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Sample preparation for ion chromatography by solid-phase extraction

INGA K. HENDERSON*, RAAIDAH SAARI-NORDHAUS and JAMES M. ANDERSON, Jr.
Alltech Associates Inc., 2051 Waukegan Road, Deerfield, IL 60015 (U.S.A.)

ABSTRACT

The most difficult part of many ion chromatographic (IC) applications is elimination of interfering components from the sample matrix. These interfering compounds may overload the column, contaminate or mask the peaks of interest or may be retained irreversibly on the packing. Some type of sample preparation is usually necessary to clean up samples prior to analysis. Sample preparation cartridges have been developed for IC using solid-phase extraction. These cartridges take part in specific, selective, solid-phase reactions with samples to eliminate interferences. Recovery of the ions after sample preparation was examined.

INTRODUCTION

Sample preparation in ion chromatography (IC) is often required to eliminate interfering components from samples before chromatographic analysis. Interferences may mask or co-elute with peaks of interest, overload the column or cause baseline disturbances, rendering analysis impossible. The column life can be reduced owing to sample components irreversibly binding to the packing. While many approaches to sample preparation exist, not all will solve the interference problems unique to IC. Two of the simplest and easiest to use sample preparation methods, dilution and filtration, are not suitable with some samples. If the analytes are present at trace levels, differ greatly in concentration or the interfering compound and the analytes are both soluble, neither method is satisfactory. Hydrophobic components in samples may be irreversibly retained on the column, thus shortening the column lifetime. Injection of samples with either a high or a low pH will often produce unacceptable chromatograms due to baseline disturbances. Naturally occurring anions such as chloride and sulfate can interfere with the determination of other ions in samples such as sea water and oil-field brine. These samples require selective removal of interfering matrix components.

Solid-phase extraction (SPE), introduced in the 1970s, is one of the fastest growing sample preparation techniques used in chromatography today [1]. SPE is frequently used to solve interference problems in high-performance liquid chromatography (HPLC) and thin-layer chromatography [1,2]. Solid-phase extraction cartridges can also be used to eliminate matrix interferences from samples prior to analy-

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sis by IC. Not all SPE devices are appropriate for IC applications. Conventional sample preparation devices were plagued with contaminants released from the cartridge or filter. Bagchi and Haddad [3] studied SPE devices packed with octadecylsilyl silica and discovered considerable amounts of extractable anions and cations. New SPE cartridges have been developed to alleviate the interference problem unique to IC without contaminating or altering the sample. Each cartridge consists of polystyrene-based packing material cleaned with ion-free water and molded into medical-grade polypropylene housing. These cartridges are available in five different chemistries to address a variety of IC sample pretreatment problems. This paper reports the recovery of ions using SPE for sample preparation before analysis by IC.

EXPERIMENTAL

Instrumentation

All chromatographic equipment, columns and reagents, unless stated otherwise, were obtained from Alltech (Deerfield, IL, U.S.A.). The ion chromatographic system, the ICM-300, includes a Rheodyne Model 9125 injection valve and a Model 315 conductivity detector with a Model 325 HPLC pump. Data collection was provided by a Spectra-Physics (Santa Clara, CA, U.S.A.) Chromjet integrator. A Model 204 UV detector (Linear Instrument, Reno, NV, U.S.A.) was used when UV detection was necessary. A 150 mm × 4.6 mm I.D. Alltech Universal Anion column, packed with hydroxyethyl methacrylate copolymer-based anion exchanger, was used to determine the recovery of anions. Cation applications were obtained using a sulfonated poly(styrene–divinylbenzene)-based Wescan Cation/R column (100 mm × 3.2 mm I.D.).

Eluents

The eluent used with the Alltech Universal Anion column was 5 m*M* *p*-hydroxybenzoic acid, adjusted to pH 7.9 with lithium hydroxide. To detect nitrite in the presence of sulfuric acid with UV detection, 15 m*M* sodium hydroxide was used with the Alltech Universal Anion column. The eluent used for cation determinations on the Cation/R column was 3.2 m*M* nitric acid. The *p*-hydroxybenzoic and nitric acid eluents were prepared using EZ-LUTE buffers from Alltech.

Reagents and chemicals

HPLC-grade water was used to prepare all standards and eluents. The standard mixtures were prepared using NBS or ACS materials. The anion mixture was prepared from the sodium or potassium salts of fluoride, chloride, nitrite, bromide, nitrate, phosphate and sulfate. The cation mixture was prepared from chloride or fluoride salts of lithium, ammonium, sodium and potassium. All other chemicals used for preparation of standards and eluents were of analytical reagent grade and were acquired from a variety of companies.

Sample pretreatment with solid-phase extraction cartridges

Each SPE cartridge (Maxi-Clean IC Cartridges; Alltech) consists of 0.5 ml of polystyrene-based packing material sandwiched between 20-μm polyethylene frits in a medical-grade polypropylene housing (Fig. 1). The chemical characteristics of the

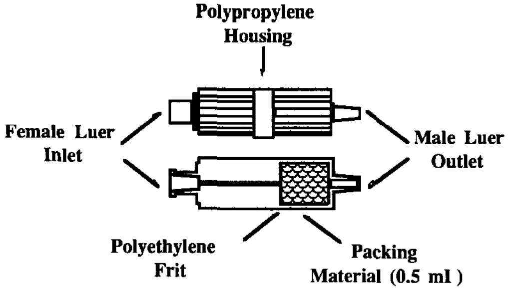

Fig. 1. Construction of the Alltech Maxi-Clean IC cartridge.

packing dictate which sample components are retained. The resin used in the SPE cartridges is polystyrene, which is free from ionic contaminants. The neutral form of the resin is used in the reversed-phase SPE cartridge. The sulfonated form of the resin is converted to the hydrogen, barium or silver form for selective retention of interfering components. The quaternary ammonium form of the resin in the hydroxide form was used in the SPE cartridge for retention of anions. Further information concerning the packing chemistries and the extraction procedure has been given previously [4].

Sample pretreatment with hollow-fiber material

An ion-exchange apparatus similar to that developed by Cox *et al.* [5] was prepared in-house to compare cation-exchange hollow-fiber material with cation-exchange resin. It consisted of a 1.5-m section of Nafion cation-exchange hollow-fiber material (Perma Pure Products, Toms River, NJ, U.S.A.) that was coiled and placed in a slurry of 100 g of Dowex AG 50W-X12 (H^+) analytical-reagent grade cation-exchange resin (50–100 mesh) (Bio-Rad Labs., Richmond, CA, U.S.A.) and 100 ml of IC-grade water. The slurry was stirred with a magnetic stirrer bar on a stir plate. One end of the Nafion tubing was connected with plastic tubing for sample collection and the other end was connected to a peristaltic pump. The anionic samples were passed through the hollow-fiber material at a rate of 1.32 ml/min. A commercially available hollow-fiber membrane-based sample preparation device (Millitrap H^+; Millipore, Milford, MA, U.S.A.) was also compared with the SPE cartridge packed with strong anion-exchange resin in the hydrogen form.

RESULTS AND DISCUSSION

Ion chromatography has been used for the determination of ions in water and in many other matrices. In most instances sample pretreatment other than filtration or dilution is not necessary. When sample matrices contain interfering components that cannot be removed by filtration or dilution, more extensive sample preparation may be required. Any handling of the sample may sometimes introduce imprecision greater than that found within the chromatographic process. The success of the analysis is often dependent on successful sample preparation. With SPE cartridges the sample is passed through the packing. Specific chemical interactions take place, which

TABLE I

CHARACTERISTICS OF SOLID-PHASE EXTRACTION CARTRIDGES (MAXI-CLEAN IC CARTRIDGES; ALLTECH)

Packing	Particle size (μm)	Retains	Capacity (mequiv.)	Applications
Polystyrene	~300	Hydrophobic components	Variable	Removal of surfactants, organic acids and other organic substances. Inorganic ions pass through
Strong anion exchanger in OH^- form	75	Anions	0.6	Exchanges anions for hydroxide. May be used to remove or concentrate anions from sample and to increase pH of acidic samples. Remove cations that form insoluble hydroxide salts
Strong cation exchanger in H^+ from	106	Cations	0.8	Exchanges cations for H^+. May be used to remove or concentrate cations from sample and to reduce pH of basic samples.
Strong cation exchanger in Ag^+ form	106	Chloride, iodide, bromide	0.8	Removes excess halides through formation of Ag halide salts
Strong cation exchanger in Ba^{+2} form	106	Sulfate	0.8	Removes excess sulfate through formation of $BaSO_4$

selectively retain certain components of the matrix in the cartridges, while the remaining components pass through unchanged. In most applications, SPE cartridges provide the means to analyze these samples without sacrificing the recovery of the analytes of interest. Table I summarizes the characteristics of these SPE cartridges.

Removal of hydrophobic compounds

The reversed-phase SPE cartridge provides a reliable method for the removal of surfactants, organic acids, proteins and other organic substances that would otherwise interfere with IC analysis or damage the column. Hydrophobic components of the sample are retained on the packing by reversed-phase mechanisms. Polar organic compounds and inorganic anions are not retained. The net result is removal of hydrophobic components from the matrix while polar components pass through intact.

Adjustment of sample pH

Samples that are strongly acidic or alkaline are difficult to analyze owing to the baseline disturbances that they create. With some IC eluents, system peaks result when the pH values of the sample and the eluent are different. The strong anion-exchange resin in the hydroxide form adjusts the pH of acidic samples prior to analysis of cations by IC. Hydroxide is weakly retained by the resin and is readily displaced by most other anions. The hydroxide contained on the packing reacts with hydronium from the sample to form water, thereby increasing the pH. In this process, anions from the sample displace the hydroxide on the resin, which is then consumed in a neutralization reaction. The net result is removal of hydronium and an equivalent amount of sample anions from the matrix.

The strong cation-exchange resin in the hydrogen form offers a reliable procedure to reduce the pH of alkaline samples prior to analysis. The hydronium contained on the packing reacts with hydroxide from the sample to form water, thereby reducing the pH. In this process, cations from the sample displace the hydronium on the resin, which is then consumed in the neutralization reaction. The net result is removal of hydroxide and the equivalent amount of sample cations from the matrix.

Selective removal of interferences by precipitation reaction

An SPE cartridge packed with strong cation-exchange resin in the barium or the silver form depends on selective precipitation reactions to remove or reduce the concentration of sulfate or halides, respectively. The barium or silver contained on the packing reacts with the sulfate or halides from the sample to form the insoluble barium sulfate or silver halide salt. In this process, cations from the sample displace the barium or silver from the resin, which is then consumed in the precipitation reaction. The net result is removal of sulfate or halides and an equivalent amount of sample cations from the matrix.

Recovery of ions with SPE

Often sample preparation results in the loss, dilution or alteration of components of interest. In most instances the SPE cartridges selectively eliminate matrix interferences without significant effects on the remaining ions. Recovery data were obtained by comparing standard solutions analyzed by IC before and after sample preparation with SPE cartridges. Table II exhibits the recovery data for these SPE cartridges. The reversed-phase SPE cartridge removes hydrophobic components without altering the anion or cation concentration in the sample. A seven-component anion standard and a four-component monovalent cation standard were passed through the reversed-phase SPE cartridge and analyzed by IC with quantitative recoveries.

The SPE cartridge packed with strong cation-exchange resin in the barium or the silver form selectively remove sulfate or halides with near perfect recovery of the remaining anions. Table III lists the solubilities of some of the silver and barium salts in aqueous solutions. As the removal of anions is dependent on the solubility of the salts in solution, fluoride, chloride, bromide, nitrite and nitrate pass through the barium SPE cartridge unchanged. Because of the limited solubility of barium phosphate, the recovery of the phosphate may be reduced by the SPE cartridges packed with strong cation-exchange resin in the barium form if phosphate is present in a high concentration. The insoluble silver halides are retained by the SPE cartridge in the silver form but fluoride, nitrite, nitrate, phosphate and sulfate ions are in mostly unaffected.

When high concentrations of halides or sulfates are present in a sample, low levels of other anions may be lost. This loss is probably due to the inclusion of the other ions in the precipitating barium sulfate or silver halides. A sample of low-level anions was passed through an SPE cartridge packed with strong cation-exchange resin in the barium form. As shown in Table IV, the recovery is good. However, when the same concentrations are present in a solution of 1000 ppm sulfate, the recovery from the barium-form cartridge ranges from 80 to 85%. Although the recovery is less than 100% under these conditions, the reproducibility of replicate injections is ac-

TABLE II

RECOVERY OF IONS WITH SPE CARTRIDGES

SPE cartridge	Concentration (ppm)	Average recovery (%)	S.D.[a] (%)	R.S.D.[a] (%)	No. of Measurements
Reversed-phase					
Fluoride	10	100.5	0.152	0.15	3
Chloride	20	100.9	0.45	0.45	3
Nitrite	20	99.1	0.65	0.65	3
Bromide	20	116.6	2.51	2.15	3
Nitrate	20	100.6	0.45	0.45	3
Phosphate	30	99.6	1.11	1.12	3
Sulfate	30	96.1	0.72	0.75	3
Litium	0.5	95.0	5.18	5.45	3
Sodium	3	97.2	6.56	6.75	3
Ammonium	3	95.8	5.75	6.00	3
Potassium	6	96.7	5.66	5.85	3
Strong cation-exchange resin in barium form					
Fluoride	10	101.7	–	–	1
Chloride	20	111.2	0.98	0.87	3
Nitrite	20	104.4	0.95	0.91	3
Bromide	20	99.3	–	–	1
Nitrate	20	101.1	0.77	0.77	3
Phosphate	30	96.5	–	–	1
Sulfate	30	0.0	–	–	1
Strong cation-exchange resin in silver form					
Fluoride	10	95.2	1.08	1.89	3
Chloride	20	0	0	0	3
Nitrite	20	90.6	15.9	17.6	3
Bromide	20	0.0	0.0	0.0	3
Nitrate	20	102.0	2.66	2.60	3
Phosphate	30	89.0	0.76	0.85	3
Sulfate	30	98.1	0.93	0.95	3
Strong anion-exchange resin in the hydroxide form					
Lithium	0.5	104.0	2.29	2.20	3
Lithium	0.05	96.0	5.30	5.53	3
Sodium	3	103.5	1.55	1.50	3
Sodium	0.3	100.1	0.56	0.60	3
Ammonium	3	103.0	0.36	0.35	3
Ammonium	0.3	100.5	2.31	2.30	3
Potassium	6	105.1	1.77	1.68	3
Potassium	0.6	98.3	2.73	2.78	3
Strong cation-exchange resin in the hydrogen form					
Fluoride	10	96.6	4.33	4.48	3
Chloride	20	102.3	2.57	2.52	2
Nitrite	20	79.1	3.71	4.70	3
Bromide	20	101.6	1.64	1.61	3
Nitrate	20	104.3	2.61	2.50	3
Phosphate	30	86.5	9.39	10.86	3
Sulfate	30	99.4	0.79	0.79	3

[a] S.D. = Standard deviation; R.S.D. = relative standard deviation

TABLE III

SOLUBILITIES OF SILVER AND BARIUM SALTS IN AQUEOUS SOLUTION [6]

Compound	Solubility (g per 100 ml)	Temperature (°C)
AgF	182	15.5
AgI	$2.8 \cdot 10^{-7}$	25
AgCl	$8.9 \cdot 10^{-5}$	10
$AgNO_2$	0.155	0
AgBr	$3.7 \cdot 10^{-4}$	100
$AgNO_3$	122	0
Ag_2SO_4	0.57	0
BaF_2	0.12	25
$BaCl_2$	37.5	26
$BA(NO_2)_2$	67.5	20
$Ba(NO_3)_2$	8.7	20
$BaHPO_4$	0.01–0.02	–
Ba_2SO_4	$2.2 \cdot 10^{-4}$	25

ceptable. Quantitative analysis should be possible by preparing standards in solutions containing the expected concentration of sulfate in actual samples and subjecting both standards and samples to the same SPE procedures. When the same experiments were repeated using the silver-form cartridge, the recovery is nearly perfect. With 1000 ppm chloride present, the recoveries of 1 ppm fluoride, nitrite, nitrate and sulfate were in the range 96–102%. The inclusion problems seem to occur only with the barium-form cartridge.

An SPE cartridge packed with strong anion-exchange resin in the hydroxide form adjusts the pH of acidic samples by neutralizing the hydronium ion. The concurrent removal of anions from the sample has little or no effect on the recovery of the cations in the sample even at low concentration levels. Some data reported in Table II shows a slight increase in the concentration of cations during sample pretreatment but it is well within the acceptable range for many applications. The reproducibility of

TABLE IV

RECOVERY OF LOW-LEVEL ANIONS WITH SPE BY PRECIPITATION REACTIONS

Strong cation-exchange resin in the barium form

	Concentration (ppm)	Average recovery (%)	S.D.[a] (%)	R.S.D.[a] (%)
Without sulfate present				
Chloride	1	102.8	0.98	0.87
Nitrite	1	104.4	0.95	0.91
With 1000 ppm of sulfate				
Chloride	1	85.3	3.46	4.06
Nitrite	1	81.4	3.71	4.56
Bromide	1	89.8	3.52	3.92

[a] $n = 3$.

the method will allow accurate quantification when the standards are pretreated similarly to the samples.

The SPE cartridge packed with strong cation-exchange resin in the hydrogen form adjusts the pH of caustic samples by neutralizing the hydroxide ion to water and removing cations in the process. As shown in Table II, the recovery of anions that are strongly ionized is nearly perfect. However, the recovery of weakly ionized anions such as nitrite and phosphate is poor after the sample treatment with this SPE cartridge.

Decrease of nitrite during sample pretreatment

The loss of of nitrite may take place through a number of different mechanisms, including evolution of nitrous oxide, oxidation to nitrate, adsorption in the resin or a combination of effects. When the nitrite ion in solution is passed through the cartridge, it is protonated to the volatile nitrous acid by the hydronium ion released from the resin. Nitrous acid is both volatile and easily oxidized to nitrate. Aqueous solutions of nitrous acid are unstable and can decompose to nitric acid, water and nitrous oxide [7]. The inconsistency of the recovery, apparent in Table II, may depend on the chemical instability and the extent of nitrite conversion to nitrous acid.

The ability of undissociated weak acids such as nitrous acid and carboxylic acid to penetrate past the functional group of the resin packing and be adsorbed in the resin is one reported explanation for the loss of nitrite [8–10]. The adsorption of the undissociated weak acid in the resin pores was examined. The nitrite ion was converted to nitrous acid using the SPE cartridge packed with strong cation-exchange resin in the hydrogen form and then passed through the neutral resin packing in the reversed-phase SPE cartridge. This sample was compared with the same nitrite sample which was passed through the reversed-phase SPE cartridge only. About 5–10% of the nitrite was lost when the sample converted to nitrous acid was passed through the reversed-phase SPE cartridge. The recovery of nitrite after passage through both SPE cartridges varied from 68 to 96%. The loss of nitrite in the nitrous acid sample after treatment with the reversed-phase SPE cartridge suggests that this undissociated weak acid is adsorbed in the resin. Although this experiment reveals some adsorption of nitrous acid in the resin it does not account for all of the nitrite lost in some replicate injections through the SPE cartridge packed with strong cation-exchange resin in the hydrogen form. Similar inconsistencies in the recovery of nitrite were also apparent when a hollow-fiber membrane was used in place of the strong cation-exchange resin in the hydrogen form. The nitrite loss was also studied using the cation-exchange hollow-fiber membrane devices described earlier. Table V compares the recoveries of anions with cation-exchange resin and hollow-fiber material. This data suggest that other mechanisms must also be active to account for the additional loss of nitrite.

Experiments were designed to observe how the recovery of nitrite ion is dependent on the conversion to nitrous acid. Increasing the total ionic concentration of the solution will increase the amount of hydronium available for conversion of nitrite salts to nitrous acid. Various concentration of sulfate were added to nitrite standards to increase the ionic strength of the solutions. After passage through the SPE packed with strong cation-exchange resin in the hydrogen form, the recoveries of nitrite in solutions containing both 10 and 50 ppm of sulfate were nearly perfect. At higher

TABLE V

RECOVERY OF ANIONS WITH CATION-EXCHANGE MATERIALS

	Concentration (ppm)	Average recovery (%)	S.D. (%)	R.S.D. (%)	No. of measurements
Strong cation-exchange resin in hydrogen form					
Fluoride	10	99.1	3.74	3.77	4
Chloride	20	102.8	2.12	2.06	4
Nitrite	20	58.3	15.3	28.24	12
Bromide	20	97.5	3.92	4.02	12
Nitrate	20	109.8	6.26	5.70	12
Phosphate	30	87.0	8.61	9.89	12
Sulfate	30	97.7	4.34	4.44	12
In-house cation-exchange membrane					
Fluoride	10	104.9	6.74	6.43	10
Chloride	20	106.6	3.05	2.86	10
Nitrite	20	49.1	13.54	27.58	11
Bromide	20	98.0	2.69	2.74	11
Nitrate	20	99.5	3.60	3.62	11
Phosphate	30	86.8	5.62	6.48	11
Sulfate	30	97.7	1.86	1.91	11
Commercial cation-exchange membrane					
Fluoride	10	79.8	10.47	13.11	9
Chloride	20	104.9	4.24	4.04	9
Nitrite	20	68.5	10.11	14.75	9
Bromide	20	108.8	6.42	5.91	9
Nitrate	20	106.4	6.68	6.38	9
Phosphate	30	94.2	6.30	6.69	9
Sulfate	30	102.1	7.09	6.95	9

sulfate concentrations the recovery of nitrite decreases. When 100 ppm of sulfate was present in the sample the nitrite recovery was in the range 74–84%, with 350 ppm of sulfate present, the recovery fell to 63% and with 1000 ppm of sulfate present the nitrite recovery was only 30–40%. Fig. 2 shows the chromatogram of nitrite in 1000 ppm sulfate before and after treatment with the SPE cartridge packed with strong cation-exchange resin in the hydrogen form. An increase in the total ionic strength of the solution reduces the recovery of nitrite. This effect can be explained by the increased concentration of hydronium available to drive the nitrite–nitrous acid reaction equilibrium to favor the formation of nitrous acid.

The ability of nitrous acid to oxidize to nitrate was examined as another mechanism for the loss of nitrite. The recovery of nitrate greater than 100% shown in Table II suggest that nitrite is being oxidized to nitrate during the cation-exchange process. To study the effect of an acidic solution on the loss of nitrite, the following experiment was performed. A 25-m*M* sulfuric acid solution was spiked with 30 ppm of nitrite. The Alltech Universal Anion column was used with 15 m*M* sodium hydroxide as eluent and UV detection to monitor the loss of nitrite with time. The nitrite was reduced from 92% to 75% over 2.5 h when compared with the nitrite standard in IC-grade water. After 4 days stored at 40°C the nitrite concentration was consid-

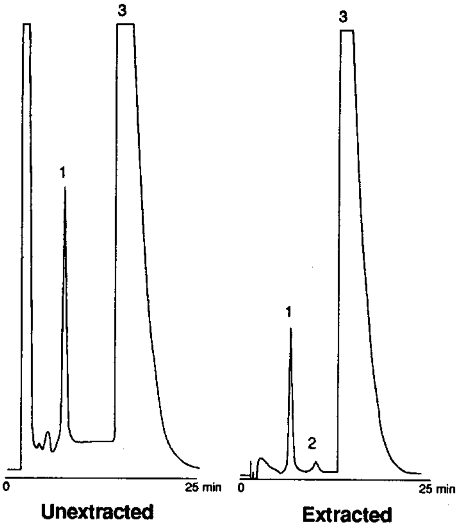

Fig. 2. Nitrite in the presence of 1000 ppm of sulfate, extracted with SPE cartridge packed with a strong cation-exchange resin in the hydrogen form. Column, 150 mm × 4.6 mm I.D.; packing, Universal Anion; eluent 5 m*M* *p*-hydroxybenzoic acid (pH 7.9); flow-rate, 1 ml/min; detector, conductivity. Peaks: 1 = nitrite (100 ppm); 2 = nitrate; 3 = sulfate (1000 ppm).

erably reduced and nitrate was present. The same nitrite concentration in water contained no nitrate after the same time period (see Fig. 3). This suggests that nitrite is oxidized to nitrate in acidic media. Nitrite samples in high concentrations of sulfate presented in Fig. 2 also show oxidation to nitrate when passed through these cartridges.

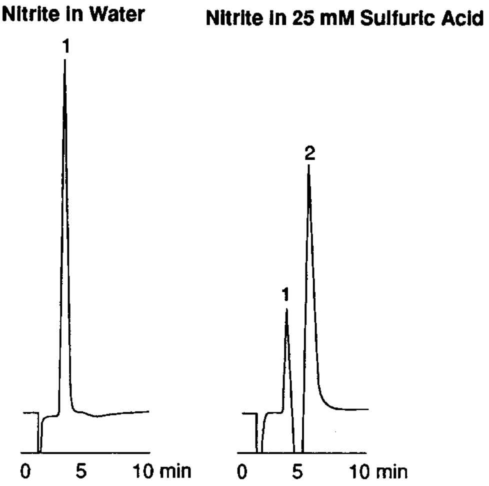

Fig. 3. Nitrite oxidation to nitrate in an acidic solution. Sample, 30 ppm nitrite stored for 4 days at 40°C. Conditions as in Fig. 2 except eluent, 15 m*M* sodium hydroxide solution, and detector, UV (214 nm). Peaks: 1 = nitrite; 2 = nitrate.

The above experiments showed that the poor recovery of nitrite after the sample pretreatment with the hydrogen-form cartridge is due to a combination of several effects which include adsorption in the resin, the formation and evolution of nitrous acid and oxidation to nitrate.

CONCLUSIONS

Solid-phase extraction cartridges provide sample preparation procedures that allow the IC analysis of complex samples. Sample matrix interferences such as acidic or alkaline solutions, interfering levels of halides or sulfate or hydrophobic components are eliminated. After sample pretreatment with SPE cartridges, recovery of all analytes (with the exception of nitrite with the SPE cartridge packed with strong cation-exchange resin in the hydrogen form) is well within the acceptable range for many applications. The exact mechanism of nitrite loss during the cation-exchange process, whether with resin or hollow-fiber, demands further investigation.

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