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Determination of bromide ion in raw and drinking waters by capillary zone electrophoresis

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Abstract

A new capillary zone electrophoretic method was developed for the determination of bromide ion in raw and drinking waters. An NaCl-based low-pH buffer caused a reduction of electroosmotic flow (EOF) in the buffer zone, whereas injected water sample resulted in higher EOF in the sample zone thus pumping out the neutral water plug. Sample stacking was used for the preconcentration. The method was applicable for waters from low to intermediate ionic strengths, i.e., the concentration of chloride should preferably be less than 40 mg/l. The method had a limit of detection of 15 μg/l at a signal-to-noise ratio of three ($S/N=3$) and a limit of quantitation of 20 μg/l. CZE results obtained with real samples were compared with ion chromatography–inductively coupled mass spectrometric results. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

The realization that bromide ion in raw waters can be converted to bromate in drinking water treatment during oxidation with ozone increased interest in this halogen [1,2]. The US Environmental Protection Agency (EPA) has established a maximum contaminant level of 10 μg/l for bromate in finished water because of its suspected carcinogenic potential. If one wishes to predict how much bromate will be formed during water treatment, the concentration of bromide in the raw water coming into the water works has to be known.

A general problem with bromide analysis from raw and drinking waters is the low analyte-to-matrix ratio, μg/l levels of bromide have to be analyzed in the presence of mg/l levels of other anions. Matrix anions consist mainly of chloride, sulfate, carbonate and nitrate. Several techniques have been employed to determine bromide. The use of ion chromatography with inductively coupled plasma mass spectrometry (IC–ICP-MS) has proved to be very effective [3,4] for bromide determination, especially for different oxyhalides, which may be determined in the same run with bromide. One of the major advantages of IC–ICP-MS is its tolerance of high salt concentrations in the samples and also the minimal need for sample pretreatment. Böhme et al. [5] developed a method using IC on coated reversed-phase material and compared their results with a commercially

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available IC method. The limits of detection (LODs) for the self-made and commercial method were 1.1 μg/l and 2.6 μg/l, respectively. However, the coated reversed-phase column method required evaporation in order to preconcentrate the samples by ten-fold.

At present, there have been few attempts to utilize capillary zone electrophoresis (CZE) in the analysis of bromide from raw and drinking waters. Bromide has a high absorbance from 190 to 200 nm and therefore direct detection is favored if the method is intended to target bromide rather than inorganic anions in general. There have been some publications with direct UV detection of bromide. Guan et al. [6] used bromide as an internal standard for the determination of nitrate and nitrite but the method could also have been adopted for bromide determination. Tetraborate was used as the carrier electrolyte. Several anions, including bromide, in water containing high concentrations of salts have been determined by Song et al. [7] using an NaCl-based buffer. Soga et al. [8] analyzed several UV-absorbing anions with a poly(ethyleneglycol)-coated capillary where electroosmotic flow (EOF) was suppressed. Using 20 m*M* phosphate buffer and sample stacking, they achieved a LOD of 24 μg/l for bromide. More recently Fukushi et al. [9] used artificial seawater to determine bromide in seawater. However, none of these methods is directly applicable for raw and drinking water analysis where the concentrations are often less than the 50 μg/l level.

This paper presents a new NaCl-based, low pH method for the determination of bromide from raw and drinking waters using sample stacking for preconcentration. The effects of sample salt content on peak area, peak height and migration time have been investigated. Finally, the proposed method was applied for the analysis of water samples from Finland.

2. Experimental

2.1. Water samples

Raw and drinking water samples were taken from more than 40 major water works in Finland, including water works using ozonation. The raw water samples were either the surface or ground water used by these plants. All samples were stored in 10-ml plastic tubes at −20°C and were stable for at least a year when frozen. Samples were analyzed on the same day they were taken from the freezer. Before analysis, particulate matter in the samples was allowed to sediment by gravity inside the plastic tubes for 10 to 20 min. A volume of 300 μl from the top of the tube was pipetted to an autosampler vial. Before this, all autosampler vials (for samples and standards as well) were sonicated (Branson 3210; Branson Ultrasonics, Danbury, CT, USA) for 1 h in 1% sulfuric acid to remove possible traces of impurities, flushed with Ultrapure water (MQ-water) obtained from an ultrapure water system (Milli-Q plus; Millipore, Molsheim, France), and dried. No caps were used with the vials. In those cases where after 20 min sedimentation there were visible particles in the top of the tube, the samples were filtered through 0.2 μm pore size cellulose acetate based syringe filters (LIDA, Windsor, UK). No pH adjustment, chemical addition, concentration nor dilution was made to the samples before capillary electrophoresis (CE).

2.2. Reagents, standard stock solutions and buffer stock solutions

MQ-water and analytical grade reagents were used to make all buffers, standards and their stock solutions unless otherwise stated. One thousand mg/l standard stock solutions were made from sodium bromide, sodium chloride, sodium sulfate, sodium nitrite, sodium nitrate and potassium iodide (Merck, Darmstadt, Germany). One hundred m*M* stock solutions for buffer preparation were made from formic acid, acetic acid, sodium sulfate (Merck), sodium dithionate (97%) (Pfaltz and Bauer, Waterbury, CT, USA), and cetyltrimethylammonium chloride (CTAC) (Fluka, Buchs, Switzerland). Ultrapure HCl (Ultrex, 37.6%) (J.T. Baker, Phillipsburg, NJ, USA) and NaOH (Merck) were mixed to produce high-purity 100 m*M* NaCl buffer stock solution. Ultrapure HCl was used because analytical grade HCl and NaCl contained too much bromide as impurity. All standard and buffer stock solutions were stored at 4°C and were stable for six months after preparation.

2.3. Working standards and buffer solutions

Bromide working standards between 10 and 500 μg/l were prepared by serial dilution of 1000 mg/l

stock solution. Chloride, sulfate, nitrite, nitrate and iodide were added to bromide standards by dilution of 1000 mg/l stock solutions in order to account for sample matrix effects. One or several of them were added at the time. Working standards were stored at 4°C and were stable for four weeks after preparation.

The running buffer was 15 m*M* sodium chloride, 5 m*M* formic acid, pH 3.50. Dilute NaOH was used for pH adjustment (Basic pH Meter; Denver Instrument Co., Denver, CO, USA).

Preliminary experiments involved buffers composed of sodium dithionate and acetic acid, sodium chloride and acetic acid, plus a buffer with sodium sulfate and CTAC. Dilute NaOH was used for pH adjustment of the first two buffers and no pH adjustment was made for the third one.

All buffers were filtered through 0.45-μm pore size filters and degassed under vacuum immediately after preparation. The filters consisted of mixed nitrate and acetate cellulose ester (Millipore, Cork, Ireland). If buffers were not used on the day of preparation, pH was checked and buffers were redegassed before use. Buffers were stored at 4°C and used within two days of preparation.

2.4. CE system

A 270-HT capillary electrophoresis system (Applied Biosystems, San Jose, CA, USA) with a UV–Vis absorbance detector was used in all experiments. Apparatus control and data processing were from a personal computer with Turbochrom Navigator Version 4.0 software (Perkin-Elmer, San Jose, CA, USA). A polyimide-coated fused-silica capillary (Composite Metal Services, Worcester, UK) of 72 cm (length to the detector 50 cm)×75 μm I.D.×375 μm O.D. was used. A small section of the capillary coating was burnt off with a match to make a window for the on-column UV detector cell. CE equipment uses 67.7 kPa (20 in.Hg) under pressure in the outlet end of the capillary to rinse the capillary and 16.9 kPa (5 in.Hg) to inject samples.

2.5. Capillary conditioning

Every new capillary was first rinsed with 1 *M* NaOH for 30 min and with MQ-water for 10 min. At the beginning of a new day or when the buffer was changed, the capillary was rinsed with 0.1 *M* NaOH for 15 min, and 10 min with MQ-water. Before runs, there was a 40 min rinse with the running buffer and 1 min equilibration with the running voltage of −20 kV from the injection end. The rinse between runs was 3 min with the running buffer. Finally, the rinse at the end of the day was 5 min with 0.1 *M* NaOH and 5 min with MQ-water.

2.6. Instrument conditions and stacking process

The running voltage was −20 kV from the injection end, the current was 30 μA, the detector wavelength was 200 nm, the temperature 30°C, the detector rise time 0.5 s and the sampling rate was 5 points/s.

Sample stacking was used as an in-capillary preconcentration method at the beginning of the run. This was done by performing a 10 s injection, which means that the injected sample volume was 180 nl and the length of the sample plug was 4.1 cm. The sample volume was calculated by the equation of Fukushi et al. [9]. Voltage was then applied and stacking of the sample anions to a narrow band and their separation occurred successively. The sample water plug was simultaneously pumped out by the EOF generated in the sample zone. In the sample stacking samples can be prepared in diluted buffer or in water [10–12]. In our case, water samples were analyzed without buffer addition to keep the stacked sample bands as narrow as possible.

3. Results and discussion

3.1. Selection of preconcentration method

To achieve μg/l levels of bromide without time consuming off-line sample pretreatment, some kind of in-capillary preconcentration method applicable to normal CZE instruments has to be used. The benefits and drawbacks of possible methods for this particular analysis are evaluated here.

3.1.1. Isotachophoretic initial state

The isotachophoretic initial state is both theoretically and practically [13–16] feasible for sample preconcentration in CZE. When bromide is the fastest anion in the water sample in a given CZE buffer, there are two possibilities for transient iso-

tachophoresis (ITP) using a single buffer: the buffer co-ion has to be faster than bromide or an anion faster than bromide has to be added to the sample. Chloride and sulfate which are always present in the samples, act as the terminating anions in the beginning of the run. Dithionate is UV transparent and faster than bromide [17], and thus suitable as a buffer co-ion. Fig. 1 shows an electropherogram, where 1 mg/l of bromide is separated from 25 mg/l of chloride and 50 mg/l of sulfate using dithionate in the buffer. The buffer pH had to be set to 5.05 to induce high enough reverse directed EOF to provide time for the separation of bromide and chloride. In the study of Boden and Bächmann [16] a set of equations are presented from which it becomes clear, that a long ITP time is required in both of the above cases to separate bromide and chloride since their electrophoretic mobilities are so close to each other.

One possibility to overcome this co-migration problem is to change the migration order of bromide and chloride and in this way to enhance their separation. This was tried with a buffer consisting of 10 mM of sodium sulfate and 2 mM of CTAC at pH 6. Here, bromide migrated much slower than chloride so chloride was already able to act as high mobile matrix ion causing transient ITP for bromide. The separation of bromide and chloride was much faster than with the dithionate buffer of Fig. 1. However, CTAC cannot be used at these concentration levels because injection of a long sample zone is not possible due to co-directed EOF.

Due to these difficulties, no more attempts were made to find a working transient ITP system before moving on to sample stacking.

3.1.2. Sample stacking

The theory of sample stacking and different means to pump out the neutral water plug remaining in the capillary after the stacking process have been presented in many reports [10–12]. In this work, normally reverse-directed EOF was suppressed using low buffer pH. When a slightly alkaline water sample enters the capillary, it generates a stronger EOF in its zone and the water plug is thus pumped out of the capillary. Therefore, no EOF modifier is needed as a buffer additive. Another advantage is that neutral particles in the sample are pumped out and thus are not adsorbed onto the capillary surface which

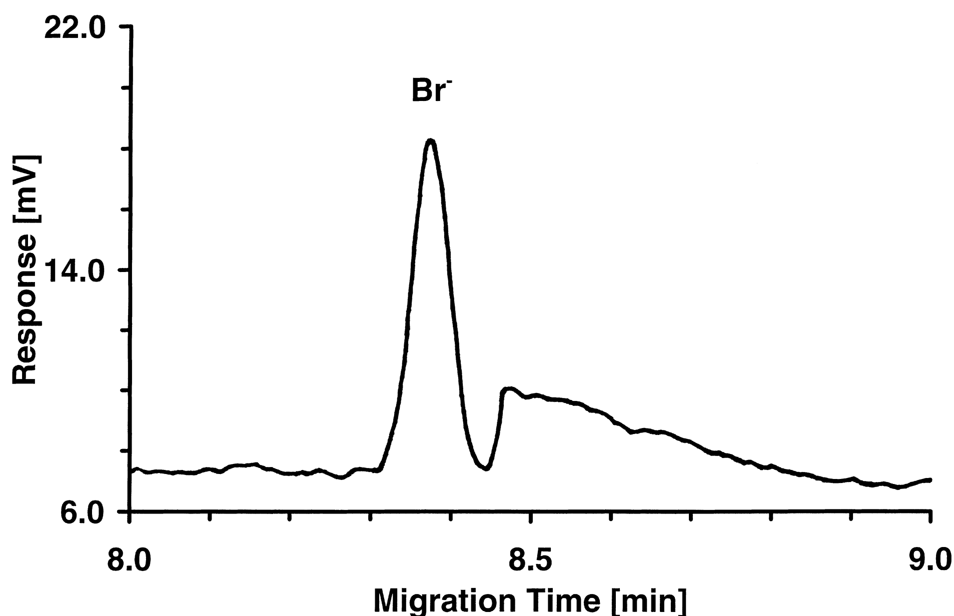

Fig. 1. Separation of 1 mg/l of bromide from 25 mg/l of chloride (flat, tailing peak after bromide) and 50 mg/l of sulfate (not shown). Buffer: 10 mM sodium dithionate, 5 mM acetic acid, pH 5.05. Instrument conditions: separation voltage −15 kV, 10 s injection at 16.9 kPa, detector wavelength 200 nm.

prolongs the capillary lifetime. For this reason in most cases sedimentation of the largest particles is sufficient sample pretreatment and filtration is rarely needed.

There are different opinions when analyte separation actually begins in sample stacking. When sample anions are stacked to a narrow band at the beginning of the run, the sample water plug still remains in the capillary behind them. According to Burgi [11], analyte separation begins only when the whole sample water plug has been pumped out. However, this study suggests that analytes penetrate to the buffer and start to separate before the pumping process is completed. This conclusion was drawn from the fact that it sometimes required almost the migration time of bromide before the final current was achieved. The final current is reached only when the whole sample plug is pumped out and the capillary is completely filled with the running buffer. The migration time of bromide is too short to confirm the theory of Burgi.

Chloride was selected as the buffer co-ion because the co-migration problem mentioned above could be avoided. One further advantage of chloride is that when there are only anions slower than chloride in the sample, they cause no peak broadening for bromide. This phenomenon is illustrated in Table 1, where migration times, peak areas, peak heights and area/height ratios are displayed for standards containing 50 μg/l of bromide and different amounts of chloride and sulfate. With addition of only sulfate (slower than chloride in all buffers used) up to 100 mg/l, no peak broadening for bromide was observed, because sulfate migrates in the ITP mode in the beginning of the run. The small irregularities seen in the area/height ratios in Table 1 for different sulfate concentrations are due to random variation. In contrast, when a sample contains only chloride, peak broadening occurs according to the basic theory of sample stacking. This effect is more clearly seen only when chloride concentration is higher than 10 mg/l. When a sample contains 100 mg/l of sulfate and increasing amounts of chloride, their combined effect on peak broadening is more pronounced than the effect of chloride alone. In this case there is some sort of combination of ITP and sample stacking at the beginning of the run. The same phenomenon was also observed in the study of Boden et al. [15], where common inorganic anions were determined in a hydrofluoric acid matrix using chromate electrolyte. Bromide was the only anion of similar or faster speed than chromate. When the concentration of the fluoride ion in the sample was increased, the peak heights of every other anion increased according to ITP theory, but the peak height of bromide remained the same up to 400 m*M* of fluoride because there was no other anion as fast as or faster than chromate to cause bromide peak broadening.

Table 1
Migration times, peak areas, peak heights and area/height ratios (A/H) for the 50 μg/l bromide peak in standards containing different amounts of sulfate and chloride[a]

$[SO_4^{2-}]+[Cl^-]$ (mg/l)	Time (min)	Area (μV·s)	Height (μV)	A/H
20+0	4.01	1147	761	1.51
50+0	4.01	1140	776	1.47
100+0	3.97	1194	742	1.61
0+10	4.01	1162	785	1.48
0+30	4.02	1039	606	1.71
0+50	4.01	1180	536	2.20
100+10	3.95	1066	648	1.65
100+30	3.93	947	464	2.04
100+50	3.90	1159	348	3.33
Mean	3.98	1115	–	–
RSD (%)	1.09	7.27	–	–

[a] Buffer: 15 m*M* NaCl, 5 m*M* formic acid, pH 3.5. Instrument conditions: −20 kV, 200 nm, 10 s injection.

Table 1 also shows that the migration time of bromide decreases when the ionic strength of the sample increases. This phenomenon is known as reverse electrostacking [18]. If great variations are observed with real samples, migration time corrected peak areas can be used [19]. With the buffer used, the relative standard deviation (RSD) of the migration times is small and no correction is required.

3.2. Optimization of the buffer composition

The optimum concentration of chloride in the buffer is a compromise between low background absorbance and sharp peaks. As stated, chloride is the main anion in the sample causing peak broadening and thus the chloride concentration in the buffer has to be selected with respect to the sample concentration. Since chloride concentrations in Finnish raw and drinking waters are usually less than 40

mg/l, the optimum buffer concentration was found to be around 15 m*M*. A lower concentration of chloride in the buffer led to too broad bromide peaks when the sample chloride concentrations were 40 mg/l. Buffer chloride concentrations up to 20 m*M* gave sharper peaks and slightly improved sensitivity but peak area reproducibility was somewhat poorer. If the concentration of chloride was further increased, increasing background absorbance started to reduce the peak size. Since a marginal increase in sensitivity at 20 m*M* was not considered very important for our purposes if it could only be achieved by sacrificing reproducibility, 15 m*M* was selected.

The EOF in uncoated fused-silica capillaries is significantly reduced when the pH is less than 5 [20]. Since some samples, especially those having high carbonate concentrations, may have considerable ability to elevate the pH inside the capillary, a buffering compound has to be used to maintain a stable EOF throughout the run. Five m*M* formic acid was selected for buffering at pH 3.5. Buffering with formic acid at pH 3.5 resulted in prolonged out pumping times and sometimes it required almost the migration time of bromide until the current had risen to its final value. Subsequently, the baseline was still increasing until the bromide peak was detected due to increasing Joule heating and thus increasing background absorbance. The benefit was that migration time reproducibilities were excellent at pH 3.5. Buffering with acetic acid at pH 4.1 was also attempted but migration time repeatability was not as good at pH 4.1 as that achieved at 3.5. In particular, day-to-day variations were much greater. This may be because small pH changes can cause greater EOF changes when the pH is higher than 4, and furthermore the buffering capacity of acetic acid is not as good so far from its pK_a value. For this reason, the buffer pH was set at pH 3.5 despite the difficulties mentioned above. More research is being done to speed up out pumping without compromising reproducibility.

3.3. Optimization of the instrument parameters

The detector wavelength was set to 200 nm, because lower values resulted in a noisy baseline, even though the peak area of bromide slightly increased at 190 nm. A voltage of −20 kV was used for the separation to keep the analysis time reasonably short and to avoid excessive current.

The length of the injected sample plug was optimized simultaneously with the concentration of chloride in the buffer. The goal was to reach a low LOD accompanied by a reasonable tolerance to increased sample chloride concentration. The optimum injection time was 10 s when the buffer chloride concentration was 15 m*M*. The suitability of this combination was confirmed by analyzing raw and drinking waters from more than 40 major water works in Finland. Bromide peak broadening did not prevent quantitation in any sample when the concentration was more than 20 μg/l.

3.4. Analytical performance of the selected buffer

Standards for LOD, limit of quantitation (LOQ), and reproducibility experiments had a background of 20 mg/l of sulfate and 10 mg/l of chloride to simulate real samples. LOD was 15 μg/l at a signal-to-noise ratio of three ($S/N=3$) but it does increase when the concentration of chloride is significantly more than 10 mg/l as can be deduced from Table 1. Determination of LOQ and reproducibility tests were made with six repeated injections for both 20 μg/l and 100 μg/l standards. The LOQ was defined as the concentration level at which the RSD of peak areas was less than 10%. RSD for peak areas at 20 μg/l was 7.58% which satisfied this requirement. RSD for migration times was 0.14% in the LOQ experiments. In the reproducibility experiments at 100 μg/l, RSD for peak areas was 2.44% and 0.14% for migration times. It must be noted that at concentrations close to the LOD, small variations in peak integration can cause significant relative variations in peak area and thus also in the calculated concentration. Fig. 2 shows an electropherogram of a 20 μg/l standard.

Since bromide migration times differ with changing ionic strength of the sample, calibration standards should have migration time equivalent to average of migration times of real samples to minimize quantitation errors due to varying migration times. Three sets of calibration standards were made with 20, 50 and 70 mg/l of sulfate added to bromide standards. Seventy mg/l was found to be an optimum background concentration of sulfate repre-

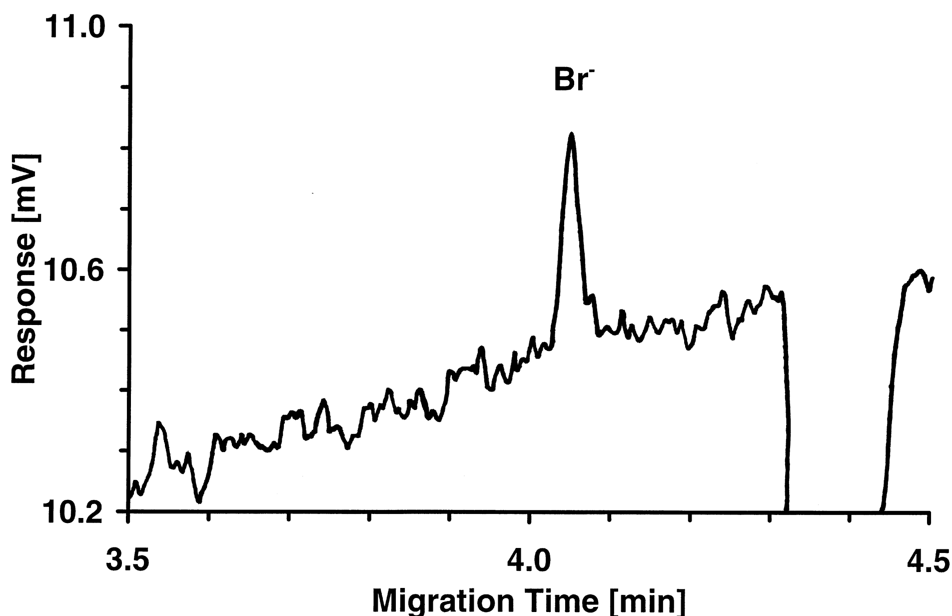

Fig. 2. Electropherogram from a LOQ standard containing 20 μg/l of bromide, 10 mg/l of chloride and 20 mg/l of sulfate (dip after bromide). Buffer: 15 m*M* sodium chloride, 5 m*M* formic acid, pH 3.50. Instrument conditions: separation voltage −20 kV, 10 s injection at 16.9 kPa, detector wavelength 200 nm.

senting the average bromide migration time for samples collected from different water works in Finland. The ionic strength of these standards may be somewhat higher than the average of Finnish raw and drinking waters, but for some undetermined reason it was found to give a better mobility match for bromide than standards with lower sulfate concentrations. The calibration graph for bromide was linear at least up to 500 μg/l. A five-point regression equation relating area responses (y, μV·s) to concentration (x, 20–500 μg/l) was $y=22.284\pm0.349x-35.394\pm24.615$, ($n=5$). From these figures it can be seen that the intercept changed from day-to-day but the slope remained relatively constant. Major changes in the intercept may have an impact on quantitation especially at low peak areas. The correlation coefficient ranged from 0.9997 to 0.9999.

Possible co-migration problems were also examined. Of the compounds sometimes present in raw and drinking waters, only the iodide peak was close to bromide but it was baseline-separated and migrated after bromide in a standard where both were present at 100 μg/l. The size of the iodide peak was about two thirds of that of bromide. The background for this standard was 10 mg/l of chloride and 50 mg/l of sulfate. Identification of bromide and iodide was not a problem, because iodide is usually present at much lower concentrations than bromide and bromide can thus serve as a reference. The concentration of iodide in Finnish waters is usually very low and it was not detected in any of the samples analyzed.

A number of water samples were analyzed with both CZE and IC–ICP-MS. The results of five of these samples are presented in Table 2. The higher concentrations obtained with CZE in samples 1 and 2

Table 2
Analytical results of bromide in water samples by CZE and IC–ICP-MS

Sample	CZE[a] (μg/l)	IC–ICP-MS (μg/l)
(1) Raw water	25	19
(2) Raw water	30	17
(3) Drinking water	136	140
(4) Drinking water	40	37
(5) Drinking water	35	30

[a] CZE conditions as in Table 1.

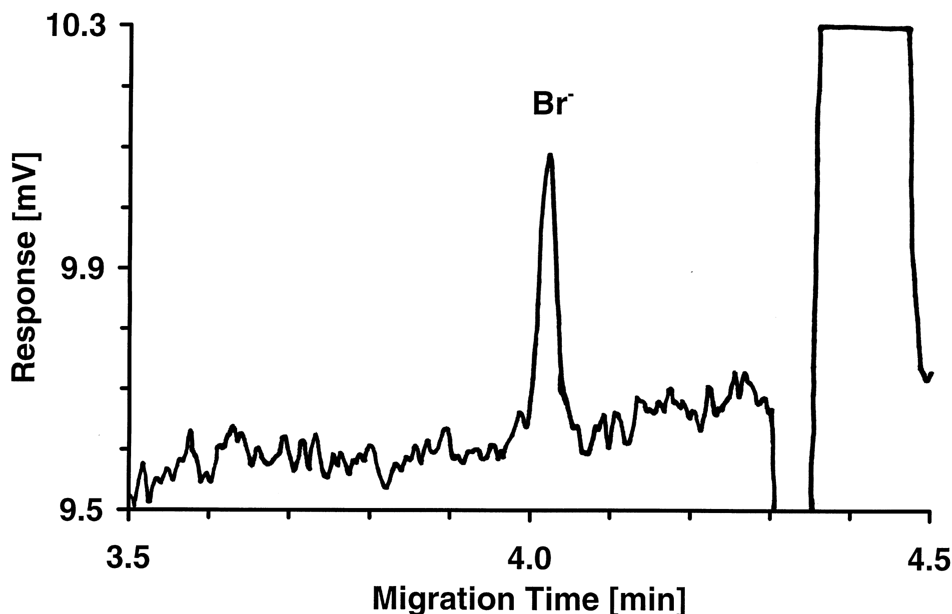

Fig. 3. Electropherogram from a raw water sample containing 30 μg/l of bromide. The dip after bromide is sulfate and the peak following sulfate is nitrate. Buffer and instrument conditions as in Fig. 2.

are perhaps due to the fact that samples were not filtered prior to CZE analysis whereas they were invariably filtered before the IC–ICP-MS analysis. It is possible that in raw water samples there was some retention of bromide onto some material not passing through the filter. For example humic compounds present in untreated Finnish surface waters could cause such adsorption. Similar results were also observed with some other raw waters but results with drinking waters were usually more identical. Fig. 3 shows the electropherogram of sample 2 in Table 2.

4. Conclusions

The developed low-pH buffer is very simple and is applicable for all basic CE instruments. Even though the concentration of chloride is the major factor increasing the LOD of the method, Finnish raw and drinking waters are in most cases sufficiently low in salinity to enable a LOQ of 20 μg/l. Migration time reproducibility was excellent with identical standards, and even with greatly varying ionic strengths, RSD of migration times was only about 1%. Peak area reproducibility was not as good as that for migration times but sufficient for reasonable accuracy in quantitation. Thus it can be concluded that the method developed is well suited for bromide analysis from raw and drinking waters when the sensitivity achieved is sufficient.

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