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Ion chromatographic separation of sulfur-containing inorganic anions with an ICP–MS as element-specific detector

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Abstract

An inductively coupled plasma mass spectrometer (ICP–MS) was used as element-specific detector for sulfide, sulfite, sulfate, and thiosulfate after their chromatographic separation on a Dionex IonPac® AS12A analytical column. The sulfur-containing anions were detected as $^{32}S^{16}O^+$ at $m/z=48$ with the ICP–MS operated with cool and normal plasma conditions, both tuned for the production of high oxide ratios. Three different mobile phases [$(NH_4)_2CO_3$, Na_2CO_3, and NaOH] were tested. The ICP–MS signal at $m/z=48$ for the sulfur-containing anions was obtained only with NaOH based eluents and application of a membrane suppressor for removal of sodium. The detection limits ($S/N=3$, calculated on elemental basis) at optimal chromatographic conditions for sulfide, sulfite, sulfate, and thiosulfate were 35 μg l^{-1}, 200 μg l^{-1}, 130 μg l^{-1}, and 270 μg l^{-1}, respectively. A lower detection limit for sulfide was found to be a result of a higher absolute sulfur amount input into the plasma due to the release of the dissolved H_2S from the aerosol droplets into the gas phase. Severe signal suppression for sulfide with cool plasma and shortening of the sulfide retention with normal plasma were observed in the case of highly saline samples. Both effects were confirmed to be caused by chloride anions. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

The endless list of natural and man-made water pollutants contains also inorganic sulfur-containing anions, such as S^{2-}, SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$. These anions were shown to have diverse impact on plants, animals and humans [1–3], therefore a growing need for their determination appeared recently.

Ion chromatography (IC) coupled with various detection techniques is a method of choice for the simultaneous determination of S^{2-}, SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$ [3–8]. The detection of the sulfur-containing species in real samples after their chromatographic separation can be severely influenced by the high concentrations of co-eluting matrix anions, like chloride, therefore selective detection techniques should be used. The selectivity of the detection can be achieved by exploiting the oxidative properties of sulfur-containing species [3–6], however the selectivity can be deteriorated by other co-eluting oxidizable inorganic anions. Indirect UV–Vis detection of

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sulfur-containing anions after their ion chromatographic separation was recently implemented in three publications of Miura et al. [7–9], by measuring the excess iodine after the postcolumn reaction with sulfur-containing anions [7,8] and by utilizing their catalytic effect on the postcolumn reaction of iodine with azide [9]. Both detection modes are however susceptible to interferences of anions, which can also react with the postcolumn reagents used. Other detection modes that also possess a certain potential for the detection of sulfur-containing anions, were recently reviewed [10].

Among the detection modes that were widely used in recent years for the detection of various ions after their chromatographic separation, ICP–MS was shown to offer unique advantages, like element specificity, a wide linear dynamic range and low detection limits, compared to other methods. The use of liquid chromatography (LC) coupled with an ICP–MS has been reviewed by Byrdy and Caruso [11], Riviello et al. [12] and recently by Sutton et al. [13]. The LC–ICP–MS mainly dealt with the detection of metals and metalloids, whereas among the nonmetallic elements only bromine [14–17] and iodine [17–19] were frequently detected. The applications of LC–ICP–MS for the detection of other nonmetallic elements was reported in the literature only by Jiang and Houk [20] for the detection of some phosphorous and sulfur compounds separated by reversed-phase liquid chromatography.

In this work an inductively coupled plasma mass spectrometer (ICP–MS) was used as an element-specific detector for the determination of the sulfur-containing anions S^{2-}, SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$ after their chromatographic separation on a Dionex IonPac® AS12A separation column. Due to the high background at $m/z=32$ and 34, the sulfur-containing anions were detected as $^{32}S^{16}O^+$ at $m/z=48$ under two different plasma conditions (cool plasma conditions after installation of ShieldTorch System and normal plasma conditions tuned to high oxide formation ratios).

2. Experimental

2.1. Reagents

The reagents used in this study (if not separately specified) were of p.a. grade (Fluka Chemie AG, Buchs, Switzerland). Deionized water with a specific resistance of 18.2 MΩ cm (Milli-Q Plus system, Millipore, Bedford, MA, USA) was used for preparing the solutions and diluting samples in all cases. All standard stock solutions (except chloride) were prepared on a daily basis.

The crystals of $Na_2S\cdot xH_2O$ ($x=7$–9) (Merck, Darmstadt, Germany) were first washed quickly with deionized water to remove surface impurities and immediately dried with a paper tissue. The standard sulfide stock solution containing approx. 1.0 g l^{-1} of sulfur was prepared by dissolving 695 mg of $Na_2S\cdot xH_2O$ ($x=7$–9) in 100 ml of water and standardized by iodometric titration. The standard stock solutions of SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$ containing 1.00 g l^{-1} of S were prepared by dissolving 393.1 mg of Na_2SO_3, 442.9 mg of Na_2SO_4, and 386.9 mg of $Na_2S_2O_3\cdot 5H_2O$ in 100 ml of water, respectively.

The chloride stock solution (50.0 g l^{-1}) was prepared by dissolving 8.24 g of NaCl in 100 ml of water.

The eluent stock solution (100 m*M* NaOH) was prepared by dissolving 4.00 g of NaOH in 1 liter of water. Working eluent solutions (60 m*M*, 70 m*M*, 80 m*M* and 90 m*M* NaOH) were prepared by mixing stock eluent solution with water using the quaternary pump in the chromatographic module.

2.2. Chromatographic conditions

A Hewlett-Packard HP1100 liquid chromatography module (Waldbronn, Germany) equipped with a Dionex (Sunnyvale, CA, USA) IonPac® AG12A (4 mm) guard column and IonPac® AS12A (4 mm) separation column was used. A Dionex anion self-regenerating suppressor ASRS-1 (4 mm, $I=300$ mA, external water mode) was added after the separation column to remove sodium ions. The samples were injected using an HP1100 autosampler equipped with a variable volume injection loop (the injection volume was 50 μl). The eluent and the H_3O^+ donor flow rates were 1.5 ml min^{-1} and 4 ml min^{-1}, respectively. The outlet of ASRS-1 was directly connected with a 70 cm polyether–ether–ketone (PEEK) tubing (I.D.=0.13 mm) to the Babington nebulizer of an HP4500 ICP–MS (Hewlett-Packard, Waldbronn, Germany), which served as an element-specific detector. The data were evaluated with the HP chromatographic software and with Microcal Origin (Microcal Software, USA) software package.

2.3. ICP–MS system

The ICP–MS detector was equipped with a Babington type nebulizer and used in two different configurations. In the case of normal plasma conditions the ICP–MS detector was adjusted (tuned) to obtain high oxide levels. The tuning was done according to the HP Operator's Manual [21] with a 10 μg l^{-1} Ce solution, to which 600 μg l^{-1} of S as SO_4^{2-} was added. The operating parameters were adjusted by monitoring signals at $m/z=48$ ($^{32}S^{16}O^+$), 140 ($^{140}Ce^+$) and 156 ($^{140}Ce^{16}O^+$) with the sampling time of 0.1 s for each monitored mass-to-charge ratio. The operating conditions producing the highest $^{140}Ce^{16}O/^{140}Ce$ oxide ratio of approx. 5000%. were: RF power 1350 W, reflected power <2 W, plasma gas 14.8 l min^{-1}, auxiliary gas 0.93 l min^{-1}, carrier gas 1.30 l min^{-1}, cooling temperature 0°C. The average count per second ($n=200$) at $m/z=48$, obtained for 600 μg l^{-1} of S as SO_4^{2-} in the tuning solution and for Milli-Q water (background), were 329 000 and 5600, respectively.

The operating parameters with cool plasma conditions were obtained by installing the ShieldTorch System and tuning using a 10 μg l^{-1} Co^{2+} solution. The additional tuning to obtain a maximum signal for $^{32}S^{16}O^+$ ion was performed on a daily basis by a sulfide solution containing 1 mg l^{-1} of S. The operating conditions for cool plasma operation were: RF power 730 W, reflected power <2 W, plasma gas 15.4 l min^{-1}, auxiliary gas 0.93 l min^{-1}, carrier gas 1.13 l min^{-1}, blend gas (argon) 0.48 l min^{-1}, cooling temperature 2°C.

3. Results and discussion

Sulfur is an element, which is difficult to determine by ICP–MS, because only approx. 15% is ionized in the argon plasma (1^{st} ionization potential is 10.360 eV) [22]. For the detection of sulfur with ICP–MS four sulfur isotopes (Table 1) are available. However, the two major sulfur isotopes ^{32}S (95.018%) and ^{34}S (4.215%) suffer from several interferences (especially severe oxygen interferences, Table 1), whereas the abundances of the other two sulfur isotopes ^{33}S (0.760%) and ^{36}S (0.014%) are not useful for analysis at low concentrations.

Due to the fact that sulfur readily forms oxides, sulfur can be detected as its oxide $^{32}S^{16}O^+$ at $m/z=48$. In this work plasma conditions for production of sulfur oxides were obtained with cool plasma after installation of ShieldTorch System and with normal plasma tuned to high oxide formation ratios. Both mentioned plasma conditions were applied for the detection of sulfur-containing anions at $m/z=48$ after their ion chromatographic separation. It should be noted that the theoretical calcium and titanium interference at $m/z=48$ were ruled out by additional monitoring of ^{43}Ca and ^{46}Ti, ^{47}Ti, ^{49}Ti, and ^{50}Ti.

3.1. Choice of the eluent type

Frequently used mobile phases (eluents) in ion-exchange separations of anions (especially in connection with the suppressor and subsequent conductometric detection) are different $Na_2CO_3/NaHCO_3$ buffer solutions [23]. When ICP–MS is employed as element-specific detector the solutes in the mobile phase should be converted to volatile compounds during their passage through the high temperature region of the plasma to avoid clogging of the cone orifices. Therefore, sodium containing mobile phases

Table 1
Relative abundance of sulfur isotopes and possible interferences in ICP–MS detection

Isotope	Relative abundance (%)	m/z	Possible interferences
^{32}S	95.018	32	$^{16}O_2$, $^{14}N^{18}O$, $^{15}N^{17}O$, $^{31}P^1H$
^{33}S	0.760	33	$^{16}O^{17}O$, $^{32}S^1H$
^{34}S	4.215	34	$^{17}O_2$, $^{16}O^{18}O$, $^{33}S^1H$
^{36}S	0.014	36	$^{18}O_2$, $^{35}Cl^1H$, ^{36}Ar

should be replaced with ammonium containing mobile phases.

It is known that high sample carbon input influences the plasma processes and leads to the suppression of the ICP–MS signals [24]. At the beginning, the results of a direct ICP–MS analysis of solutions containing 10 mg l^{-1} of S as SO_4^{2-} in two different carbonate matrices [3 m*M* $(NH_4)_2CO_3$ and 3 m*M* Na_2CO_3] were compared with a result obtained for a solution of 10 mg l^{-1} of S as SO_4^{2-} in Milli-Q water. The sample solutions were fed to the Babington nebulizer via a peristaltic pump at a flow rate of 0.4 ml min^{-1}. Opposite to the water solution, the absence of the signal for $^{32}S^{16}O^+$ was found in both cases of carbonate matrices, therefore high carbonate input into the plasma under cool plasma conditions should be avoided. Since the sodium input into the plasma is not desired as well due to clogging of the sample cones, appropriate chromatographic conditions had to be chosen. Such conditions were achieved with a NaOH eluent and by the removal of the sodium ions prior to the ICP–MS detector with the self-regenerating membrane suppressor installed after the chromatographic column.

3.2. Chromatographic separation of S^{2-}, SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$ with NaOH based eluents

The following set of experiments was performed to obtain a chromatographic separation of S^{2-}, SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$ using NaOH based eluents. The sulfur-containing anions were detected with normal plasma tuned to high oxide ratios. It should be noted, that the highest possible NaOH concentration was 100 m*M*, because at higher concentrations the capacity of the self-regenerating membrane suppressor was overcome.

The influence of the eluent ion concentration $[E^{y-}]$ on the capacity factor k'_A, which is a measure for the retention time of the selected analyte ion A^{x-}, can be expressed as follows [25]:

$$\log k'_A = \frac{1}{y}\log K_{A,OH} + \frac{x}{y}\log\frac{Q}{y} + \log\frac{w}{V} - \frac{x}{y}\log[E^{y-}] \tag{1}$$

where $K_{A,OH}$ is the selectivity coefficient, Q is the ion-exchange capacity of the column, w is the mass of the stationary phase, V is the volume of the mobile phase and x and y are the charges of the analyte and eluent ion. If the analyte ion A^{x-} and the separation column are the same ($K_{A,OH}$, Q, w, and V are constant), whereas $[E^{y-}]$ is changed, Eq. (1) can be rewritten:

$$\log k'_A = C_1 - \frac{x}{y}\log[E^{y-}] \tag{2}$$

where C_1 is a constant term. Eq. (2) shows that a plot of $\log(k'_A)$ vs. $\log[E^{y-}]$ results in a linear relationship with a slope equal to the ratio of the charge numbers of the analyte and eluent ion.

The effect of $[OH^-]$ on the retention times for S^{2-} ($pK_1 = 7.04$, $pK_2 = 11.96$ [22]), SO_3^{2-} ($pK_1 = 1.81$, $pK_2 = 6.91$ [22]), and SO_4^{2-} ($pK_2 = 1.92$ [22]) can be observed in Fig. 1. In accordance with the theory linear relationships for the plot of $\log(k'_A)$ *vs.* $\log[OH^-]$ were obtained for S^{2-}, SO_3^{2-}, and SO_4^{2-} with the slopes of -1.0, -1.9, and -1.9, respectively (see the inset in Fig. 1). The slope of -1.0 obtained for S^{2-} indicates, that the S^{2-} was actually separated as a singly charged HS^-, whereas the other two sulfur-containing anions SO_3^{2-} and SO_4^{2-} were both separated as doubly charged anions.

The eluent that was chosen for the separation of S^{2-}, SO_3^{2-}, and SO_4^{2-} in the following experiments was 60 m*M* NaOH, because at higher NaOH concentrations unsatisfactory resolution between SO_3^{2-} and SO_4^{2-} was obtained due to their close retention times. The retention times for S^{2-}, SO_3^{2-}, and SO_4^{2-} obtained with the 60 m*M* NaOH eluent solution were 3.0, 6.5, and 7.7 min, respectively. The elution of the thiosulfate anion $S_2O_3^{2-}$ resulted in a very broad peak at a long retention time of 23.5 min with 60 m*M* NaOH eluent, which is not desirable for routine measurements. Therefore, the highest possible eluent concentration (100 m*M* NaOH) was used to elute $S_2O_3^{2-}$ after the elution of the other three sulfur-containing anions. Under this conditions the $S_2O_3^{2-}$ eluted within a shorter time frame at the retention time of 16.1 min. A chromatogram obtained for the mixture of the four sulfur-containing anions using a two-step isocratic separation conditions is shown on Fig. 2.

3.3. Calibration plots and detection limits for S^{2-}, SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$

The calibrations graphs, plotted as peak area vs.

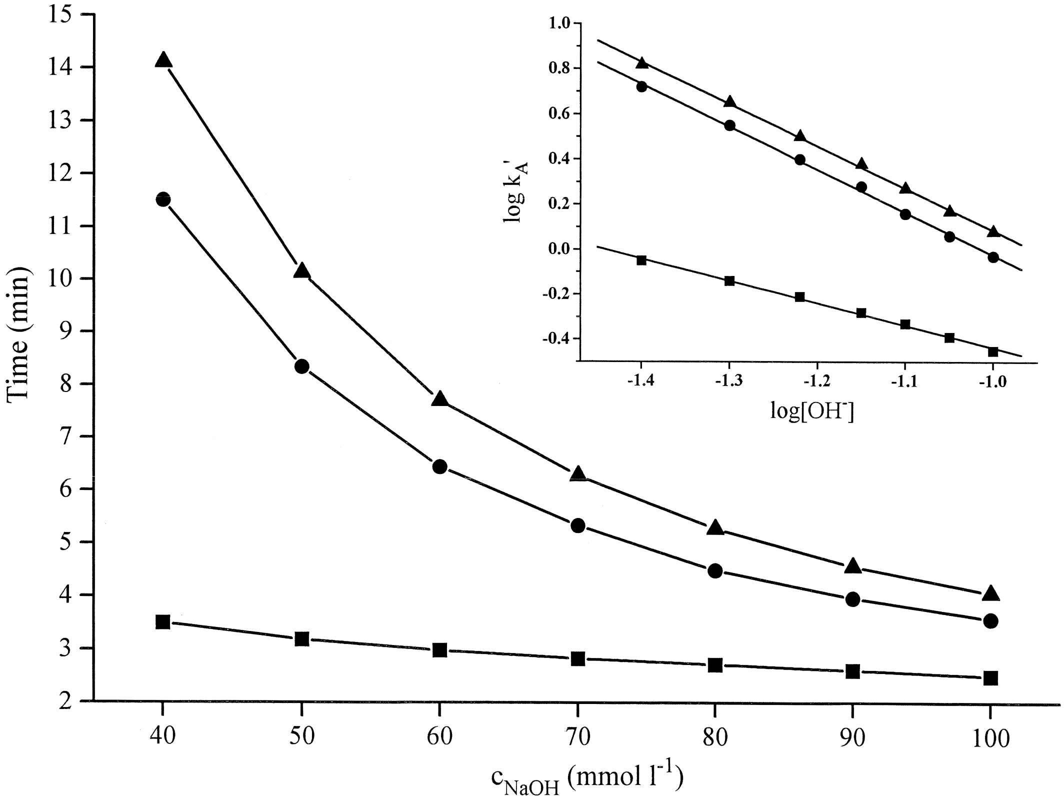

Fig. 1. The influence of NaOH concentration on the retention times of sulfur-containing anions (inset: the plot of log(k') vs. log[OH^-]) (■ – S^{2-}, ● – SO_3^{2-}, ▲ – SO_4^{2-}). Column: Dionex AS12A; Flow rate, 1.5 ml min^{-1}; Sample, 0.5 mg l^{-1} S^{2-}, 5 mg l^{-1} SO_3^{2-}, 5 mg l^{-1} SO_4^{2-}, and 5 mg l^{-1} $S_2O_3^{2-}$ (calculated on elemental basis); Injection volume, 50 μl.

concentration (calculated on elemental basis), gave linear calibration curves up to 50 mg l^{-1} for S^{2-} and 100 mg l^{-1} for the other three sulfur-containing anions. The detection limits ($S/N=3$, injection loop 50 μl) calculated on elemental basis for S^{2-}, SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$ were 35 μg l^{-1}, 200 μg l^{-1}, 130 μg l^{-1}, and 270 μg l^{-1}, respectively.

The higher response for S^{2-} in comparison to the other three investigated anions can be explained taking the dissociation constants of H_2S ($pK_1=7.04$, $pK_2=11.96$ [22]) and the pH of the effluent coming from the membrane suppressor ($pH_{measured}$ 5.3) into account. In solutions (pH<6) dissolved H_2S is the predominant sulfide species. A portion of the dissolved H_2S can be released into the gas phase from the aerosol cloud formed by the nebulizer and directly carried by the carrier argon into the plasma. Therefore, in comparison with SO_3^{2-}, SO_4^{2-}, and $S_2O_3^{2-}$, an additional amount of sulfur is introduced to the plasma. The net result is the higher peak area for the same total sulfur concentration in the case of S^{2-}. The confirmation of this hypothesis was obtained in an experiment, where a T-piece was installed after the membrane suppressor for the post-column addition of 40 m*M* HNO_3 solution. The 40 m*M* HNO_3 solution was added at a flow rate of 0.5 ml min^{-1} by means of a Dionex post-column reagent delivery module. The consequence of the post-column addition of 40 m*M* HNO_3 on the sulfide peak can be seen in Fig. 3.

The post-column addition of HNO_3 resulted in a solution with pH 2.0, in which more sulfide is present in form of dissolved H_2S compared to the solution with pH 5.3 (obtained with the post-column addition of water to enable direct comparison). Consequently, more H_2S is evaporated into the gas phase and a signal with a higher peak height can be observed (Fig. 3).

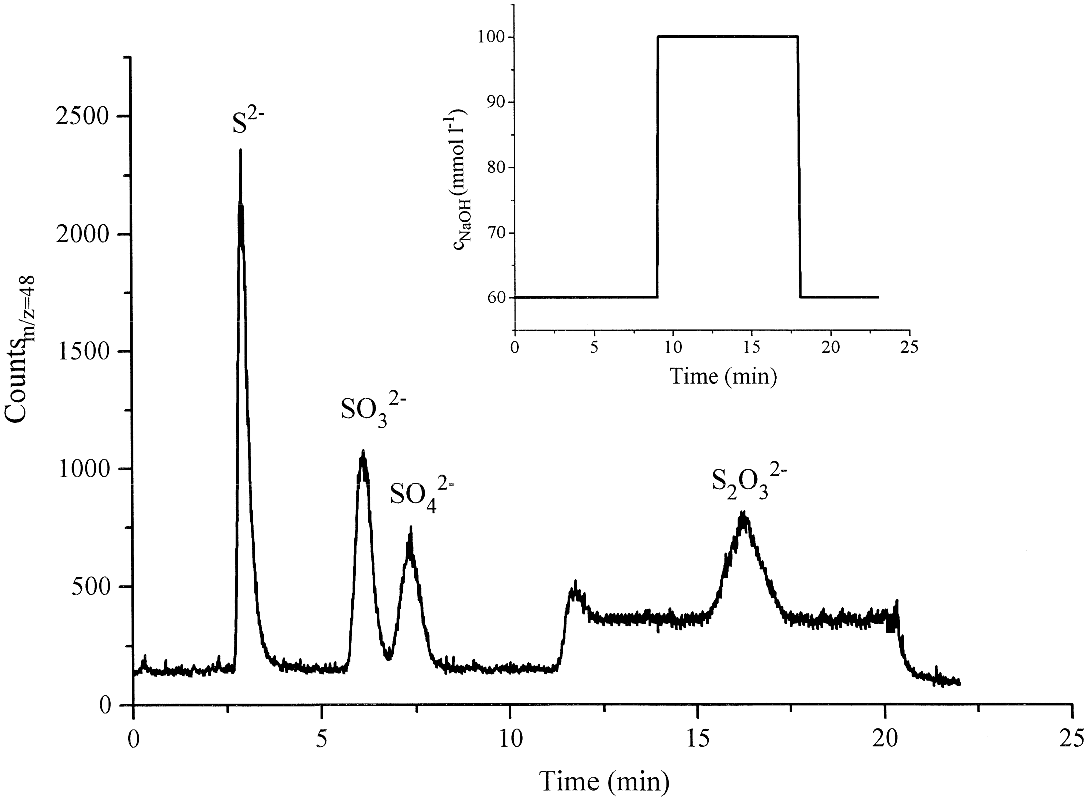

Fig. 2. Chromatogram of a standard solution of sulfur-containing anions (inset: two-step isocratic eluent program). Eluent: 0–9 min 60 m*M* NaOH/9.1–18 min 100 m*M* NaOH/18.1–23 min 60 m*M* NaOH; other conditions as in Fig. 1.

3.4. Interferences of high sample matrix concentrations of other ions

The chromatographic resolution between the four sulfur-containing anions and other anions, such as common inorganic anions (Cl^-, Br^-, NO_3^-, and PO_4^{3-}), is in the case of the implementation of an element-specific detector, not a prerequisite for the detection of the analyte anions. Nevertheless, the co-eluted matrix anions can influence both the elution and the detection of the analyte anions.

It was found in this work that Cl^- and S^{2-} were not separated at the chromatographic conditions used (60 m*M* NaOH). Therefore the influence of Cl^- on the chromatographic behavior and the detection of S^{2-} was investigated by analyzing solutions containing 1 mg l^{-1} of S as S^{2-} in a chloride matrix at both cool and normal plasma conditions. A severe signal suppression for S^{2-} was obtained with the cool plasma for samples containing chloride above 0.5 g l^{-1} (Fig. 4A). The ionization of high amounts of chloride, which co-elutes with S^{2-}, lowered the plasma energy needed for production and ionization of SO fragment, hence severe signal suppression was observed. The signal suppression under normal plasma conditions was not observed even at the chloride concentration of 5 g l^{-1}, but an effect of shortening of the retention time for S^{2-} was observed (Fig. 4B). This effect resembles the self-elution effect, which was first mentioned by Jackson and Haddad [26].

The shortening or prolonging of the retention times of the analyte anions was recently shown by Novic et al. [27] to occur, when samples with high matrix anion concentrations (sea water, body fluids, meat and milk extracts, mineral waters, etc.) were analyzed. The self-elution effect was shown to occur, when the matrix anion possessed significantly higher affinity towards the stationary phase than the eluent

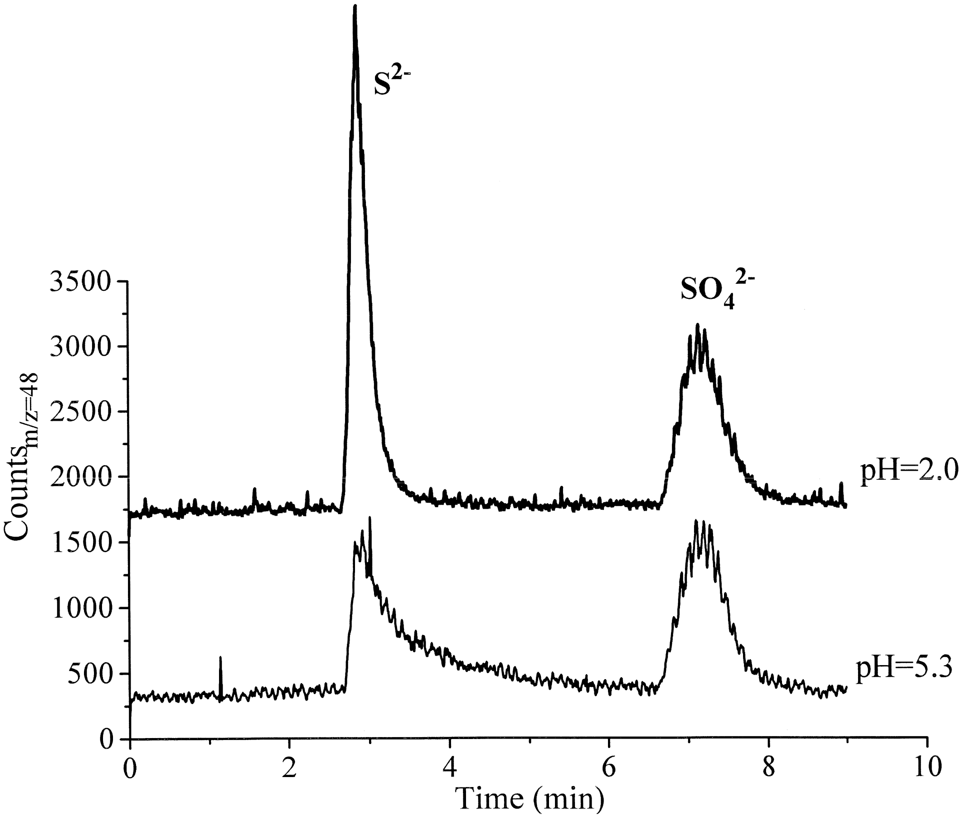

Fig. 3. Influence of the effluent pH on the detection response for S^{2-}. Column: Dionex AS12A; Eluent, 60 m*M* NaOH; Flow rate, 1.5 ml min^{-1}; Sample, 0.5 mg l^{-1} S^{2-} and 10 mg l^{-1} SO_4^{2-}; Injection volume, 50 μl.

ion. In the case of Cl^- and OH^- as the matrix and eluent anions, respectively, the conditions for the self-elution effect mentioned in the literature [27] are fulfilled and accordingly, shorter retention time for S^{2-} in matrixes with high chloride concentrations were observed. It should be mentioned that the retention times of SO_3^{2-}, SO_4^{2-} and $S_2O_3^{2-}$ were not changed at chloride concentrations up to 5 g l^{-1}.

Other sample matrices, such as biological matrices with high Ca content, phosphate matrices or carbonate matrices, can also occasionally be encountered. All three mentioned matrices can produce a signal at $m/z=48$ and can therefore theoretically influence the detection of sulfur-containing anions. The elution of calcium within void volume can produce a signal at $m/z=48$ (^{48}Ca), which would envelope the sulfide peak at high calcium sample concentrations. However, this interference was already avoided considering the chromatographic conditions used in this work, because the anion self-regenerating suppressor quantitatively removes the calcium cations from the column effluent (provided the suppressors capacity is not exceeded). Nevertheless, the possibility of calcium interference was also experimentally checked by analysing samples with sulfide concentration of 0.5 mg l^{-1} in different calcium matrices (0–200 mg l^{-1} Ca^{2+}). Interference of ^{48}Ca on S^{2-} was not observed.

In the case of phosphate matrix, PO_4^{3-} was found to be well separated from the investigated sulfur-containing anions (retention time 14.9 min), therefore the possible interference of $^{31}P^{16}O^{1}H^+$ at $m/z=48$ was excluded. The influence of carbonate matrices was investigated by analysing samples with sulfide concentration of 0.5 mg l^{-1} in different carbonate matrices (0–11 m*M* CO_3^{2-}). No interference was

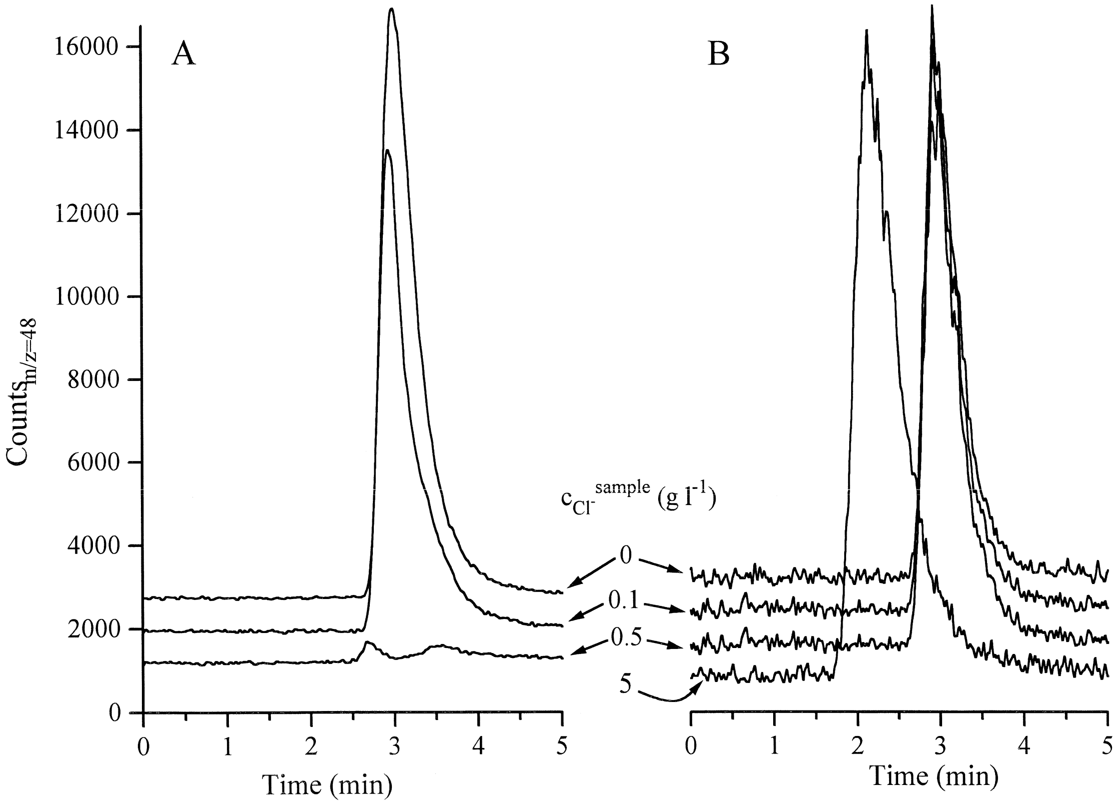

Fig. 4. Chromatograms of solutions containing 1 mg l^{-1} S^{2-} and different chloride concentrations (A – cool plasma, B – normal plasma). Column: Dionex AS12A; Eluent, 60 m*M* NaOH; Flow rate, 1.5 ml min^{-1}; Injection volume, 50 μl.

found at carbonate concentration below 8 m*M* CO_3^{2-}. At 11 m*M* CO_3^{2-} in the sample severe signal suppression for S^{2-} was observed.

4. Conclusions

In the present work S^{2-}, SO_3^{2-}, SO_4^{2-} and $S_2O_3^{2-}$ were separated using NaOH based eluent on Dionex IonPac® AS12A separation column and detected with an ICP–MS as element-specific detector at $m/z=48$ ($^{32}S^{16}O^+$). One of the major advantages of ICP–MS connected to ion chromatography is that the chromatographic separation of the analyte and other ions is not a prerequisite for the detection of the analyte ions, as long as the samples contain comparable concentration levels of analyte ions and potentially co-eluting ions. In the opposite case, the co-eluting ions may have a profound influence on the detection as well as on the retention times of the analyte ions. This was shown in our work with the interference of chloride with sulfide anions. In such cases the use of calibration curves for the routine determinations is excluded and the standard addition method has to be applied. Additionally, one should be aware of the fact that the influence of interfering ions on the retention times of analyte ions makes the use of automatic peak detection options provided by modern chromatographic software impossible. The chromatographic conditions used in this work were found to be applicable for the analysis of investigated sulfur-containing anions in samples with biological matrices.

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