-fluorescent radiation. This method has high sensitivity and a low determinate limit, but its cost is very high and the system is complicate.

Also, a lot of studies are carried out for the development of electrochemical sensors for nitrogen oxides [19, 20]. The operation principle for electrochemical sensors is an electrochemical cell using a solid electrolyte. Solid electrolytes are suitable for high-temperature electrochemical devices and have shown a remarkable potential for many applications in industry and automotive applications. However, cross sensitivities, long-term instabilities, and long sensor response times are the common disadvantages of many existing NO_x sensors.

With the advantages of simplicity, rapidness, and high sensitivity, spectrophotometry is an effective and convenient method. NO_x is usually absorbed in a suitable solution and then converted into nitrite, followed by the determination of nitrite, in which diazo and coupling reactions are often employed. In acids, the diazo reaction is specific to the nitrite and primary aromatic amine. The reaction has the advantages of having low interference, high sensitivity, and excellent selectivity [21]. Nowadays the standard Griess–Saltzman reaction [22] is the most used method for the determination of nitrogen oxides. Diazo and coupling reactions are carried out in the same medium. This standard method is easily operated. However, N-(1-napthyl) ethylenediamine hydrochloride, the coupling compound, is carcinogenic and seriously toxic [23]. Therefore, it is necessary to exploit new highly sensitive chromogenic agents. As for the oxidation, in the standard method ASTM D 3608-1995 [24], NO is oxidized to NO_2 by CrO_3 and then absorbed with NO_2 in the air in one solution. This method can only detect the total nitrogen oxides concentration but not the NO and NO_2 concentrations separately. At the same time, another new Chinese standard method HJ 479-2009 [25] proposes a new kind of oxidation in an agent-potassium permanganate solution. This method can determine the NO and NO_2 concentration separately at once. Furthermore, potassium permanganate is greener for the environment than CrO_3.

This paper selected and tested the structure of diazo and coupling compounds by many experiments. Meanwhile, the conditions and methods of the diazo reaction and the coupling chromogenic reaction as well as the accuracy and precision of measurement were investigated. A new accurate, sensitive, rapid, and convenient measurement for the determination of nitrogen oxides in the air was established.

Materials and methods. *Reagents and equipment.* This study utilized analytical reagent grade chemicals. Double-distilled water was used throughout the experiments to dilute the reagents and samples. Sodium nitrite, hydrochloric acid, sodium carbonate, sodium hydroxide, potassium bromide, 2-amino-5-naphthol-7-sulfonic acid (J acid), 4-amino-5-hydroxynaphthalene-2, 7-disulfonic acid (H acid), sulfanilamide, potassium permanganate were purchased from the Sinopharm Chemical Reagent Co., Ltd.

Stock sodium nitrite solution (1.00×10^{-2} mol/L) in a 250 mL volumetric flask was prepared by dissolving 0.173 g of sodium nitrite in distilled water. Working sodium nitrite standard solution (1.0×10^{-4} mol/L) was prepared by diluting 1.0 mL of stock sodium nitrite solution to 100 mL with distilled water. N-ethyl J acid was synthesized by J acid and bromoethane in acetonitrile solvent. Acid potassium permanganate solution (0.160 mol/L) was prepared by dissolving 25.0 g of potassium permanganate with 500 mL of distilled water and then adding 500 mL of 1 mol/L sulfuric acid solution to it. The solution was stored in an amber laboratory bottle. The absorber was prepared by adding 5.0 mL of sodium hydroxide solution (2.0×10^{-2} mol/L) and 1.0 mL of potassium bromide solution (0.2 mol/L) into a dry fritted bubbler.

For the experiment we used a 723N Spectrophotometer (Purkinje General Instrument Corporation, Beijing, China), a QC-2 Air-Metering Device (Municipal Institute of Labour Protection, Beijing, China), and an FE20 Precision Acidity Meter (Mettler-Toledo Instrument Corporation, Shanghai, China).

General procedures. A 1.0 mL portion of potassium bromide solution (0.2 mol/L), 0.5 mL of sulfanilamide solution (1.0×10^{-2} mol/L), 0.5 mL of hydrochloric acid solution (1.5 mol/L), and 3.0 mL of distilled water were added into a 10 mL volumetric flask. Then sodium nitrite standard solution was slowly added, and the whole shaken thoroughly and kept at room temperature (25°C) for 3 min. The mixture was transferred into another 10 mL volumetric flask, containing 1.0 mL of sodium carbonate solution (2.0 mol/L) and 0.5 mL of N-ethyl J acid (J acid, H acid) solution (1.0×10^{-2} mol/L). The volumetric flask was filled with distilled water to the mark, and then the mixture was shaken thoroughly and left for 5 min. Using distilled water as a reference, we measured the absorbance in 1.00 cm quartz cells at 478, 480, and 530 nm, respectively.

Determination of nitrogen oxides in the air. A 6.0 mL portion of absorbing reagent was added into two dry fritted bubblers, respectively. Then 5–10 mL of acid potassium permanganate solution was added into an oxidation tube and linked between two dry fritted bubbles by a silicone rubber tube. Air was drawn through a dry fritted bubbler at a rate of 0.4 L/min for 10–60 min to sufficiently develop the final color. The total air volume was noted. The air temperature and pressure were also measured and recorded. After the sampling, the absorbing reagent was analyzed according to the general procedures.

Results and discussion. *Selection of diazo and coupling compounds.* The authors selected the structure of diazo and coupling compounds. Aromatic amino compounds were selected as diazo compounds. Moreover, a sulfo group was introduced to increase water solubility and electron-withdrawing property. Coupling compounds should be aromatic compounds with a strong electron-donating group, such as hydroxyl or amino. In the molecular structure, the coplanar and big conjugate system has a strong light-absorbing ability. Furthermore, in the conjugated system of azo compounds, electrons flow from coupling compounds to diazo compounds through the nitrogen and nitrogen double bond. Some substituent groups promoting the PI electron cloud in the whole conjugated system moving from coupling compounds to diazo compounds in one way could greatly improve the sensitivity of the method.

Among coupling compounds, J acid has higher sensitivity [26]. However, J acid has the tendency to start the self-coupling diazotizing reaction. Therefore, in order to overcome this, the authors carried out the derivation reaction of the primary amino group in the naphthalene ring and made some derivatives with the secondary amino group. The molar absorptivity of the azo compounds was determined, and the results are given in Tables 1 and 2. According to the experimental data, the molar absorptivity of the sulfanilamide-N-ethyl J acid azo compound reached up to 4.31×10^4 L/(mol · cm). Its molecular formula is shown in Fig. 1.

TABLE 1. Selection of Coupling Compounds in the Acid Medium (pH 3.5)

Diazo compound	Coupling compound	λ_{max}, nm	ε, L/(mol · cm)
Sulfanilamide	J acid	480	2.54×10^4
Sulfanilamide	N-ethyl J acid	478	9.82×10^3
Sulfanilamide	H acid	530	2.25×10^4
p-Aminobenzenesulfonic acid	J acid	480	1.86×10^4
Aniline	J acid	480	1.02×10^4
Aniline	N-(1-napthyl)ethylenediamine hydrochloride	550	2.27×10^4

TABLE 2. Selection of Coupling Compounds in the Alkaline Medium (pH 9.6)

Diazo compound	Coupling compound	λ_{max}, nm	ε, L/(mol · cm)
Sulfanilamide	N-ethyl J acid	478	4.31×10^4
Sulfanilamide	N-phenyl J acid	495	4.27×10^4
Sulfanilamide	Di-J-acid	500	4.19×10^4
Sulfanilamide	J acid	480	4.10×10^4
Sulfanilamide	H acid	530	3.18×10^4
p-Aminobenzenesulfonic acid	N-ethyl J acid	480	4.17×10^4
p-Aminobenzenesulfonic acid	N-phenyl J acid	495	4.12×10^4
p-Aminobenzenesulfonic acid	Di-J-acid	500	4.05×10^4
p-Aminobenzenesulfonic acid	J acid	480	4.01×10^4
p-Aminobenzenesulfonic acid	H acid	530	2.40×10^4
Aniline	N-ethyl J acid	486	3.22×10^4
Aniline	J acid	480	3.63×10^4
Aniline	H acid	530	2.15×10^4
2-naphthylamine-1-sulfonic acid	N-ethyl J acid	480	2.89×10^4
1,8-amino naphthalene sulfonic acid	N-ethyl J acid	515	9.89×10^3

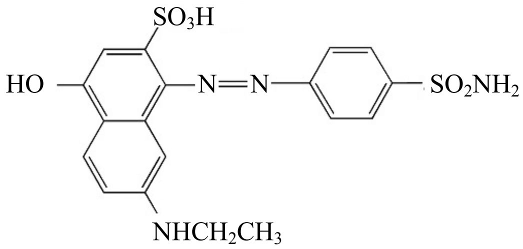

Fig. 1. Structure of sulfanilamide-N-ethyl J acid azo compound.

Absorption spectrum. The absorption spectra of the azo compounds, formed by the diazonium salt of sulfanilamide with N-ethyl J acid, J acid, and H acid, are shown in Fig. 2. The wavelengths of 478, 480, and 530 nm were found to be optimum for getting the best result, respectively.

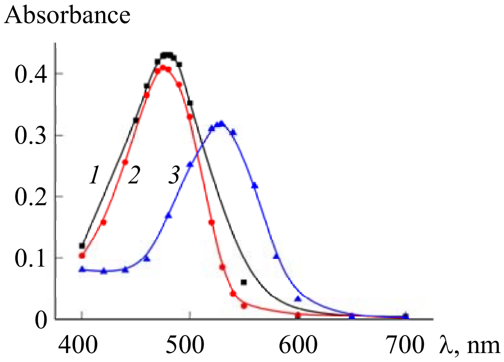

Fig. 2. The absorption spectrum of azo compounds, sulfanilamide-N-ethyl J acid (1), sulfanilamide-J acid (2), sulfanilamide-H acid (3).

Effect of the pH on the diazo reaction. According to the experimental method, the effect of the pH on the diazo reaction was tested. The results showed that when the pH of the solution was between 0.5 and 1.5, the absorbance was maximum and stable (Fig. 3a). Hence, 0.5 mL of 1.5 mol/L hydrochloric acid was chosen in the experiment. The sodium nitrite can easily and fully react with aromatic primary amine because of the fast reaction rate in a low pH. Furthermore, low pH levels can increase the stability of the diazonium salt. However, a pH value less than 1.0 influences the speed of the diazo reaction.

Effect of the pH on the coupling reaction. According to the experimental method, the pH of the coupling reaction was chosen. Figure 3b shows that the absorbance increased with the rise of the pH. When the pH rose within the range 8.0–10.0, the absorbance of the azo compound was maximum and stable. Coupling in an alkaline medium helped to form an anion of hydroxyl in the naphthalene of N-ethyl J acid. However, when the pH was more than 10.0, diazoate converted to inert trans-diazoate and did not couple. Therefore, 1 mL of 2 mol/L sodium carbonate solution was chosen in the study.

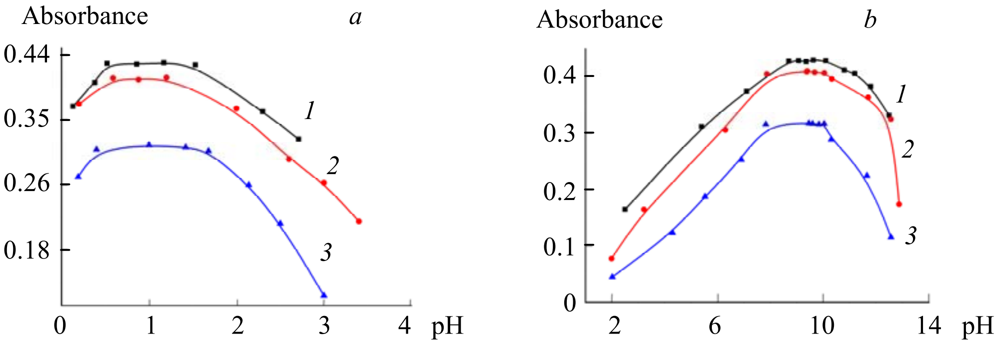

Fig. 3. The pH effect on the diazo (a) and coupling (b) reaction, sulfanilamide-N-ethyl J acid (1), sulfanilamide-J acid (2), sulfanilamide-H acid (3).

Effect of sulfanilamide and N-ethyl J acid concentrations. The effect of varying concentrations of sulfanilamide and N-ethyl J acid on the color intensity and stability was studied by adding sulfanilamide and N-ethyl J acid solution respectively in the range 0.01–1.0 mL (1.00×10^{-2} mol/L) to a series of fixed nitrite concentrations in 10 ml volumetric flasks. It was found that the maximum intensity and stability of the coloration formed were obtained with 0.5 ml of 1.00×10^{-2} mol/L sulfanilamide and N-ethyl J acid solution.

Effect of time and temperature on the stability of the color. Because the sulfonyl group contained in sulfanilamide is an electron-withdrawing group, the speed of the diazo reaction was relatively fast. In order to further accelerate the speed of the diazo reaction, potassium bromide was added as a catalyst. A 1 mL portion of 0.2 mol/L potassium bromide was added into the diazo reaction. According to the experimental method, the effect of time was investigated on the sulfanilamide diazotization. The results showed that the most stable and highest absorbance was within 3–20 min. Thus, 3 min was selected for the diazo reaction in the experiment. In the coupling reaction, the maximum color intensity of the azo compounds was obtained after 5 min and remained stable for 1 h.

No variation of the absorbance values of the color was observed within the temperature range 0–30°C. Hence, the reactions were carried out at room temperature (25±5°C).

Interferences studies. The effects of coexistent ions on the determination of 1×10^{-5} mol/L nitrite were investigated to evaluate the selectivity of the procedure. The tolerance level, which is defined as the relative error produced by foreign species, did not exceed ±5%. The coexistent ions, such as SO_2, CO_2, and NH_3 (added in the form of SO_3^{2-}, CO_3^{2-}, and NH_4^+ salts) were tested at 5–10-fold concentration levels based on their concentrations in the air, and no influence was observed.

Linearity and detection limit. Under optimum conditions, based on plotting the observed data versus the nitrite concentration, a graphical calibration curve was obtained. The calibration curve is a straight line passing through the origin. The results are shown in Table 3. The equation is

$$A = 0.937c + 2.30\times10^{-3},$$

were A is the measured absorbance and c is the concentration of nitrite (mgNO_2^-/L). The response was linear in the concentration range from 2.00×10^{-3} to 0.600 mol/L, and the correlation coefficient was 0.999. Correspondingly, for nitrogen oxides, the determinate limit of the measurement was 0.015 mg/m^3 and the determinate range 0.024–2.00 mg/m^3.

TABLE 3. Calibration Graph

Methods	Regression equation, c(mgNO_2^-/L)	R^2	Linear range, mgNO_2^-/L	ε, L/(mol · cm)
Sulfanilamide-N-ethyl J acid	$A = 0.937c+2.30\times10^{-3}$	0.9995	~0.002–0.600	4.31×10^4
Sulfanilamide-J acid	$A = 0.891c-4.60\times10^{-3}$	0.9995	~0.003–0.700	4.10×10^4
Sulfanilamide-H acid	$A = 0.691c+1.10\times10^{-3}$	0.9996	~0.0045–0.900	3.18×10^4
HJ 479-2009	$A = 0.919c+2.50\times10^{-3}$	0.9992	~0.002–0.600	4.22×10^4

Application of the method. In order to assess the suitability of the proposed method, it was applied for the determination of nitrogen oxides in the atmosphere. The concentration of nitrogen oxides can be calculated using the following formula:

$$V_0 = VP/103.1\times298.15/T, \quad (1)$$

where V_0 is the volume of air under standard conditions, V is the measured volume of air, P is the average atmospheric pressure, kPa, T is the average temperature of the air sample, K, 101.3 kPa is the pressure of the standard atmosphere, and 298.15 K is the temperature of the standard atmosphere.

The concentration of NO_x in the sample is calculated as follows:

$$\rho_{NO_2} = (A_1 - A' - a)V'D/(bfV_0), \quad (2)$$

$$\rho_{NO} = (A_1 - A' - a)V'D/(bfV_0K), \quad (3)$$

$$\rho_{NO_x} = \rho_{NO_2} + \rho_{NO}, \quad (4)$$

where A_1 and A_2 is the absorbance of samples No. 1 and 2, A' is the reagent blank absorbance, a is the intercept of the standard curve, b is the slope of the standard curve, absorbance mL/μgNO_2^-, V' is the sample reagent volume, mL, V_0 is the sample volume corrected to 25°C and 101.3 kPa, K is the oxidation factor of NO to NO_2, 0.68, D is the dilution factor of the sample reagent, and f is the conversion factor of NO_2 to NO_2^-, 0.85.

The results obtained by the proposed method were compared statistically with the results of the standard methods [25, 27] in terms of t-test and F-test. The results are listed in Table 4, sampled in the University of Shanghai for Science and Technology (flow rate 0.4 L/min, sampling time 60 min, $P = 102.9$ kPa, $T = 293.15$ K). It can be seen that the difference between the results is insignificant. According to the standard addition method, Table 5 shows that the recommended measurement recovery was 98.7–100.9% with RSD ($n = 8$) of 1.85%.

TABLE 4. The Results of Determination

Methods	Sulfanilamide-N-ethyl J acid	Sulfanilamide-J acid	Sulfanilamide-H acid	HJ 479-2009	ISO 7996-1985
C_{NO}, $mgNO_2/m^3$	0.0576	0.0587	0.0581	0.0565	0.0571
C_{NO_2}, $mgNO_2/m^3$	0.0352	0.0370	0.0349	0.0346	0.0348
C_{NO_x}, $mgNO_2/m^3$	0.0928	0.0937	0.0898	0.0911	0.0919

TABLE 5. The Results of Recovery Experiment

Methods	Measuring object	Nitrite found, $mgNO_2^-/L$	RSD, % $n = 8$	Nitrite added, $mgNO_2^-/L$	Total nitrite found, $mgNO_2^-/L$	Recovery, %
Sulfanilamide-N-ethyl J acid	NO_2	0.0702	1.85	0.230	0.301	100.4
				0.460	0.524	98.7
	NO	0.0766		0.230	0.306	99.6
				0.460	0.541	100.9
HJ 479-2009	NO_2	0.0709	2.06	0.230	0.298	98.7
				0.460	0.533	100.6
	NO	0.0785		0.230	0.308	99.7
				0.460	0.540	100.4

Conclusion. The proposed method requires low cost equipment, and the reagents are green for the environment and health. N-ethyl J acid is a sensitive coupling compound. It is easily soluble in water and has low volatility. Moreover, its toxicity is much lower than that of N-(1-napthyl) ethylenediamine hydrochloride. The whole reaction lasts for only 8 min, and it could be carried out at room temperature.

It was found that the maximum absorption wavelength of the Sulfanilamide-N-ethyl J acid azo compound was at 478 nm, and the molar absorptivity was determined to be 4.31×10^4 L/(mol · cm) with a recovery of 98.7–100.9% and RSD of 1.85%. The determinate limit of measurement was 0.015 mg/m^3 for nitrogen oxides, and the determinate range was 0.024–2.000 mg/m^3 for nitrogen oxides. Moreover, a high degree of correlation was observed between the results obtained by the proposed method and the standard methods. The proposed method has distinct advantages: simplicity, rapidness, sensitivity, and selectivity. It can be easily applied for the determination of nitrogen oxides in the air.

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