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Journal of Chromatography A, 918 (2001) 411–421

JOURNAL OF
CHROMATOGRAPHY A

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Capillary zone electrophoresis for the determination of light-absorbing anions in environmental samples[☆]

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Received 20 November 2000; received in revised form 3 March 2001; accepted 12 March 2001

Abstract

A rapid and simple method for separation and determination of inorganic anions by capillary zone electrophoresis was described. The detection was carried out directly with a diode array detector. The experimental conditions, such as concentration of carrier electrolyte, capillary length, voltage, and temperature were optimized. In order to improve selectivity, different organic modifiers were also investigated. The baseline separation of 10 light-absorbing anions was accomplished within 3.5 min with a background electrolyte consisting of 50 mM sodium tetraborate containing 5% MeOH. Linear plots were obtained in the concentration range of 0.1–10 µg/ml. With sample stacking injection, the quantitation limits of the anions were found to be in the range of 0.02–0.1 µg/ml. The proposed method was successfully applied to the determination of inorganic anions in environmental samples and in effluents of a power plant. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Buffer composition; Water analysis; Environmental analysis; Inorganic anions

1. Introduction

The demand for a reliable and rapid method for the determination of inorganic anions has increased because of the toxicity of some of these compounds [1,2]. Inorganic anions are usually analyzed using ion chromatography (IC), but in the last decade, capillary electrophoresis (CE) has been shown a powerful tool for inorganic ion analysis [3]. This methodology appeared to be a viable alternative to

ion chromatography for the determination of inorganic ions and other low-molecular-mass ionic solutes [4]. CE offers a number of advantages over ion chromatography, including simplicity, greater separation efficiency, shorter analysis time, and lower consumption of reagents, all of which make it ideal for the analysis of ionic solutes in very complex matrices, such as environmental samples [5,6].

Usually, in order to obtain high-speed electrophoretic separation of anions, the co-electroosmotic mode has been developed by the addition of various cationic surfactants to the buffer electrolyte to reverse the electroosmotic flow (EOF) [7–9]. Other authors used polymer-coated capillaries to suppress the EOF and to avoid wall adsorption [10,11]. However, both in the EOF reversal and in suppres-

[☆]Presented at the 29th Scientific Meeting of the Spanish Group of Chromatography and Related Techniques, Alcalá de Henares (Madrid), 12–14 July 2000.

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sing techniques, complicated buffers have to be used. Ion analysis by CE has generally utilized indirect detection methods because most anions lack a chromophore [12,13]. In the determination of a specific trace-level anion in a sample containing high concentrations of other non-absorbing anions, it is preferable to use direct UV detection provided that the trace-level anion shows UV absorption, whereas the high-concentration anions do not [14].

The aim of this work is to develop a feasible method for determination of absorbing anions of potential interest in a variety of environmental samples. For this purpose, sodium tetraborate at pH 9.3 was used as carrier electrolyte. Reduction of electroosmotic flow to near zero values may be achieved by selecting relatively high buffer concentrations, thus eliminating the need for an external flow modifier or having to work with low pH values [15]. A systematic investigation of instrument parameters such as: effective capillary length, voltage and temperature were performed. The addition of diverse organic solvents to the buffer has also been studied to improve separation selectivity.

2. Experimental

2.1. Chemicals

All standards and buffers were prepared with analytical reagent grade chemicals and 18 M deionized water obtained from a Milli-Q system (Millipore, Milford, MA, USA). Sodium or potassium salts were purchased from Merck (Darmstadt, Germany) and used to prepare 1000 µg/ml stock standard anion solutions containing a single anion. Mixed anion solutions were prepared with deionized water daily. Polyethylene containers were utilized for the anion solutions and samples. Methanol (MeOH), tetrahydrofuran (THF), acetonitrile (MeCN), acetone, 2-propanol and *n*-butanol were HPLC grade and were obtained from Romil (Cambridge, UK). Solutions of sodium hydroxide 1 M and 0.1 M and 50 mM tetraborate buffer solution, pH 9.3 for CE were obtained from Hewlett-Packard (Waldbronn, Germany).

All organic modifier-containing buffers were filtered and degassed by sonication before use.

2.2. Apparatus and procedures

All experiments were carried out on a HP^{3D}CE capillary electrophoresis system (Hewlett-Packard, Palo Alto, CA, USA) fitted with an on-column diode array detector. Spectra were acquired in the range 200–400 nm using 200 nm for signal monitoring.

Polyimide-coated fused-silica capillaries of 22 cm effective length × 50 µm I.D. were prepared from a large capillary supplied by Composite Metal Service (Teknocroma, Barcelona, Spain). On-column detection windows were made by burning a small section (0.5 cm) of the external polyimide coating and removing the burned residue with methanol. Newly installed capillaries were flushed with 1.0 M NaOH for 10 min, with 0.1 M NaOH for 10 min, with deionized water for 5 min and finally rinsed with buffer for 30 min. Support buffer was 50 mM tetraborate at pH 9.3 with organic modifier (5%, v/v, methanol). Capillary temperature was maintained at 20°C and separation voltage was –23 kV (current was –150 µA). Samples and standards were injected by pressure of 20 s at 50 mbar. A 3-min capillary purge with carrier electrolyte solution was performed prior to each injection.

3. Results and discussion

3.1. Selection of carrier buffer

The electrolyte composition has an important effect on the mobility of the compounds and on electroosmotic flow within the capillary. For the present study, sodium tetraborate has been selected as buffer, because it is one of the most commonly used and easily available in analytical laboratories of CE. Moreover, this buffer presents low absorbance at working wavelengths, does not require special preparation and a pH of 9.3 is compatible with most of the samples to be analyzed.

The determination of low-molecular-mass anions by CZE is usually carried out by placing the detector at the anode end and reducing or reversing the electroosmotic flow to achieve shorter analysis times. As mentioned previously, there are different ways of reducing this electroosmotic flow, such as using modified-wall capillaries, addition of different re-

agents to the buffer, especially cationic tensoactive agents or working with low pH values [16]. A very simple way of reducing electroosmotic flow is by using relatively high buffer concentrations [17,18]. Moreover, this process has another advantage in that it limits the coulometric interactions of the analytes with the capillary wall.

The effect of tetraborate concentration on the separation was examined increasing the concentration from 20 to 50 mM, as shown in Fig. 1. A substantial baseline drift is observed when using a

tetraborate concentration of 20 mM, the slower anions do not reach the detector, also producing a distortion of the peaks. Apparent electrophoretic mobility of the anions is also affected by the ionic strength of the buffer. This effect is especially noticeable for thiosulfate, that at low concentrations migrates before nitrite, at 40 mM both ions overlap partially and at 50 mM, the order of migration is reversed. Ferrocyanide is another ion that undergoes major variations on its mobility. This anion elutes before the nitrate ion at tetraborate concentrations of

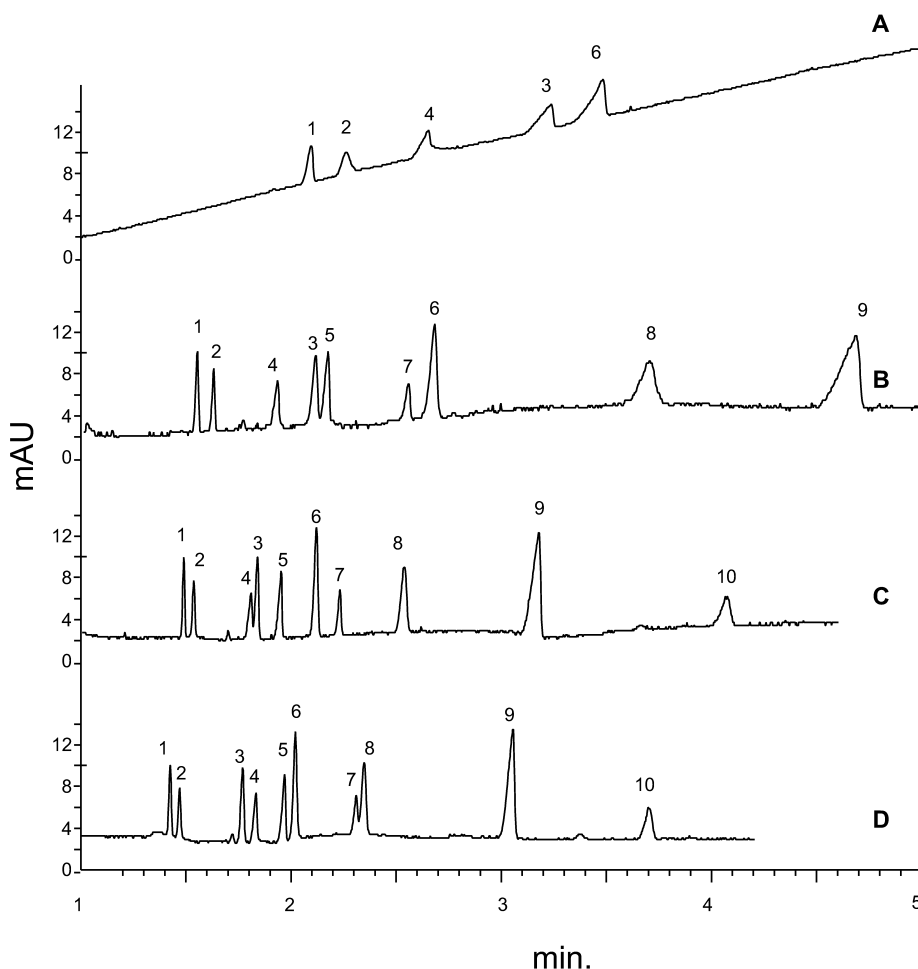


Fig. 1. Effect of tetraborate concentration (pH 9.3) on separation of anions using direct UV detection. (A) 20 mM; (B) 30 mM; (C) 40 mM and (D) 50 mM. Peaks: (1) Br^- , (2) I^- , (3) NO_2^- , (4) $\text{S}_2\text{O}_3^{2-}$, (5) CrO_4^{2-} , (6) NO_3^- , (7) $\text{Fe}(\text{CN})_6^{4-}$, (8) SCN^- , (9) MoO_4^{2-} , (10) WO_4^{2-} . Experimental conditions: capillary internal diameter, 50 μm ; capillary effective length, 22 cm; applied current, $-150 \mu\text{A}$ (-23KV); capillary temperature, 20°C ; injection pressure, 50 mbar during 20 s; detection, 200 nm. Concentration of the anions: 1 $\mu\text{g}/\text{ml}$ of each analyte.

20 mM and it is closer to sulfocyanide when working at 50 mM. Instead, the selectivity between univalent anions remains similar. This observation is consistent with predictions of Li et al. [19] based on the Pitts equation. This equation states that the mobility of multiply charged anion will be more significantly affected by the ionic strength, thus the impact of changes in the ionic strength of the buffer are directly related to the charge on the anion. This is due to the deviations on the real radii of the anions when the analyte is dissolved in concentrate buffers. The mobility of an anion can be also influenced by the ion association with the buffer counter ion (Na^+). The electrophoretic mobility of most of the monovalent and divalent anions do not change significantly even when Na^+ is present in large amounts. With multivalent anions there is generally stronger ion association [20], and their ($-\mu_{\text{ep}}^*$) values decreased with an increase in Na^+ concentrations. This can explain the change observed for $\text{Fe}(\text{CN}_6)^{4-}$. A good baseline and an acceptable resolution of the ten anions under study was achieved when 50 mM tetraborate was selected as carrier electrolyte.

3.2. Study of various instrumental parameters in the separation

3.2.1. Capillary length

In order to obtain lower migration times, different effective capillary lengths were tested briefly: 40, 22 and 8 cm. The two latter lengths are obtained by using the same capillary. The 8-cm effective length was obtained injecting through the outlet end. The analysis time is reduced as the effective length decreases, but the efficacy of the system also decreases. A working length of 22 cm has been selected given that with 8 cm not all the components of the sample can be resolved.

3.2.2. Influence of the applied potential

Although the most commonly used practice when working with CZE is to maintain the voltage constant during the analysis, in this study the applied current is maintained constant instead, favoring the reproducibility of the migration times [17]. To study the effect of this parameter on the separation of the anions, the current was progressively increases from

–100 to –175 μA (Fig. 2). As expected, the analysis time is reduced and the separation efficiency is enhanced. Moreover, the apparent mobility of certain anions also undergoes changes, especially those with greater effective charge. With –100 μA the analysis time is above 5 min, nitrate and chromate ions do not resolve to baseline and ferrocyanide and thiocyanate comigrate. When –125 μA is applied, the analysis time is reduced by more than 1 min. Now the pairs nitrate–chromate and ferrocyanide–sulfocyanide are not resolved. For –150 μA the separation of the 10 compounds is obtained in 3.7 min. Now chromate migrates before nitrate, both ions separating at the baseline and ferrocyanide and thiocyanate are partially resolved. When the current is increased to –175 μA the analysis time practically does not improve and although a better separation of ferrocyanide and thiocyanate is achieved, resolution is lost between nitrate and thiosulfate. No undesirable effects due to Joule heating were observed. In addition, the selectivity was enhanced increasing the applied current. In view of these results –150 μA will be selected as the current value to be applied during the remaining analysis.

3.2.3. Effect of temperature

The effect of temperature of the capillary was studied in the range 20–40°C. For this specific case, temperature does not play a decisive role in the mobility of the anions. In fact, only lower resolution is appreciated between nitrite and thiosulfate as temperature increases. Therefore, during the analysis the capillary temperature was kept thermostated at 20°C.

3.3. Influence of diverse organic modifiers on selectivity

The use of organic modifiers is relatively common in CE to improve the resolution of diverse species, especially metallic ions [21,22]; however, they are used less in the determination of anions [23]. In this work, the influence of several organic solvents such as methanol, acetonitrile, acetone, tetrahydrofuran, isopropanol and butanol on the resolution of 10 anions has been studied. For this, different amounts of solvent were added systematically until achieving

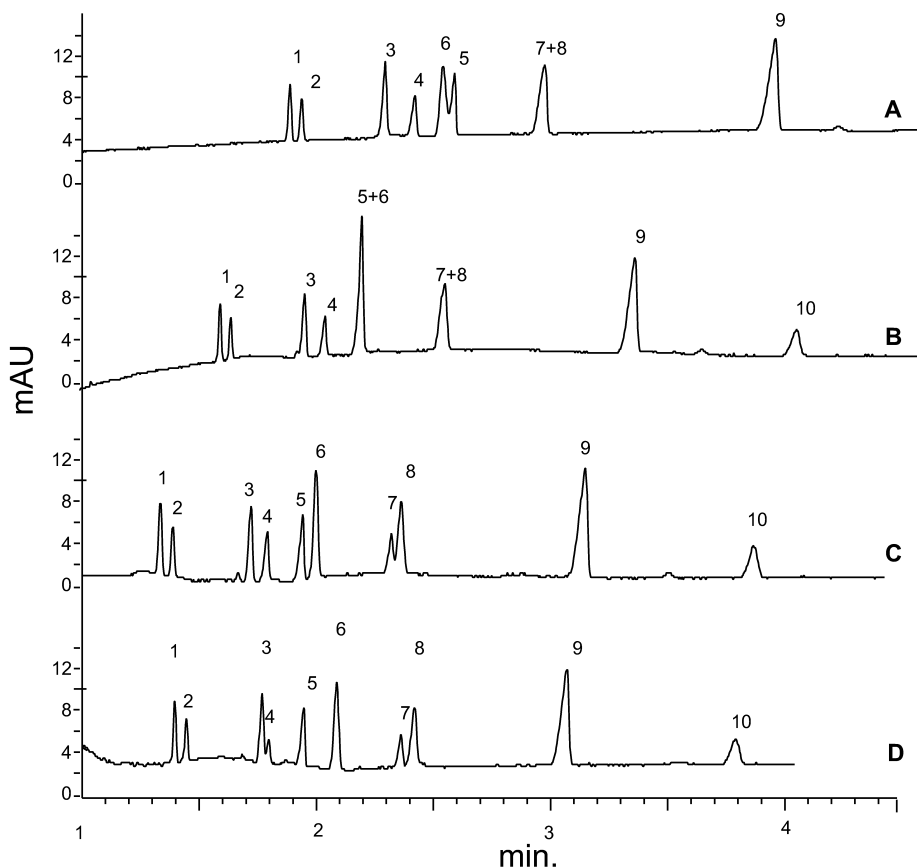


Fig. 2. Effect of applied current on separation of inorganic anions. (A) $-100 \mu\text{A}$; (B) $-125 \mu\text{A}$; (C) $-150 \mu\text{A}$; (D) $-175 \mu\text{A}$. Peak numbers, and experimental conditions as in Fig. 1. Except the applied current (variable) and the buffer concentration that was 50 mM for this experience.

an adequate separation of all the compounds under study.

3.3.1. Methanol

Fig. 3A shows the effect of the addition of 5 and 10% (v/v) methanol to the carrier buffer (50 mM tetraborate), keeping all other experimental conditions constant. As observed, the presence of 5% methanol has a significant effect on mobility of the anions studied. The separation of thiosulfate and nitrite ion is increased, and migration order of chromate and nitrate is reversed. The ferrocyanide mobility is significantly changed. Reductions in electroosmotic flow as well as electrophoretic velocities are caused by viscosity variations on the buffer solutions [24]. Furthermore, changes in the solvation

of the anions can also play a fundamental role in decrease in electrophoretic mobility. The higher the charge of the anion is, the apparent electrophoretic mobilities of anion decrease [25]. The addition of greater amounts of this solvent compromises the resolution of bromide and iodide and thiosulfate overlaps completely with nitrate. Therefore, the addition of 5% methanol to the buffer is the amount selected for the baseline separation of all the anions studied due to the better resolution achieved (Fig. 4).

3.3.2. Acetonitrile

Acetonitrile is another organic solvent frequently employed to improve selectivity of various electrophoretic systems [26]. With this solvent, it is necessary to add 15% (v/v) to resolve all the anions

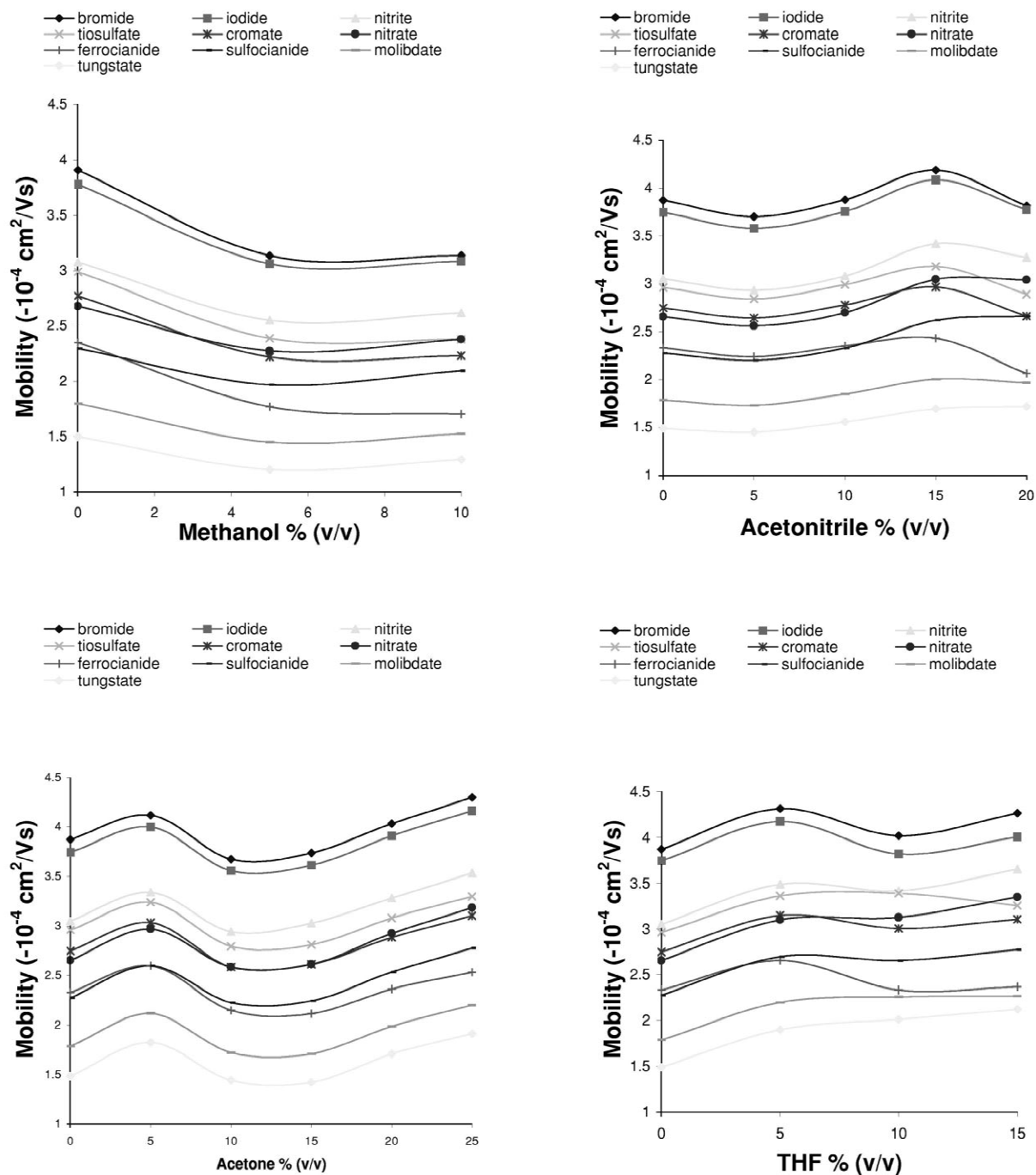


Fig. 3. Plots of electrophoretic mobility against the concentration of diverse organic modifiers. (A) methanol; (B) acetonitrile; (C) acetone; (D) tetrahydrofuran. Buffer concentration was 50 mM tetraborate. Applied current $-150 \mu\text{A}$. Other experimental conditions as in Fig. 1. Peak numbers as in Fig. 1. The mobility data are the media of the values obtained for three runs for each analyte.

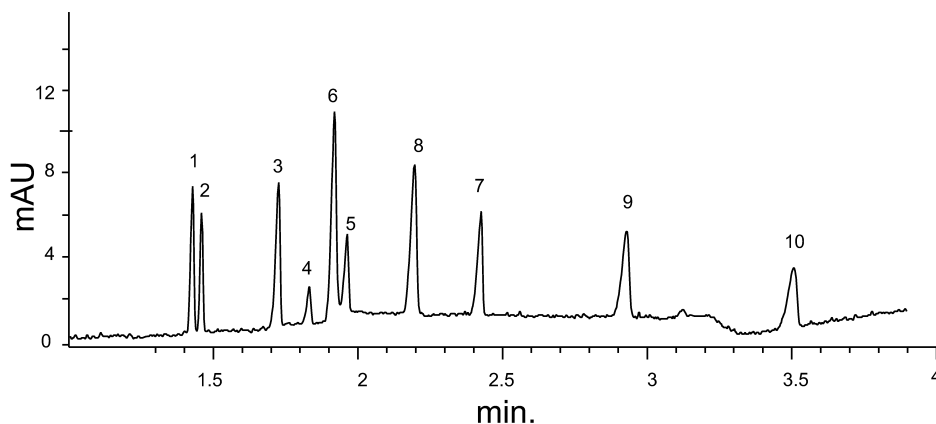


Fig. 4. Optimized separation of test anion mixture (1 $\mu\text{g}/\text{ml}$ of each analyte, except for WO_4^{2-} which concentration is 2 $\mu\text{g}/\text{ml}$). Electrolyte solution 50 mM tetraborate pH 9.3 with 5% (v/v) methanol. Other experimental condition and peak numbers as in Fig. 1.

studied (Fig. 3B). With 5%, changes in mobility are practically unappreciable in any of the anions. Ten percent is not sufficient for achieving separation between ferrocyanide and sulfocyanide, the addition of up to 15% acetonitrile being necessary to reach a similar situation as that achieved with 5% methanol. The addition of more acetonitrile decreased the resolution between bromide and iodide, chromate and sulfocyanide and thiosulfate and nitrate reverse their migration order.

3.3.3. Acetone

When acetone is used as modifier (Fig. 3C), the main problem is the separation between chromate and nitrate, which is not achieved until 25% modifier is added to the buffer. With this amount, a separation situation similar to that obtained with the two modifiers studied previously is achieved.

3.3.4. Tetrahydrofuran

This solvent is the only one that improves the separation between bromide and iodide (Fig. 3D). With the addition of 15% (v/v) THF a baseline resolution is achieved for all the ions under study, in this case nitrate eluting before thiosulfate. Furthermore, a greater reduction of migration times is accomplished for all the anions, completing the separation in less than 2.5 min. However, this solvent considerably decreases the detector response, reducing the absorbance values practically in half.

3.3.5. 2-Propanol and n-butanol

Although the addition of these alcohols was also studied, the problem arose that at concentrations above 5% it was practically impossible to degas the buffer, and the formation of microbubbles inside the capillary resulted in extremely complicated electropherograms. Therefore, it was concluded that these solvents are not ideal as organic modifiers for this particular electrophoretic system.

To summarize the results obtained, we can conclude that the utilization of organic solvents has a marked influence on the selectivity of the electrophoretic system. Depending on the nature of the solvent, different percentages of solvent need to be added for significant variations in the resolution to be produced (MeOH 5%; MeCN 15%; acetone 25% and THF 15%). The final result reached is quite similar for all solvents. In the separation performed with methanol, acetonitrile and acetone, no appreciable changes are produced in the detection of the ions under study and the response obtained in each case is of the same order. However, tetrahydrofuran reduces absorbance values, despite it being the solvent that produces the greatest reduction in migration times and improves the selectivity of monovalent ions. The difference in selectivity pattern observed in the different organic solvent can be attributed to their different solvation properties. Methanol is capable of solvating anionic species to a greater extent compared to acetonitrile, acetone or THF. So, methanol has been chosen as organic modifier for future analyses. In addition, the desired resolution is

achieve with the addition of the least amount of modifier.

3.4. Validation of the method

In order to obtain good detection limits, necessary for environmental analyses, the injection technique of sample stacking was used. The effect of injection volume on the separation of the anions was studied, gradually increasing the injection time and maintaining the injection pressure at 50 mbar. The separation efficiency is acceptable up to injection of 20 s. Fig. 4 shows the electropherogram obtained using the operating conditions established (50 mM tetraborate, pH 9.3, with 5% of methanol, 20°C; current, $-150 \mu\text{A}$ and injection of 50 mbar during 20 s).

Under these conditions, the validation of the method was carried out testing the parameters listed in Table 1. Calibration curves for all ions were linear in the range 0.1–10 $\mu\text{g/ml}$ with good correlation coefficients for all of them. The quantitation limits, established for the ratio $S/N=10$, are at 0.04 $\mu\text{g/ml}$ for the nitrate ion and 0.22 $\mu\text{g/ml}$ for tungstate. The repeatability of the migration times and peak areas was studied by injecting 10 times a standard solution of anions at a concentration of 1 $\mu\text{g/ml}$ of each analyte. The coefficient variation for peak areas is between 1.5 and 3% and for migration times is below 1%.

Comparing with literature, the limit of quantitation (LOQ) achieved for all the anions are better than

those obtained by indirect UV detection method [27]. The LOQ values are two order lower than those obtained by hydrodynamic injection and similar than the LOQ obtained with electrokinetic injection. Comparing with other UV absorbing methods, the LOQ ($S/N=10$) obtained with the proposed method are better than the limit of detection (LOD) ($S/N=3$) reported by other authors for all the anions [10,23]. The CE proposed method is 2–3 times faster than IC and than other CE methods reported by other authors [28]. CE provides also higher separation efficiencies and requires lower sample and electrolyte volumes than IC. In this case, the sensibility is better and the precision is in agreement with the IC method [28].

3.5. Analytical applications

The aim of the proposed method is to determine directly absorbing anions without any treatment, except filtration or dilution, when necessary.

3.5.1. Natural waters

Several samples of environmental interest have been analyzed such as rain water, river water, and drinking water. In all samples, the only anion detected was nitrate, finding values of 0.82 $\mu\text{g/ml}$ in rainwater, 3.53 $\mu\text{g/ml}$ in river water and 2.52 $\mu\text{g/ml}$ in tap water. All these samples have been analyzed directly, without any treatment except filtration, not offering any difficulties in their determination. Fig. 5B shows the resulting electropherogram of the analysis of rainwater, as an example.

3.5.2. Effluents of a power plant

Different samples collected at different stages of thermal power plant processes such as: river water, tap water, process water, coal pile run off water, water from slag plate, liquid waste (waste water from coal mine run off and operational process), final effluent of the treatment plant and sludge, were also analyzed with this method. The addition of standards showed neither matrix effect nor interference, thus analysis of these anions was possible without any problems. Except for the process water samples, in which none of the anions studied has been detected, nitrate ion was quantified in all the samples. As an example, Fig. 5C shows the resulting electropherogram of the analysis of the sample taken from the

Table 1

Validation date: repeatability of peak area and migration time, linearity and quantitation limits obtained with the proposed CE method (experimental conditions as in Fig. 4)

Anion	RSD ($n=10$) %		Linearity correlation (r^2)	LOQ ($S/N=10$) $\mu\text{g/ml}$
	Migration time	Peak area		
Br^-	0.73	1.76	0.9991	0.10
I^-	0.70	2.88	0.9999	0.15
NO_2^-	0.74	1.49	0.9998	0.09
$\text{S}_2\text{O}_3^{2-}$	0.64	2.85	0.9998	0.17
CrO_4^{2-}	0.66	2.25	0.9939	0.13
NO_3^-	0.82	1.32	0.9973	0.04
$\text{Fe}(\text{CN})_6^{4-}$	0.66	2.96	0.9983	0.13
SCN^-	0.89	2.55	0.9999	0.06
MoO_4^{2-}	0.92	2.70	0.9999	0.04
WO_4^{2-}	1.18	2.42	0.9999	0.22

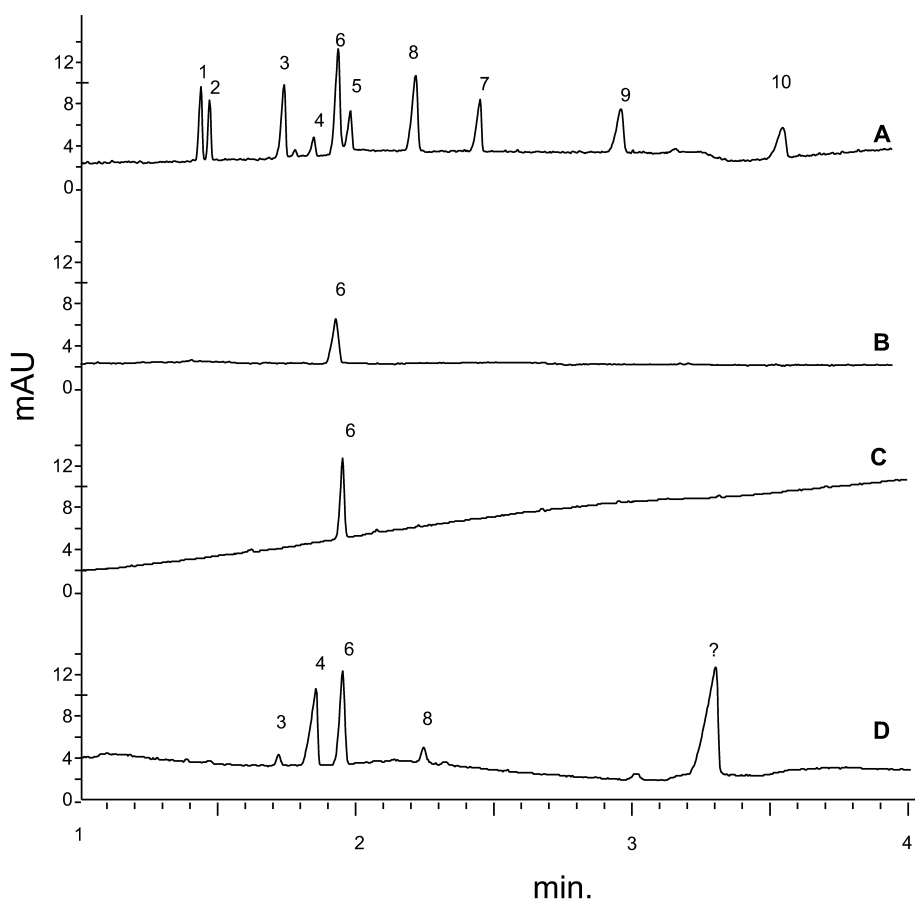


Fig. 5. Electropherograms of different real samples: (A) test mixture (1 $\mu\text{g}/\text{ml}$ except for WO_4^{2-} which concentration is 2 $\mu\text{g}/\text{ml}$); (B) rain water; (C) slag plate (dilute 1:5); (D) aqueous extract of sludge. Experimental conditions as in Fig. 4. Peak numbers as in Fig. 1 (?=unknown peak).

Slag-plate. Fig. 5D corresponds to the analysis of the aqueous extract of sludge sample, where besides nitrate, other anions are also found such as nitrite, thiosulfate or sulfocyanide.

3.5.3. Industrial residual water

A residual water sample from a chromium plating industry was analyzed with this method without further manipulation other than the 1:10 dilution, necessary to be within the linear range. Fig. 6A shows the electropherogram obtained for this sample and the same sample spiked with the studied anions at 1 $\mu\text{g}/\text{ml}$ (Fig. 6B). As observed, nitrate and chromate ions have been quantified. In the original

sample, the concentration of these two anions was 48.33 $\mu\text{g}/\text{ml}$ nitrate and 17.62 $\mu\text{g}/\text{ml}$ chromate ion.

4. Conclusion

The utilization of 50 mM sodium tetraborate as carrier electrolyte allows a rapid separation of absorbing anions without the need for the addition of external flow modifier. The presence of organic solvent in the buffer affects the selectivity of the electrophoretic system, even the migration order can be reversed, especially with ions of high charge. With the optimized method good detection limits are achieved, which allows the direct determination of

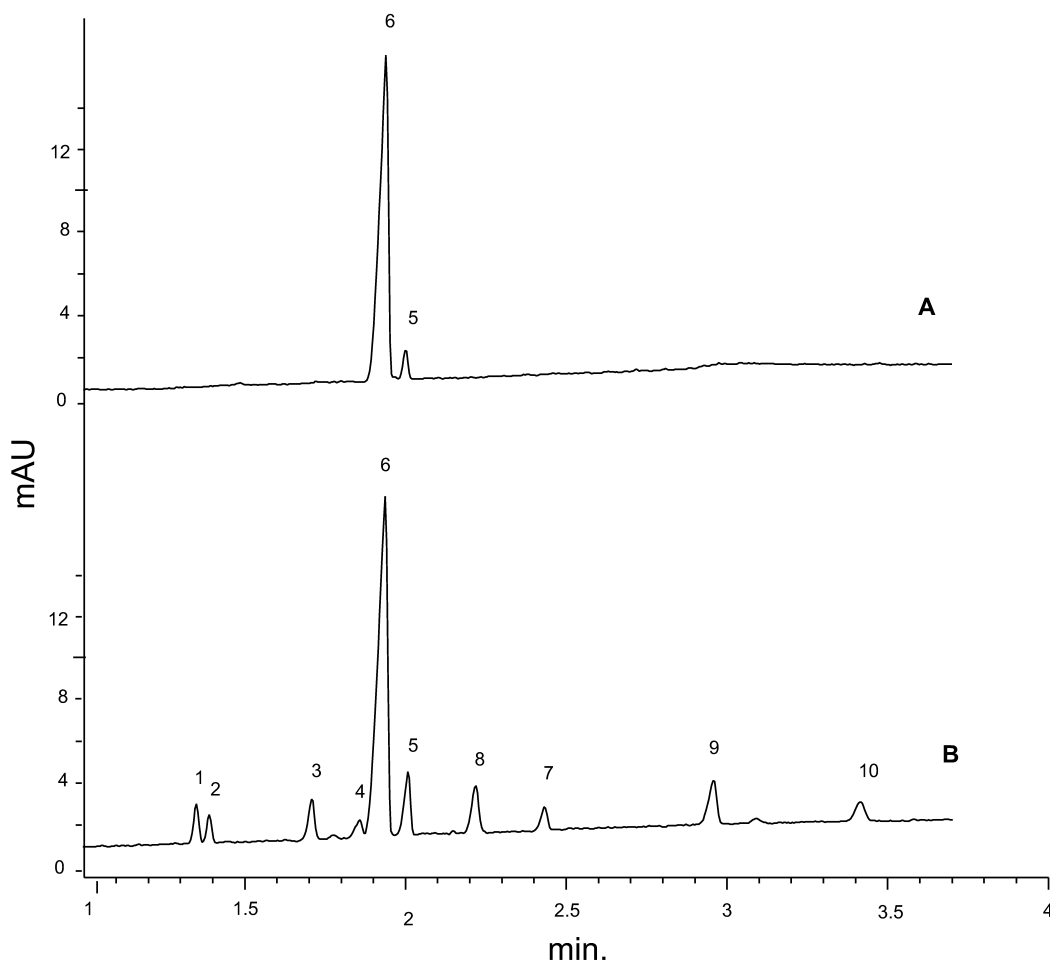


Fig. 6. Electropherograms corresponding to (A) sample of wastewater of a chromium plating industrial process; (B) the same sample spiked with the studied anions at 1 $\mu\text{g}/\text{ml}$. Experimental conditions as in Fig. 4. Peak numbers as in Fig. 1.

these compounds in diverse environmental matrices. In the samples analyzed in this work, no matrix effect has been observed for the determination of the ions under study.

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