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Ion chromatography of nitrite at the ppb level with photometric measurement of iodine formed by post-column reaction of nitrite with iodide

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Abstract

The difficulty in ion-chromatographic determination of nitrite in aqueous solutions containing a high concentration of chloride arises mainly from incomplete resolution of the peaks for these anions on the separation column whose efficiency is not high. A photometric measurement of iodine formed by a reaction of nitrite with iodide has been found to make it possible to determine, chromatographically, trace amounts of nitrite without any interference from chloride; chloride does not oxidize iodide to produce iodine. The proposed method was based on the separation of nitrite from matrix anions on a silica-based anion-exchange column with a $1.5 \cdot 10^{-3}$ M phthalate eluent (pH 5.0), followed by photometric measurement of the iodine (as triiodide) formed via a post-column reaction of the separated nitrite with iodide. The optimal conditions for the post-column reaction were established by varying the concentrations of iodide, copper(II) and nitric acid in a post-column-reaction solution and the length of a reaction tube. A calibration graph for nitrite, plotted as peak heights versus concentrations, was linear up to $1.50 \cdot 10^{-5}$ M (690 ppb). The detection limit, defined at $S/N=3$, was $1.00 \cdot 10^{-7}$ M (4.60 ppb) nitrite. The presence of chloride ions up to 0.01 M did not give any interference to the determination of nitrite. This method was successfully applied to the determination of nitrite in lake water, river water, sewage works water and snow samples without any pretreatment. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Detection, LC; Water analysis; Environmental analysis; Nitrite; Inorganic anions; Iodine

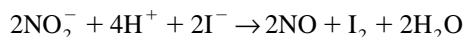
1. Introduction

Nitrite is produced during the biodegradation of organic substances containing nitrogen, and the reaction of nitrite with secondary amines affords carcinogenic nitrosamino compounds [1]. Therefore, there has been growing concern about the sensitive determination of nitrite in natural waters. Various methods of determination such as, spectro-

photometry [2–5], fluorometry [6–8], flow-injection analysis [9–13] and ion chromatography [14–24] have been published. These chromatographic techniques were based on the complete separation of trace amount of nitrite from large amount of chloride using a highly efficient column and the detection of nitrite with each photometric [14,16,18–24], electrochemical [18,22] or conductivity [23] detector. When the efficiency of the separation column is not high, ion chromatographic determination of nitrite often becomes difficult in the presence of a large excess of chloride because nitrite elutes shortly after chloride and the nitrite peak overlaps partially with the large peak of chloride. A highly efficient column such as a

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Dionex IonPac AS12A (number of theoretical plates (N) > 5000, height equivalent to a theoretical plate (HETP) < 0.004 mm) is used for resolution of several anions. However, the efficiency of the column is usually decreased by chromatographic runs over a long period of time. This decrease in column efficiency causes incomplete separation of trace amount of nitrite from a high concentration of chloride. In this case, an additional detection technique is desirable for the selective determination of the nitrite separated incompletely from a high concentration of chloride. We have previously reported ion chromatographic methods for the sensitive and/or selective determination of sulfide, sulfite, thiosulfate and L-ascorbic acid [25,26]; they were based on the photometric measurement of excess iodine (as triiodide) due to its post-column reaction with each species separated on the separating column. On the other hand, iodine is known to be produced by the reaction of nitrite with iodide in an acidic medium according to following equation:



If photometric measurement of the iodine formed was utilized as a detection technique in ion chromatography, nitrite in the presence of large amounts of chloride can be expected to be selectively determined, even when both ions are not separated completely. However, no consideration has been given in the literature to chromatographic determination of nitrite using its pre-separation from matrix on the column and photometric measurement of the iodine (as triiodide).

In this work, nitrite in the effluent from a silica-based separating column using a phthalate eluent was detected by the photometric measurement of iodine (as triiodide) formed via a post-column reaction of the nitrite with iodide. The proposed method gave a higher sensitivity for the determination of nitrite as compared with earlier chromatographic methods [14,15,18,20,23] and also permitted the presence of a higher concentration of chloride (up to 0.01 *M*) than previous methods [14,16,21,24]. This method has been applied successfully to the determination of nitrite in lake water, river water, sewage works water and snow samples without any pretreatment.

2. Experimental

2.1. Apparatus

The chromatographic system used in this study comprised a Model LIC-10-P1 pump with dual pistons (Denki Kagaku Keiki, Tokyo, Japan), a Model SVI-6U7 sample injection-valve (Sanuki Industry, Yamagata, Japan) equipped with a sample loop of 50 μl , a LIC-10SA1 silica-based anion-exchange column (5 cm \times 4.6 mm I.D., Denki Kagaku Keiki) and a Model LIC-10-U1 photometric detector (Denki Kagaku Keiki). A Model CDD-6A conductivity detector (Shimadzu, Kyoto, Japan) was also used instead of the photometer. Another pump (Model LIC-10-P1) was used to allow a post-column-reaction solution to flow through a liquid mixing tee, in which the post-column-reaction solution was mixed with the effluent from the separating column. A Model U-135 recorder (Shimadzu) was used to record the chromatograms.

2.2. Chemicals

The water used in this study was distilled twice and then deionized with a Model Milli-QII instrument (Nippon Millipore, Yonezawa, Yamagata, Japan). All of the chemicals were of analytical reagent grade and were used without further purification.

A 0.025 *M* sodium phthalate solution was prepared by adding 500 ml of 0.05 *M* phthalic acid to 250 ml of 0.2 *M* sodium hydroxide and diluting to 1 l. A 0.025 *M* sodium hydrogenphthalate solution was obtained by mixing 500 ml of 0.05 *M* phthalic acid with 250 ml of 0.1 *M* sodium hydroxide and diluting to 1 l. A $1.5 \cdot 10^{-3}$ *M* phthalate eluent (pH 5.0) used in Section 2.3 was prepared by mixing sodium hydrogenphthalate ($1.5 \cdot 10^{-3}$ *M*) with sodium phthalate ($1.5 \cdot 10^{-3}$ *M*) in an equal volume ratio. The eluent obtained was filtered through a membrane filter (pore size, 0.2 μm) before use.

A standard nitrite solution (0.01 *M*) was prepared by dissolving 0.3503 g of sodium nitrite (98.5%), dried in a desiccator containing silica gel, in water containing 50 ml of 0.125 *M* sodium hydroxide as a stabilizer [27,28] and diluting to 500 ml with water.

This standard solution proved to be stable; no measurable concentration change was found in this work even after six months when it was stored at room temperature in the dark. Working nitrite solutions were prepared by suitable dilution of the standard solution with water before use.

A post-column-reaction solution for nitrite was prepared by adding 6 ml of 0.1 M potassium iodide to 100 ml of a 1.0 M nitric acid– $1.0 \cdot 10^{-4}$ M copper(II) nitrate solution and diluting to 200 ml with water; water used for the preparation was deaerated with nitrogen gas.

2.3. Recommended procedure

A $1.5 \cdot 10^{-3}$ M phthalate eluent (pH 5.0) was pumped at a flow-rate of 0.8 ml min^{-1} , and then a 50 μl aliquot of a sample solution containing nitrite was injected to the separating column. The effluent from the column was allowed to flow through a mixing tee. Moreover, a post-column-reaction solution [$3 \cdot 10^{-3}$ M KI–0.5 M HNO_3 – $5 \cdot 10^{-5}$ M $\text{Cu}(\text{NO}_3)_2$] was caused to flow at a rate of 0.8 ml min^{-1} through the mixing tee in order to mix with the effluent. The mixture obtained was passed through a reaction tube (5 m \times 0.5 mm I.D.), in which the nitrite oxidized the iodide to produce iodine. The absorbance of the iodine (as triiodide) formed in the stream was continuously measured with the photometer at 350 nm, and then chromatograms was recorded.

3. Results and discussion

3.1. Selection of eluent

Three eluents, phthalate, benzoate and sulfanilate, were studied for resolution of various anions containing nitrite ion on the LIC-10SA1 separating column. Chromatograms of anions in their mixtures are shown in Fig. 1. In these cases, concentration and pH of each eluent were adjusted to give optimum resolution for the anion mixtures and anions in the effluent were monitored with photometric detector. A $1.5 \cdot 10^{-3}$ M phthalate eluent (pH 5.0) resolved eight anions in a mixture at retention times within 10 min

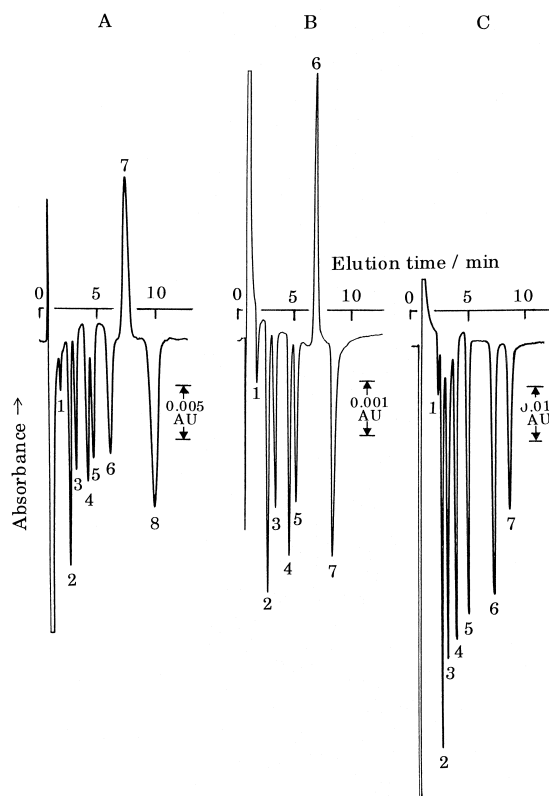


Fig. 1. Separations of various anions in a mixture using eluents of phthalate, benzoate and sulfanilate. Eluents were used at a flow-rate of 0.8 ml min^{-1} . (A) Obtained with a $1.5 \cdot 10^{-3}$ M phthalate eluent (pH 5.0) and detection at 260 nm. Peaks; 1, IO_3^- ($1 \cdot 10^{-4}$ M); 2, Cl^- ($1 \cdot 10^{-4}$ M); 3, NO_2^- ($1 \cdot 10^{-4}$ M); 4, Br^- ($1 \cdot 10^{-4}$ M); 5, NO_3^- ($1 \cdot 10^{-4}$ M); 6, succinate ($1 \cdot 10^{-4}$ M); 7, sulfanilate ($2.5 \cdot 10^{-5}$ M); 8, SO_4^{2-} ($1 \cdot 10^{-4}$ M). (B) Obtained with a $7 \cdot 10^{-3}$ M benzoate eluent (pH 5.0) and detection at 285 nm. Peaks; 1, IO_3^- ($1 \cdot 10^{-4}$ M); 2, Cl^- ($1 \cdot 10^{-4}$ M); 3, NO_2^- ($1 \cdot 10^{-4}$ M); 4, Br^- ($1 \cdot 10^{-4}$ M); 5, NO_3^- ($1 \cdot 10^{-4}$ M); 6, sulfanilate ($2 \cdot 10^{-5}$ M); 7, SO_4^{2-} ($5 \cdot 10^{-5}$ M). (C) Obtained with a $7 \cdot 10^{-3}$ M sulfanilate eluent (pH 5.7) and detection at 305 nm. Peaks; 1, IO_3^- ($7.5 \cdot 10^{-4}$ M); 2, lactate ($7.5 \cdot 10^{-4}$ M); 3, formate ($7.5 \cdot 10^{-4}$ M); 4, Cl^- ($7.5 \cdot 10^{-4}$ M); 5, NO_2^- ($7.5 \cdot 10^{-4}$ M); 6, Br^- ($7.5 \cdot 10^{-4}$ M); 7, NO_3^- ($7.5 \cdot 10^{-4}$ M).

(Fig. 1A), and both eluents of $7 \cdot 10^{-3}$ M benzoate (pH 5.0) and $7 \cdot 10^{-3}$ M sulfanilate (pH 5.7) resolved seven anions (Fig. 1B and C). Compared with benzoate and sulfanilate, a lower concentration of phthalate was used in the eluent. The number of theoretical plates and height equivalent to a theoretical plate, calculated from the nitrite peak obtained

Table 1

Number of theoretical plates (N) and height equivalent to a theoretical plate ($HETP$) for nitrite peak using eluents of phthalate, benzoate and sulfanilate^a

	Eluent		
	$1.5 \cdot 10^{-3} M$ Phthalate (pH 5.0)	$7 \cdot 10^{-3} M$ Benzoate (pH 5.0)	$7 \cdot 10^{-3} M$ Sulfanilate (pH 5.7)
N	980	720	920
$HETP/mm$	0.051	0.069	0.054

^a A $1 \cdot 10^{-5} M$ nitrite was used.

using three eluents, are listed in Table 1. Phthalate proved to be more effective as an eluent for the elution of nitrite than benzoate and sulfanilate. Consequently, a $1.5 \cdot 10^{-3} M$ phthalate solution (pH 5.0) was selected in this work.

3.2. Optimal concentrations of iodide, copper(II) and nitric acid in post-column-reaction solution

At first, detection of nitrite ($5 \cdot 10^{-5} M$) in the presence of chloride of $5 \cdot 10^{-5}$ and $1 \cdot 10^{-3} M$ was tried by using a conductivity detector and a photometer; a $1.5 \cdot 10^{-3} M$ phthalate eluent (pH 5.0) was used but the post-column reaction was not carried out. The results are shown in Fig. 2. In Fig. 2A obtained

by a photometric detection, nitrite and chloride gave a decrease in absorbance of phthalate at 260 nm; Fig. 2B was obtained by conductivity detection. In both detections, the nitrite (peak 2) was separated from the $5 \cdot 10^{-5} M$ chloride (peak 1), but when the chloride concentration increased to $1 \cdot 10^{-3} M$, the nitrite (peak 2) could not be separated satisfactorily from the chloride (peak 3) because the efficiency of the column used in this work was not high ($N=980$, $HETP=0.051$ mm) as can be seen in Table 1. Therefore, in order to selectively determine nitrite in the presence of large amounts of chloride even when the column efficiency is not high, an attempt was made to measure, photometrically, iodine (as triiodide) formed by post-column reaction between iodide and nitrite in the effluent from the column. In this experiment, an iodide–copper(II)–nitric acid solution was employed for the post-column reaction of nitrite. Fig. 3 shows the effects of concentrations of iodide, copper(II) and nitric acid in the post-column-reaction solution, in which a $1 \cdot 10^{-6} M$ nitrite solution was also used as a sample. When various iodide solutions ($5 \cdot 10^{-4}$ – $6 \cdot 10^{-3} M$) containing $5 \cdot 10^{-5} M$ copper(II) and $0.5 M$ nitric acid were employed, the chromatographic peak height for the nitrite was increased by increasing the iodide concentration up to $3 \cdot 10^{-3} M$. Maximal peak height was obtained by the use of iodide solutions ranging in concentrations from $3 \cdot 10^{-3}$ to $6 \cdot 10^{-3} M$ (Fig. 3A).

The height of the nitrite peak was also found to be affected by trace amounts of copper(II) added to the post-column-reaction solution. Therefore, to measure the effect of concentration of copper(II) on the nitrite peak-height, iodide ($3 \cdot 10^{-3} M$)–nitric acid ($0.5 M$) solutions containing various amounts of copper(II) were used. When copper(II) ranged from $5 \cdot 10^{-5}$ to $1 \cdot 10^{-4} M$, the highest peak height for nitrite was

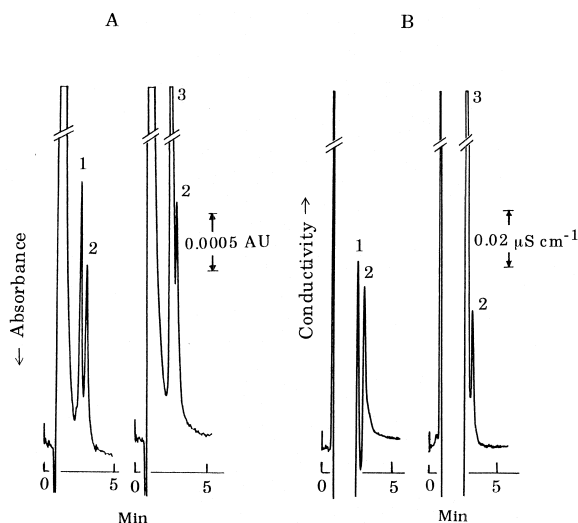


Fig. 2. Chromatogram of nitrite in the presence of chloride. A $1.5 \cdot 10^{-3} M$ phthalate eluent (pH 5.0) was used at a flow-rate of 0.8 ml min^{-1} . Peaks; 1, Cl^- ($5 \cdot 10^{-5} M$); 2, NO_2^- ($5 \cdot 10^{-5} M$); 3, Cl^- ($1 \cdot 10^{-3} M$). (A) Obtained using a photometric detector with 260 nm. (B) Obtained using a conductivity detector.

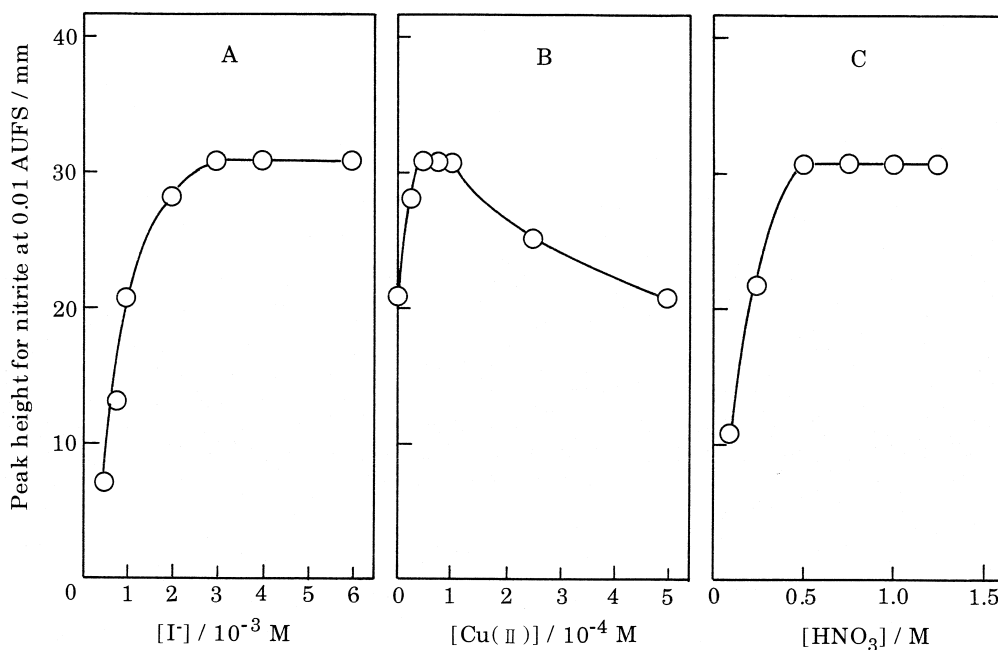


Fig. 3. Effect of concentration of iodide, copper(II) and nitric acid in a post-column-reaction solution on nitrite peak-height. A $1.5 \cdot 10^{-3}$ M phthalate eluent (pH 5.0) and a post-column-reaction solution were allowed to flow at a rate of 0.8 ml min^{-1} , and $50 \mu\text{l}$ of $1 \cdot 10^{-6}$ M nitrite solution was injected. (A) Effect of concentration of iodide in the mixture containing $5 \cdot 10^{-5}$ M copper(II) and 0.5 M nitric acid. (B) Effect of concentration of copper(II) in the mixture containing $3 \cdot 10^{-3}$ M iodide and 0.5 M nitric acid. (C) Effect of concentration of acid in the mixture containing $3 \cdot 10^{-3}$ M iodide and $5 \cdot 10^{-5}$ M copper(II).

obtained (Fig. 3B). At a concentration of copper(II) above $1 \cdot 10^{-4}$ M, the peak height was decreased.

Nitrite oxidizes iodide to form iodine in an acidic medium. Therefore, we tried to add various amounts of acetic, hydrochloric, nitric and sulfuric acids to a $3 \cdot 10^{-3}$ M iodide– $5 \cdot 10^{-5}$ M copper(II) solution in order to acidify the post-column-reaction mixture. Acetic and hydrochloric acids gave very low peak height for nitrite even at concentrations as high as 1.25 M. But, nitric acid and sulfuric acid greatly increased the peak-height of nitrite by an increase in their concentrations. When the concentration of each nitric acid and sulfuric acid was ranged from 0.5 to 1.25 M, the nitrite peak attained its maximum height; nitric acid gave the same height for the nitrite peak as sulfuric acid. Fig. 3C shows the effect of concentration of nitric acid on the nitrite peak-height.

Consequently, a solution of $3 \cdot 10^{-3}$ M iodide, $5 \cdot 10^{-5}$ M copper(II) and 0.5 M nitric acid was employed for the post-column reaction of nitrite in Section 2.3. This solution gave a slight increase in

the baseline of the chromatograms as a process of time due to the formation of iodine via the air-oxidation of iodide; the increase did not interfere with the nitrite determination, but a precipitate of CuI did not appear for at least 8 h at room temperature.

3.3. Effect of length of post-column-reaction tube

The length of the reaction tube was thought to affect the nitrite peak-height and the baseline noise. Therefore, reaction tubes (0.5 mm I.D.) of various length (1, 3 and 5 m) were studied. In this case, each $1.5 \cdot 10^{-3}$ M phthalate eluent (pH 5.0) and the post-column-reaction solution was permitted to flow at a rate of 0.8 ml min^{-1} , and a $50 \mu\text{l}$ aliquot of the nitrite solution was injected. The results obtained are shown in Table 2. An increase in the tube length caused a decrease in the peak height of the nitrite as a result of an increase in the peak width and it also

Table 2
Effect of the length of the post-column-reaction tube on the peak height and the detection limit of nitrite

Length of post-column-reaction tube (m)	Peak height ^a for $1 \cdot 10^{-5} M NO_2^-$ (mm)	Detection limit as $S/N=3$ (M)
1	172.6	$2.61 \cdot 10^{-7}$
3	159.8	$1.88 \cdot 10^{-7}$
5	151.2	$1.00 \cdot 10^{-7}$

^a Obtained at 0.02 AUFS.

caused a decrease in the detection limits of nitrite because of a decrease in the baseline noise. Consequently, a 5 m tube was used for the post-column reaction of the nitrite with iodide.

3.4. Calibration plots

A series of standard solutions (50 μ l) of nitrite was treated exactly as according to Section 2.3. The chromatogram obtained is shown in Fig. 4. A

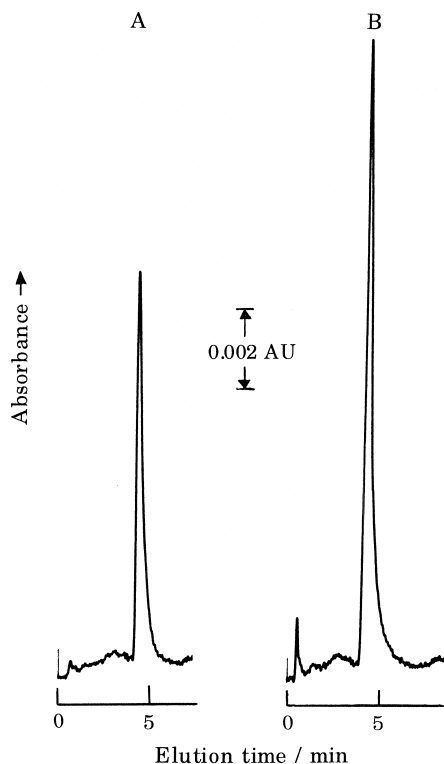


Fig. 4. Chromatogram of standard nitrite. (A) $6 \cdot 10^{-6} M NO_2^-$; (B) $1 \cdot 10^{-5} M NO_2^-$.

calibration graph for nitrite, plotted as peak-height versus concentration, was linear up to $1.50 \cdot 10^{-5} M$ (690 ppb) with a correlation coefficient of over 0.999. Detection limit defined as $S/N=3$ was $1.00 \cdot 10^{-7} M$ (4.60 ppb). This detection limit is lower than that in each previous methods [14,15,18,20,23]. From six results for a 50 μ l of $7.00 \cdot 10^{-6} M$ (322 ppb) nitrite solution, the proposed method afforded a mean value of $6.91 \cdot 10^{-6} M$ (318 ppb) with a standard deviation of $5.04 \cdot 10^{-8} M$ (2.32 ppb) and a relative standard deviation of 0.73%.

3.5. Determination of nitrite in real samples

The effect of foreign ions on the determination of nitrite was investigated firstly. Anions of fluoride, lactate, formate, acetate, propionate, malate, butylate, valerate and chloride and cations of potassium(I), sodium(I), ammonium(I), calcium(II), magnesium(II) and manganese(II) did not give any interferences at concentrations as high as 0.01 M, because these ions were separated from nitrite on the anion-exchange column and also did not react with both iodide and iodine. In an acidic solution, bromate and iodate oxidize iodide to iodine, and sulfide and sulfite also reduce iodine to iodide. Bromate, iodate and sulfide were eluted at much shorter retention times than nitrite and sulfite was eluted at longer retention time than nitrite. Bromate and iodate (up to 0.01 M) and sulfide and sulfite (up to 0.001 M) did not interfere. Iron(II), iron(III) and cobalt(II) were eluted rapidly because they were not retained on the column. These cations up to 0.001 M did not give any interference. Therefore, if ions such as bromate, iodate, sulfide, sulfite, iron(II), iron(III) and cobalt(II) were not separated, they should give interference with nitrite determination.

For the detection of nitrite in a river water sample, the proposed method was compared with both conductivity detection and photometric detection without the post-column reaction; the effluent from the column was passed through a conductivity detector and a photometer at 260 nm, respectively. The results are shown in Fig. 5. The nitrite peak (peak 2) obtained without the post-column reaction (Fig. 5B and C) gave a poor shape and could not be separated from the chloride peak (peak 1), respectively. However, as shown in Fig. 5A, the chromatogram ob-

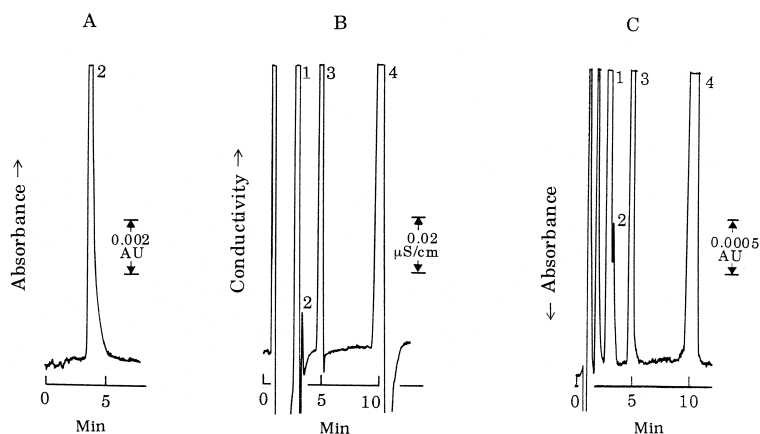


Fig. 5. A chromatogram of nitrite in a river water sample (A) This method used the post-column reaction of the nitrite with iodide; (B) conductivity detection in which the effluent was passed through a conductivity detector; (C) photometric detection in which the effluent was passed through a photometer at 260 nm. Peaks; 1, Cl^- ; 2, NO_2^- ; 3, NO_3^- ; 4, SO_4^{2-} .

tained using the post-column reaction (the proposed method) afforded a single peak for nitrite; Fig. 5A did not show any chloride, or other, peaks. The facts reveal that the post-column reaction of nitrite with iodide is very useful for the chromatographic determination of nitrite in the presence of large amount of chloride.

The proposed method was applied to the determination of nitrite in real samples. The results are shown in Table 3. The recoveries for the nitrite added to lake water (a) diluted 1.2-fold and river water diluted 10-fold ranged from 98.8 to 101.7%. This prove that matrix ions in the real samples did not give any interference with the determination of nitrite. The precision of this method was based on five replicate analyses of lake water (a). This method afforded a mean value of 9.00 ppb with a standard

deviation of 0.15 ppb and a relative standard deviation of 1.67%. The present method could be applied successfully to the determination of nitrite at the ppb level in real samples.

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Table 3
Determination of nitrite in lake water, snow, river water and sewage works water

Sample	Dilution (fold)	Added (ppb)	Found (ppb)	Found in sample (ppb)	Recovery (%)
Lake water (a)	–	–	9.00	9.00	–
	1.2	11.5	18.9	8.88	99.3
	1.2	23.0	30.3	8.76	99.2
Lake water (b)	–	–	6.07	6.07	–
Snow	–	–	12.6	12.6	–
River water	10	–	94.2	94.2	–
	10	46.0	141	950	101.7
	10	230	322	915	98.8
Sewage works water	10	–	342	3420	–

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