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Combination of suppressed and non-suppressed ion chromatography with atmospheric pressure ionization mass spectrometry for the determination of anions

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Abstract

Non-suppressed and suppressed ion chromatography in combination with atmospheric pressure ionization mass spectrometry are compared with special respect to sensitivity for the analysis of low-molecular-mass anions. Iodate, bromate, bromide, sulfate, thiosulfate and bromide could be separated by non-suppressed ion chromatography using a low-capacity anion-exchange column and ammonium citrate as mobile phase. Absolute detection limits between 0.4 and 0.7 ng could be achieved; employing a column requiring a flow-rate of 1 ml/min for optimum performance, splitting was necessary so that only 120 μl/min entered the interface of the mass spectrometer resulting in detection limits between 0.03 and 0.06 mg/l. The same stationary phase (packed into a narrow-bore column which allowed operation without splitting) was suitable for the separation of oxyhalides in the suppressed mode with detection limits of 0.5 μg/l (50 pg) with sodium carbonate as eluent. The method was applied to the analysis of drinking water for oxyhalides. The sample pretreatment for the removal of matrix anions (sulfate, chloride and hydrogencarbonate) is described.

Keywords: Water analysis; Inorganic anions; Oxyhalides

1. Introduction

Ion chromatography (IC) is a versatile and widely accepted technique for the analysis of inorganic and organic anions. Beside conductivity, UV absorbance, electrochemical and refractive index detection techniques, mass spectrometry (MS) has gained importance for specific applications because of its advantage of combining high sensitivity with mass selectivity. Recent developments of interfaces for the combination of a liquid chromatographic separation system and MS brought up several new techniques for the analysis of anions using either inductively coupled plasma (ICP) MS, particle beam (PB) MS or atmospheric pressure ionization (API) MS. Eluents described for IC in combination with ICP-MS include potassium nitrate [1], sodium carbonate/hydrogencarbonate [2], ammonium salts of nitrate [3,4], sulfate [5], carbonate [6] and tartrate [7] in the non-suppressed mode or sodium hydroxide [8] in the suppressed mode. Although these eluents are non-volatile some of them are also employed in API-MS measurements in the non-suppressed mode such as ammonium nitrate [9] and ammonium sulfate [10]. Sodium hydroxide [11] and sodium carbonate [12] have been reported for API-MS after eluent suppression. IC–PB-MS analysis can be performed employing ammonium formate as mobile phase [13].

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Among the anions commonly analyzed in water samples, oxyhalides (especially bromate) are of special interest because they may be generated during the disinfection procedures of drinking water containing halides and can involve a certain cancer risk. For bromate the Environmental Protection Agency proposes a maximum concentration of 10 μg/l in drinking water [14], which requires accurate analysis down to the low μg/l level. In this paper methods for the analysis of some oxyhalides as well as other anions are presented in both the non-suppressed and the suppressed IC mode with API-MS detection in order to conduct a critical comparison of the two techniques.

The choice of the mobile phase is strongly influencing the sensitivity in the non-suppressed mode. Especially the kind and concentration of the electrolytes in the eluent affects background noise and may be a reason for low transfer rates of analyte anions from the liquid eluent to the gas phase in the API interface which usually results in low sensitivity. Employing IC in the suppressed mode, the number of mobile phases suitable for the combination of IC and MS are generally limited to eluents such as sodium carbonate and sodium hydroxide; due to the removal of the electrolyte from the mobile phase by suppression the sensitivity of API-MS detection can be expected to increase compared to non-suppressed IC–MS, although a systematic comparison of sensitivities seems to be still missing.

2. Experimental

2.1. Instrumentation

IC was performed on a HP 1100 HPLC System equipped with a vacuum degasser, quaternary pump, UV–Vis diode array detector and a HP 1050 autosampler (all Hewlett-Packard, Palo Alto, CA, USA); MS measurements were done on a quadrupole system HP 5989B using an atmospheric pressure ionization interface HP 59987A (Hewlett-Packard) equipped with a RF-only hexapole (Analytica of Branford, Branford, CT, USA).

2.2. Chemicals

Ammonium formate was purchased from Sigma (St. Louis, MO, USA), sodium carbonate, ammonium carbonate, ammonium sulfate, ammonium acetate, sulfuric acid and formic acid from Merck (Darmstadt, Germany) and acetonitrile from Baker (Phillipsburg, NJ, USA), all of analytical grade.

High-purity water was prepared by a Milli-Q water purification system (Millipore, Milford, USA). One hundred mg/l stock solutions of bromate, iodate, chlorate, bromide, iodide, sulfate and thiosulfate were prepared from their analytical grade sodium salts by dissolving the appropriate amount in Milli-Q water.

2.3. Columns and mobile phases

For the experiments in the non-suppressed IC mode an IC-Pak Anion HR column (Waters, Milford, MA, USA) was employed.

The column for the suppressed IC mode was a 130×2 mm stainless steel column packed with the stationary phase of a Waters IC-Pak Anion HR column by the slurry packing technique. The suspension medium as well as the packing liquid was 5 m*M* sodium carbonate and the packing process was performed at a constant pressure of 160 bar. When no further decrease of the flow was observed, the flow was kept constant for approximately 1 h at about 0.5 ml/min. Afterwards the column was conditioned with the mobile phase for the suppressed IC separation, i.e., 5 m*M* sodium carbonate containing 10% acetonitrile.

The suppressor column was prepared from a 60× 4.6 mm stainless steel column packed with highly crosslinked porous sulfonated polystyrene–divinylbenzene (degree of crosslinkage: 50%, mean particle size: 4 μm, ion-exchange capacity: 1.3 mequiv./g). The slurry packing procedure was performed with methanol as both the suspension and packing medium at a constant pressure of 400 bar. The column was brought into the H^+ form by purging with 50 m*M* sulfuric acid at a flow of 0.8 ml/min for about 30 min. The column was washed with Milli-Q water containing 10% acetonitrile for 45 min prior to use. Beside sulfuric acid, 50% (v/v)

aqueous formic acid was tried as regenerant in a series of experiments.

For non-suppressed IC carbonate and sulfate eluents (prepared from their ammonium salts as 1 m*M* solutions), an oxalate eluent (prepared from 1 m*M* oxalic acid) and a citrate eluent (prepared from a 0.5 m*M* citric acid) were employed. Adjustment of pH was done using a 1 *M* ammonia solution. These solutions were mixed with acetonitrile at a ratio of 90:10 (v/v) so that the final concentration of the carbonate, sulfate and oxalate eluents was 0.9 m*M* and 0.45 m*M* for the citrate eluent. All experiments with these eluents in the non-suppressed mode were performed at pH 9 if not stated otherwise.

IC in the suppressed mode was performed using a 5 m*M* sodium carbonate solution containing 10% acetonitrile at pH 11.3 without any pH adjustment.

2.4. Sample pretreatment for drinking water

Sample pretreatment of water samples included the removal of sulfate, chloride and hydrogencarbonate by solid-phase extraction using a strong cation-exchange (SCX) material. Beside a commercially available SCX cartridge (Bond Elut, 3 ml, Analytichem International) also self-made cartridges were tested using the same cation-exchange material as for the preparation of the suppressor column. Two hundred and fifty mg of these particles was filled into each of three glass cartridges (12 mm I.D.) and conditioned by passing 5 ml of methanol, 10 ml of 50 m*M* sulfuric acid and 15 ml of Milli-Q water through the bed. For sulfate removal one cartridge was then flushed with 5 ml of a 50 m*M* barium nitrate solution, for chloride removal another one was treated with 5 ml of a 100 m*M* silver nitrate solution. Finally the cartridges were washed with 30 ml Milli-Q water to remove the nitrate and excess barium or silver.

Samples of drinking water were first passed through the cartridge in the barium form, then through the one in silver form and at last through the cartridge in the H^+ form. The collected samples were directly injected into the high-performance liquid chromatography (HPLC) system. One cartridge could be used for the pretreatment of at least seven samples without notable decrease of efficiency.

3. Results and discussion

3.1. Separation of anions in the non-suppressed mode

Using conductivity detection, which is the most important detection technique with IC, typically employed eluents for ion-exchange chromatography in the non-suppressed mode are aromatic acids and their salts [15–19], aliphatic carboxylic acids and their salts [19–22], aromatic and aliphatic sulfonic acids and their salts [23–26] as well as borate complexes [26–29].

In this work different compositions of the mobile phase were tested to obtain the highest sensitivity with MS detection. The ideal eluent would consist of a volatile electrolyte of high elution strength so that it can be used at a very low concentration and exhibits minimal interference with the MS detection. Volatile eluents such as ammonium formate and acetate resulted in high retention times when used at a low concentration on the one hand and low sensitivity when used at higher concentrations (up to 100 m*M*) on the other hand. In a series of experiments different non-volatile electrolytes such as ammonium carbonate, ammonium sulfate, ammonium oxalate and ammonium citrate solutions were investigated as possible eluents. Citrate at an alkaline pH exhibited the highest elution strength and promised the best results for the separation of a mixture of both early and late eluting anions like iodate, bromate, bromide, sulfate, thiosulfate and iodide on a Waters IC-Pak Anion HR column with respect to low retention times and high sensitivity. By adjustment of the pH with 1 *M* ammonia solution the retention times of the analytes could be varied in a wide range. Iodide, which is the last ion to elute with citrate as mobile phase, showed a retention time of 15.7 min at pH 5.6 and 7.8 min at pH 10.5. Fig. 1 compares the separation of the above six anions with an eluent consisting of 90% of 0.5 m*M* ammonium citrate at different pH and 10% acetonitrile (in this case the detection was performed by UV absorbance at 210

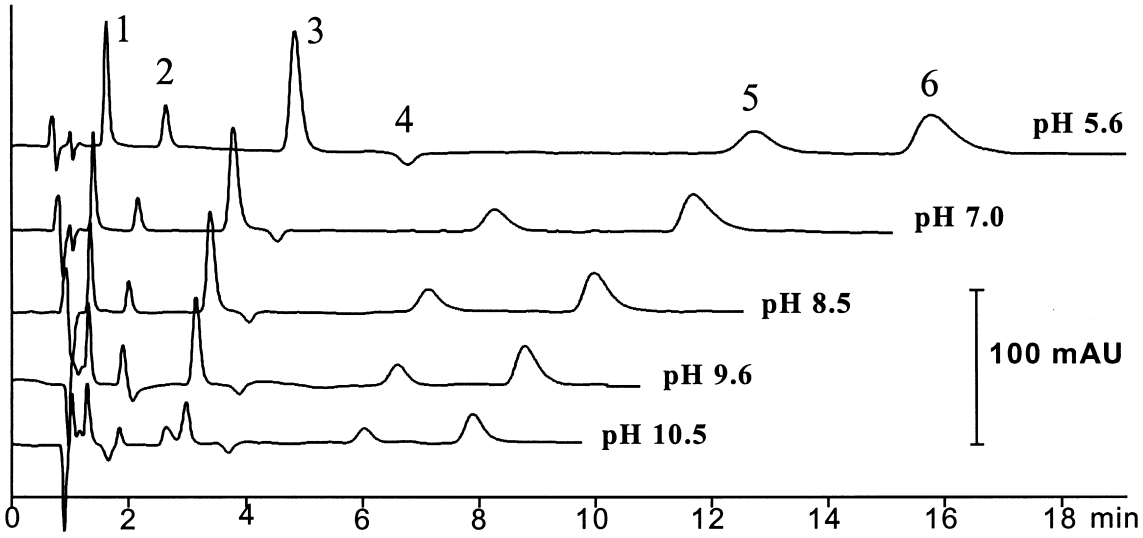

Fig. 1. Influence of mobile phase pH on the retention times of six anions. Column: Waters IC-Pak Anion HR, 75×4.6 mm. Mobile phase: 90% 0.5 m*M* ammonium citrate pH 9, 10% acetonitrile. Flow-rate: 1 ml/min. Injection volume: 10 μl. UV detection at 210 nm. 1=Iodate, 2=bromate, 3=bromide, 4=sulfate, 5=thiosulfate, 6=iodide. Concentrations: 10 mg/l each.

nm). With increasing pH the retention times could be lowered as expected but at pH 9.6 the baseline showed some disturbances after bromate and at pH 10.5 in the region of bromate and bromide (the interferences might partly be due to the presence of carbonate, although more detailed investigations have not been carried out). For the following investigations a pH of 9 was chosen which offered both low retention times and a good baseline stability.

The influence of the citrate concentration (pH 9) and acetonitrile content on the MS detection was investigated by flow injection of standard solutions of iodate, bromide and iodide at a flow-rate of 150 μl/min. As can be seen in Fig. 2, the intensity of the MS signal increased for all analyte anions with increasing acetonitrile content and decreasing citrate concentration. Since the separation column did not allow a higher acetonitrile content than 12%, the mobile phase of choice consisted of 90% of a 0.5 m*M* ammonium citrate solution at pH 9 and 10% (v/v) acetonitrile. Because the column required a flow-rate of about 1 ml/min, the eluent flow had to be split before the MS detector and only 120 μl/min entered the API interface. For optimum sensitivity the highest possible injection volume of the autosampler (100 μl) was applied. With these IC parameters the MS conditions were adjusted for optimum sensitivity. The nebulizing gas pressure was kept at 60 p.s.i. (N_2), the drying gas flow was adjusted to 7 l/min and kept at a temperature of 300°C (1 p.s.i.= 6894.76 Pa). In some cases it proved to be useful not to detect the molecular but fragment ions of the analytes. By carefully adjusting the voltage at the exit of the transfer capillary of the API interface (collisionally induced dissociation, CID) either by the tuning procedure or manually, fragmentation could be enforced or inhibited offering the possibility to increase sensitivity and gain some additional information about the analytes. Monitoring fragment ions beside molecular ions is therefore an important tool for identification and secure assignment of analytes. Finally, iodate was detected at m/z values of 174.9 and 126.9, bromate and also bromide at m/z 78.9 and 80.9, sulfate at m/z 97.0, thiosulfate at m/z 112.9 and iodide at m/z 126.9. Fig. 3 shows an example for a typical separation of anions. Detection limits for a injection volume of 100 μl were found to range from 0.03 mg/l for iodate up to 0.06 mg/l for iodide. Table 1 gives the linearity data and detection limits for six anions in more detail. Each of the standards with different concentrations was injected

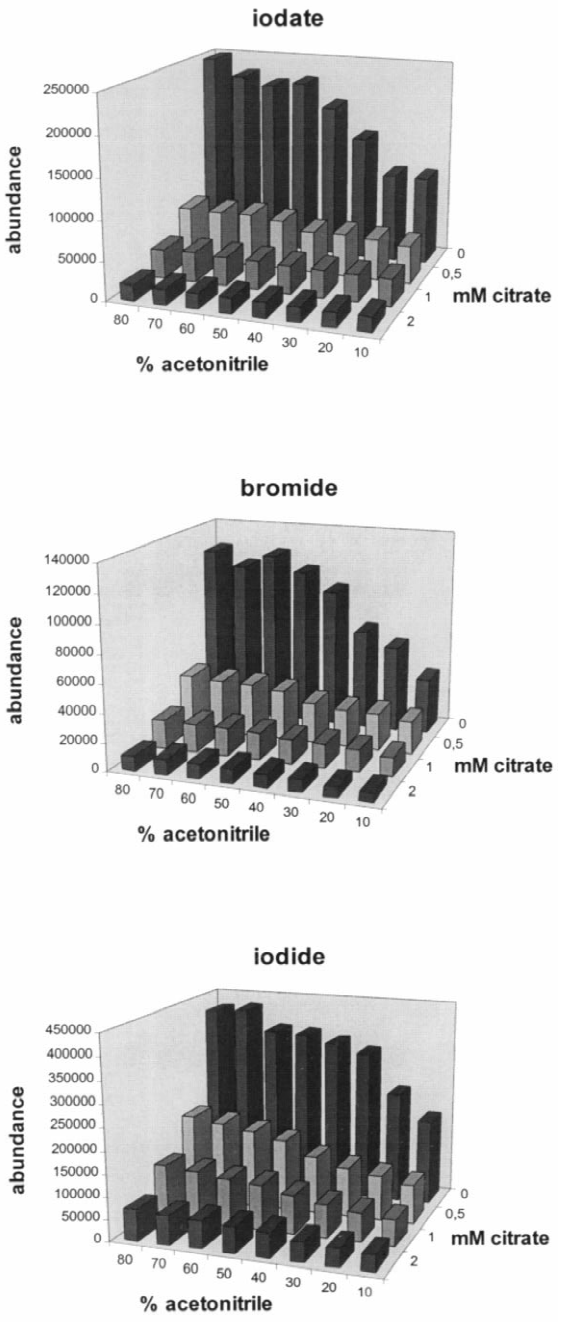

Fig. 2. Dependence of the MS signal intensities of iodate, bromide and iodide on the citrate concentration and the acetonitrile content.

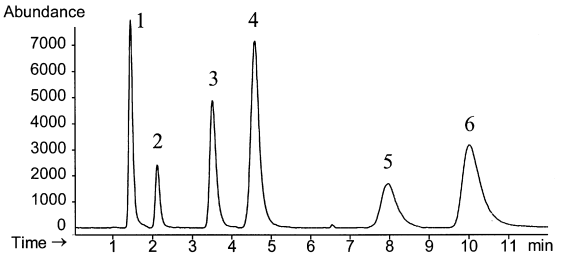

Fig. 3. Separation of anions by ion-exchange chromatography in the non-suppressed mode with API-MS detection after background subtraction. Column: Waters IC-Pak Anion HR, 75×4.6 mm. Mobile phase: 90% 0.5 m*M* ammonium citrate, pH 9, 10% acetonitrile. Flow-rate: 1 ml/min. Injection volume: 10 μl. MS detection. The eluent was split so that only 120 μl/min entered the MS interface. 1=Iodate, 2=bromate, 3=bromide, 4=sulfate, 5=thiosulfate, 6=iodide. Concentrations: 10 mg/l each.

twice and the mean value was used for further calculations; detection limits were calculated by the S/N criterion ($S/N=3$).

The use of ammonium citrate as the eluent did not result in contamination of the API interface during a period of at least 1200 h run time. An explanation could be the possible degradation of citrate at elevated temperatures which are present in the API interface. Interferences with analyte ions in selected ion monitoring (SIM) acquisition were hardly observed because the m/z values of the main fragment ions of citrate (m/z 89 and 131) differed from those of the analyte ions.

3.2. Separation of oxyhalides in the suppressed mode

API-MS detection as described above for non-suppressed IC leads to attractive detection limits that may be sufficient for many applications. On the other hand, these detection limits are not yet low enough for monitoring oxyhalides (especially bromate) in drinking water. Therefore, suppressed IC – a common technique in combination with conductivity detection – was investigated, as the removal of electrolytes from the mobile phase after the separation column is a promising approach to increase sensitivity of MS detection. Suppression was performed using a stainless steel column (60×4.6 mm I.D.) packed with a sulfonated polystyrene–divinylbenzene material which was brought into the

Table 1
Linearity data and detection limits for six anions in the non-suppressed mode[a]

	Linear range (mg/l)	Correlation coefficient	Detection limit ($S/N=3$)
Iodate	0.03–10	0.9997	0.03 mg/l (0.36 ng)
Bromate	0.04–10	0.9996	0.04 mg/l (0.48 ng)
Bromide	0.06–10	0.9999	0.06 mg/l (0.72 ng)
Sulfate	0.05–10	0.9997	0.05 mg/l (0.60 ng)
Thiosulfate	0.05–1	0.9993	0.05 mg/l (0.60 ng)
Iodide	0.06–10	0.9994	0.06 mg/l (0.72 ng)

[a] Number of data points: 14 (two replicates each).

protonated form using 50 m*M* sulfuric acid or 50% (v/v) aqueous formic acid. Although the regeneration column was rinsed with water for half an hour, there was still some sulfate eluting from the suppressor column when using sulfuric acid as regenerant, resulting in a higher background noise and lower sensitivity especially during the first few runs after regeneration. Alternatively, 50% (v/v) aqueous formic acid was tested as regenerant; although formic acid is a rather weak acid compared to sulfuric acid it was sufficient acidic to protonate the sulfonyl groups of the SCX material. The advantage of formic acid over sulfuric acid was the significantly lower background noise and a higher sensitivity even for the first runs after regeneration due to the volatility of formic acid and therefore formic acid was preferred over sulfuric acid as regenerant.

To simplify the instrumentation, splitting was avoided and a 130×2 mm I.D. column packed with the same resin as for the experiments in the non-suppressed mode was used. Since the HPLC system being used throughout this work was not compatible with sodium hydroxide solutions, an aqueous sodium carbonate solution containing 10% acetonitrile for better nebulization was selected as the mobile phase.

The analytes investigated were limited to those with low affinity to the stationary phase, i.e., iodate, bromate and chlorate because of the low elution strength of carbonate. A higher concentration of carbonate in the mobile phase was not expedient because the regeneration cycle of the suppressor column would have decreased to an undesirable level. With 5 m*M* carbonate the suppressor column allowed measurements for approximately 3.5 h without regeneration and therefore this concentration of carbonate was used for further experiments. The eluent flow-rate was adjusted to 150 μl/min. With these conditions the three oxyhalides could be separated within 12 min. The mass spectrometer operating conditions were the same as described for the non-suppressed IC separations except for the SIM acquisition. In the suppressed mode it was favorable to choose m/z values of 174.9, 126.9. and 83.0 for the detection of iodate, bromate and chlorate respectively. In this case CID experiments did not result in higher sensitivity but could be performed for monitoring fragment ions in order to ensure identification of the oxyhalides.

Fig. 4 shows a chromatogram of a standard mixture of iodate, bromate and chlorate (5 μg/l each). Under these experimental conditions linearity and detection limits were determined with standard solutions. Linearity data and detection limits are given in Table 2. Each of the standards with different concentrations was injected twice, detection limits were calculated by the $S/N=3$ criterion and found to be 0.5 μg/l for all analytes (100 μl injected) which corresponds to an absolute detection limit of 50 pg. Comparing the absolute detection limits of IC sepa-

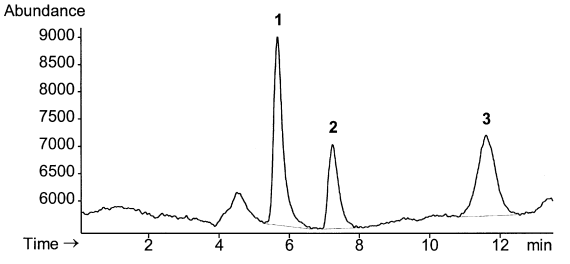

Fig. 4. Chromatogram of a standard mixture of iodate, bromate and chlorate (5 μg/l each) acquired in the suppressed IC mode. Column: Waters IC-Pak Anion HR, 130×2 mm. Mobile phase: 5 m*M* sodium carbonate, 10% acetonitrile. Flow-rate: 150 μl/min. Injection volume: 100 μl. MS detection. 1=Iodate, 2=bromate, 3=chlorate.

Table 2
Linearity data and detection limits for iodate, bromate and chlorate in the suppressed mode[a]

	Linear range (μg/l)	Correlation coefficient	Detection limit ($S/N=3$)
Iodate	0.5–100	0.9997	0.5 μg/l (50 pg)
Bromate	0.5–100	0.9979	0.5 μg/l (50 pg)
Chlorate	0.5–100	0.9998	0.5 μg/l (50 pg)

[a] Number of data points: 10 (two replicates each).

rations in the suppressed mode to those in the non-suppressed mode it may be noted that the increase of sensitivity is almost 10-fold for iodate and bromate using suppressing conditions.

3.3. Application of suppressed IC–MS to the analysis of oxyhalides in drinking water

Determination of oxyhalides like iodate, bromate and chlorate in real samples using the method described above without sample pretreatment is only possible for contents higher than about 100 μg/l. If one wants to analyze these anions at the low μg/l level and sub-μg/l level sample pretreatment is unavoidable. Sulfate elutes closely after chlorate and may lower the peak area of this analyte because of partial overlapping at sulfate concentrations higher than 30 mg/l. Chloride and hydrogencarbonate influence the peak shape and peak area of iodate and bromate to a great extent and have to be removed as well. Nitrate below 50 mg/l showed virtually no influence on the quantification of iodate and bromate although chlorate showed some decrease in peak area because of partially overlapping with the chlorate peak. In the case of nitrate levels higher than 20 mg/l the quantification of chlorate should be performed by standard addition.

Removal of sulfate and chloride was performed by formation of insoluble barium sulfate and silver chloride using solid-phase extraction cartridges in the barium or silver form, respectively.

The commercially available strong cation-exchange cartridges proved to be not suitable for the removal of disturbing matrix anions because precipitated AgCl could not be held back and the sulfate concentration was only lowered to one third of the original value. Therefore cartridges with 250 mg of sulfonated polystyrene–divinylbenzene particles (4 μm diameter) were prepared and brought into Ba^{2+} and Ag^{+} form respectively. With these cartridges it was possible to remove chloride and lower the sulfate concentration down to about 10% of the originally present content. After the following removal of hydrogencarbonate with a cartridge in the H^{+} form the samples could be analyzed with the same sensitivity as standard solutions of oxyhalide ions. Fig. 5 shows the comparison of the chromatograms of spiked drinking water samples before and after removal of sulfate, chloride and hydrogencarbonate. Without sample pretreatment (Fig. 5A) especially the peaks of iodate and bromate show unsymmetrical peak shapes and a high background noise. After removal of matrix anions all analytes

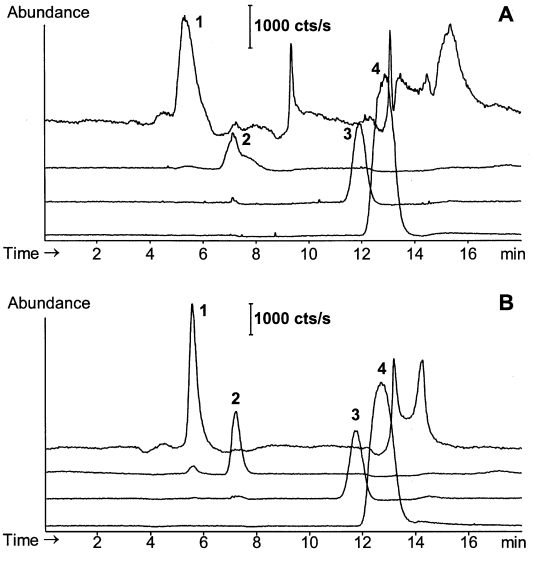

Fig. 5. Comparison of chromatograms (suppressed IC mode) of drinking water spiked with 10 μg/l each of oxyhalide before (A) and after (B) removal of sulfate, chloride and hydrogencarbonate (see Section 3.3 for explanation of additional peaks in chromatogram B). Conditions as in Fig. 4. 1=Iodate, 2=bromate, 3=chlorate, 4=nitrate.

have more symmetrical peak shapes and the background noise is considerably lower (Fig. 5B). The disturbance of the baseline between 13 and 15 min is caused by the residual sulfate which could not be removed in the sample preparation step.

In further experiments the recovery of the oxyhalides was determined using a drinking water sample spiked with 10 μg/l of each oxyhalide. Mean recoveries after removal of sulfate, chloride and hydrogencarbonate were found to be 96.1% for iodate, 95.6% for bromate and 98.7% for chlorate; relative standard deviations were 8.7% for iodate, 6.5% for bromate and 7.3% for chlorate respectively (four replicates). The repeatability for bromate in real water samples was between 1.8 and 5.1% (expressed as relative standard deviation for four different samples and six replicates each). Accuracy will also be checked from the results of a currently running inter-laboratory trial for trace determination of bromate.

4. Conclusions

The combination of IC and API-MS is a versatile analysis technique for the determination of low-molecular-mass inorganic anions. Non-suppressed IC offers the advantage of varying the mobile phase composition in a wide range with respect to the kind of the eluting species and its concentration. The sensitivity of the MS detection depends strongly on the concentration as was demonstrated with ammonium citrate as eluent. Careful optimization of IC and MS conditions is very important to obtain high sensitivity.

In the suppressed IC mode the removal of the electrolytes in the mobile phase before entering the MS interface is connected to an almost 10-fold increase in sensitivity for certain oxyhalides. With a 100 μl injection absolute detection limits of 50 pg are achieved without the need of a concentration column before the separation column or a high-volume injection.

Drawbacks of suppressed IC in the setup used in this work are the limited period of time available for analysis without interruption determined by the regeneration cycle of the suppressor column. Nevertheless detection limits of less than 1 μg/l for certain oxyhalides are a convincing argument to carry on analyzing inorganic anions by IC–API-MS.

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