

Sensitive Determination of Metal Ions in Drinking Water by Capillary Electrophoresis Coupled with Contactless Conductivity Detection Using 18-Crown-6 Ether and Hexadecyltrimethylammonium Bromide as Complexing Reagents

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Abstract

A simple, economical, and sensitive capillary electrophoresis (CE) method integrated with capacitively coupled contactless conductivity detection was developed for the determination of metal ions such as K^+ , Na^+ , Mg^{2+} , Sr^{2+} , Ca^{2+} in drinking water. 18-Crown-6 ether and Hexadecyltrimethylammonium Bromide (CTAB) were employed as complexing reagents. The effects of electrolyte additives, citric acid buffer solution, and other separation conditions of CE were comprehensively investigated and carefully optimized. The best results were obtained in a running buffer solution composed of citric acid (12 mM), 18-crown-6 ether (0.2 mM), and CTAB (0.015 mM) at pH 3.5. Under these conditions, a complete separation of five metal ions was successfully achieved in less than 12 min. The limits of detection for the optimal procedure were determined to be in the range of 0.02 - 0.2 $mg \cdot L^{-1}$. The repeatability with respect to migration times and peak areas, expressed as relative standard deviations, was better than 2.3% and 5.1%, respectively. Evaluation of the efficiency of the methodology indicated that it was reliable for the determination of metal ions in six different brands of drinking water samples.

Keywords

Capillary Electrophoresis, Contactless Conductivity Detection, Metal Ions, Food, Drinking Water

1. Introduction

Most of the alkali and alkaline earth metal ions play an important role in numerous processes in the human body, such as volume and osmotic regulation, myocardial rhythm, blood coagulation, and neuromuscular excitability. The detection of any deviation from the normal concentration ranges of these species is very useful in the diagnosis of metabolic disorders and abnormalities [1] [2]. Common metal ions including potassium (K^+), sodium (Na^+), magnesium (Mg^{2+}), strontium (Sr^{2+}), and calcium (Ca^{2+}) are monitored routinely in a wide variety of drinking water samples. Access to safe drinking water with appropriate limits of metal ions as nutrients has always been a great challenge. Therefore, nowadays, many drinking water industries are constantly emerging. Diverse factors such as the belief that mineral waters are of medicinal value, fueled by the impression that mineral waters are always associated with metal ions as portrayed in the advertisements, have contributed to this popularity [3]. Determination of metal ions in drinking water is an active topic in analytical science. Therefore, a simple, rapid, and reliable method for the simultaneous determination of these metal ions in real samples is highly desirable.

The traditional methods for the analysis of metal ions in environmental samples include spectrophotometry and atomic absorption spectroscopy, capable of detecting only a single element at a time. Therefore, these methods are time-consuming and laborious [4]. Inductively coupled plasma-mass spectrometry is a powerful tool for elemental analyses except for its high operating costs. Capillary electrophoresis (CE) has emerged as a valuable alternative that offers considerable advantages including rapid analysis, cost-effectiveness, high separation efficiency, and reagent consumption [5]. CE has benefit of simultaneous multielement determination. The traditional detection method used in CE system is optical detection; however, most metal ions lack enough absorbance in the ultraviolet (UV) region, which significantly limits its practical application range [5]. In order to overcome the abovementioned issue, a method involving indirect UV detection was developed [6] [7]. In this method some reagents such as imidazole, possessing an intense chromophore group in the UV region, were added to the running buffer. However, UV-absorbing buffer additives and more complex buffer systems were needed to facilitate indirect absorbance detection [8], which led to weaker quantitative performance [9] [10]. Unlike optical detection techniques, electrochemical detection can be easily miniaturized with no diminution in analytical performance. It includes three detection techniques: amperometric, potentiometric, and conductivity detection. Among these three methods, potentiometric detection is much less widely used than the other two detections and amperometric detection is affected by the CE voltage. Capacitively coupled contactless conductivity detection (C^4D) is considered as a universal detection technique for CE and it exhibits a good sensitivity for all the ionic species. The basic principle of conductivity detection involves the measurement of a difference in conductivity between migration analyte zones and the background electrolyte (BGE), which favors the application of C^4D for the determination of non-absorbing species [11].

Till date, there are few studies describing the CE determination of inorganic ions in formulations of drinking water. Some of these studies have described the determination of K^+ , Na^+ , Mg^{2+} , and Ca^{2+} ions in real samples [12] [13] [14] [15]. However, the analysis of elements that have similar electrophoretic mobilities, such as Mg^{2+} and Sr^{2+} , by CE-C⁴D has not been frequently described in literature. Nonetheless, the occurrence of Sr^{2+} in drinking water is very likely. Considering the need to determine different metal ions in drinking water, as well as the analytical difficulties related to the simultaneous separation of some cations, this study aims to describe the separation of five metal ions, which are common and important as nutrients in drinking water. Introduction of a supplementary separation mechanism, based upon the selective complexation of the cation was highly desirable to improve the analyses of ionic samples. Typical protocols for CE-C⁴D separation of metal ions, in general, involve the use of a buffer system containing 2-(N-morpholino)ethanesulfonic acid/histidine (MES/His) as working electrolytes, and complexing agents, such as 18-crown-6 ether, acetic acid, citric acid, lactic acid, and 2-hydroxyisobutyric acid as additives [16] [17] [18] [19]. In this study, we developed a simple and compact CE-C⁴D method based on citric acid buffer solution (Cit) by using 18-crown-6 ether and hexadecyltrimethylammonium bromide (CTAB) as complexing reagents to determine K^+ , Na^+ , Sr^{2+} , Mg^{2+} , and Ca^{2+} ions. The proposed method was successfully applied to the quantitation of above metal ions in six different brands of drinking water.

2. Experimental

2.1. Reagents, Solutions, and Samples

All chemicals used were of analytical-grade. Acetic acid, L-histidine (His), MES, 18-crown-6 ether, potassium chloride (KCl), sodium chloride (NaCl), magnesium chloride ($MgCl_2$), strontium chloride ($SrCl_2$), and calcium chloride ($CaCl_2$) were purchased from Sinopharm Chemical Reagent Co., Ltd. Organic additives, 18-crown-6 ether and CTAB were obtained from Aldrich (Milwaukee, WI, USA).

KCl, NaCl, $MgCl_2$, $SrCl_2$, and $CaCl_2$ were dissolved in deionized water (Milli-Q water purification system, Millipore, Milford, MA) to prepare the stock solution with concentration of $100\text{ mg}\cdot\text{L}^{-1}$. For the analyses of inorganic ions, a 10 mM stock solution of 18-crown-6 ether and 1 mM stock solution of CTAB were prepared and added to the running buffer in order to allow the complete separation of five metal ions. Fresh running buffer solutions were prepared daily. All the solutions were stored in a refrigerator at 4°C until their use. Prior to the experiments, all the solutions were filtered through $0.22\ \mu\text{m}$ polypropylene Acrodisc syringe filter (Xinya Purification Instrument Factory, Shanghai, China) and sonicated for 5 min to remove bubbles.

Drinking water samples were obtained from local supermarket in Shanghai, China. Prior to the analysis, the water samples were filtered through polypropylene Acrodisc syringe filter (nominal pore size $0.22\ \mu\text{m}$). All the samples were kept in a refrigerator until the experiments were performed and were diluted to specified concentration in our testing range with the buffer solution prior to analyses.

2.2. Instrumentation and Analytical Procedure

The CE-C⁴D system used was similar to a previously reported system [20]. A high-voltage power supply (0 ± 30 kV, Shanghai Institute of Nuclear Research, Shanghai, China) was utilized. Electrochemistry experiments were performed on a capacitively coupled contactless conductivity detector (eDAQ, Denistone East, NSW, Australia). The C⁴D operates with a sine-wave signal at a frequency of 600 KHz and an effective voltage of 65 V. Analyses were performed in uncoated fused silica capillaries (Yongnian Optical Fiber Factory, Hebei, China) with an internal diameter of 75 μm , an external diameter of 375 μm , and a total length of 65 cm (effective length of 50 cm). Each new capillary was activated by sequentially flowing (pressure driven by a medicine syringe) 0.5 M hydrochloric acid, 0.5 M sodium hydroxide (NaOH) solution, deionized water, and then the running buffer for 30 min, respectively. The capillary was then equilibrated in the buffer under an electric field of 17 kV for about 30 min prior to the sample injection. Otherwise, pretreatment was carried out daily by washing with 0.1 M NaOH solution, deionized water and running buffer for 5 min, respectively. The capillary was rinsed with the running buffer between analyses to improve reproducibility. A voltage of 30 kV was then applied for 60 min with the BGE. Data acquisition and analysis were performed using e-corder data acquisition system powerchrom 280 (eDAQ, Denistone East, NSW, Australia). The pH was monitored using a PHS-3C Acidometer (Shanghai Jicheng Instrument Factory, China). Experiments were performed at constant laboratory temperature of $24^\circ\text{C} \pm 1^\circ\text{C}$ measured inside the detection compartment of the CE-C⁴D system.

All the quantitative determinations of metal ions in drinking water samples were made by the standard addition method ($n = 3$), where the peak area obtained for each analyte was used for quantitative interpretation and calculations. All data treatment was performed by using Microcal Origin 7.0 (Microcal Software, Northampton, MA, USA).

3. Results and Discussion

3.1. Optimization of Separation Conditions

3.1.1. Selection of Running Buffer Solution

In C⁴D, the response arises from the difference in conductivity between analytes and BGE co-ions. In order to achieve a high signal-to-noise ratio (S/N), a large difference between the conductance of the analytes and electrolyte is needed [21] [22]. The BGE employing MES/His has commonly been used as a running buffer for the separation of small ions in CE-C⁴D attributed to its low specific conductivity and significantly high ionic strength. Unfortunately, for the separation of K^+ , Na^+ , Mg^{2+} , Sr^{2+} , and Ca^{2+} ions, this BGE is not ideal because the peaks corresponding to Na^+ and Mg^{2+} strongly overlap with the peaks corresponding to Ca^{2+} and Sr^{2+} , respectively (Figure 1). Therefore, similarities in the effective ionic radii and equivalent conductance imposed the need for the addition of an auxiliary complexing agent of considerably low pH (3 - 5) to the BGE, in order to achieve the complete separation of the cationic species [23]. Therefore, for varying the complexing constant of the complexing agent, an ideal approach involved the

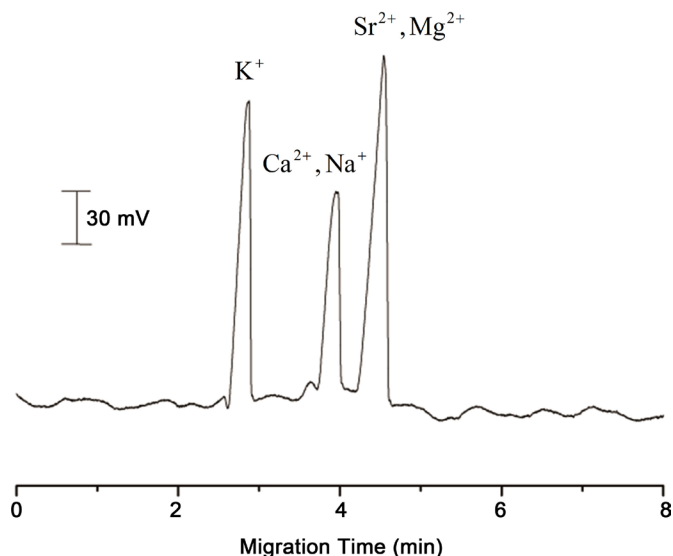


Figure 1. Electropherograms for mixture containing K^+ , Ca^{2+} , Na^+ , Sr^{2+} , and Mg^{2+} ions. The concentration of five metal ions was $5 \text{ mg}\cdot\text{L}^{-1}$. Electrolyte: 10 mM MES + 10 mM His; Capillary column: $50 \text{ cm} \times 75 \text{ }\mu\text{m}$; separation voltage: +16 kV; injection: electrokinetic injection using a voltage of 16 kV for 4 s. Sample diluted with running buffer.

replacement of MES/His by Cit, capable of complexing with Mg^{2+} and Sr^{2+} , and Ca^{2+} at low pH. In systems involving contactless conductivity detection, parameters such as concentration of the electrolyte solution, pH of the BGE, and concentration of the complexing agents must be taken into account; therefore, in this study, these parameters were investigated in detail.

3.1.2. Effect of Concentration of Cit

Next parameter investigated was the concentration of Cit in the BGE. The effect of the concentration of Cit on the migration time was studied in the range 8 - 15 mM. **Figure 2** exhibits the effect of the concentration of Cit on the migration time, revealing that an increase in the concentration of Cit improves the separation degree of K^+ , Na^+ , Mg^{2+} , Sr^{2+} , and Ca^{2+} ions; however, higher concentration of Cit beyond 15 mM leads to comigration of Na^+ , Ca^{2+} and Mg^{2+} , Sr^{2+} . Moreover, 15 mM Cit is disadvantageous to the resolution and separation efficiency, making the baseline unstable and fluctuant at a separation voltage of 16 kV due to the excess *Joule heat*. Consequently, for a simultaneous separation of metal ions, it was better to select an optimized electrolyte concentration of 12 mM Cit.

3.1.3. Effect of CTAB Concentration

In order to improve the resolution of analytes, a small amount of the cationic surfactant CTAB was added to the BGE. Adsorption of CTAB micelles onto the capillary surface resulted in the formation of a layer with positive charge causing a reduction or even a complete reversal of the electroosmotic flow. **Figure 3** shows the effect of the CTAB concentration in the electrolyte on the migration times for the selected ions. Notably, with an increase in the concentration of CTAB, the separation of ions migration be-

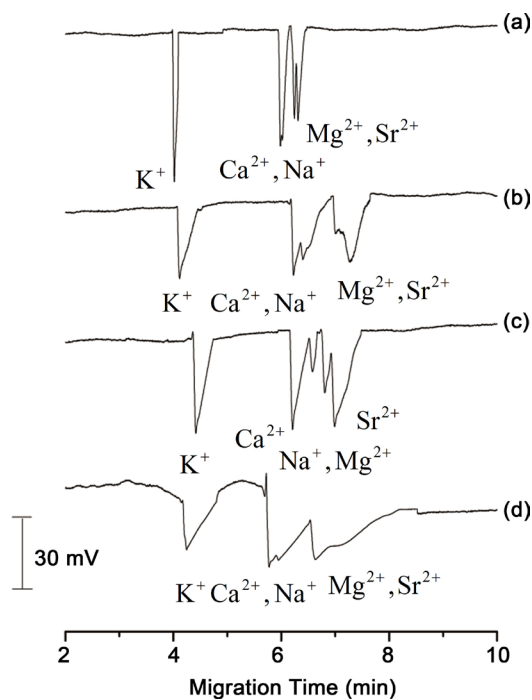


Figure 2. Influence of the Cit concentration on the separation of five metal ions. (a) 8 mM Cit; (b) 10 mM Cit; (c) 12 mM Cit; and (d) 15 mM Cit; concentration of K^+ , Ca^{2+} , Na^+ , Mg^{2+} , and Sr^{2+} were 3, 5, 5, 5, and 12 $mg\cdot L^{-1}$, respectively. Other conditions were similar to those mentioned in **Figure 1**.

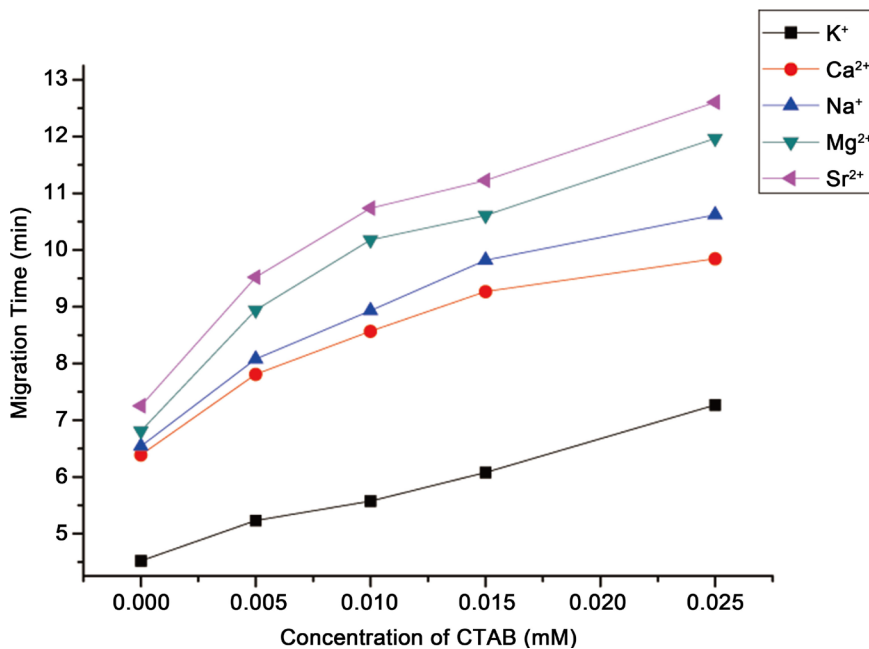


Figure 3. Influence of the CTAB concentration on the separation of five metal ions. Other conditions were similar to those mentioned in **Figure 2**.

tween Ca^{2+} and Na^+ improves. Nonetheless, the baseline becomes unstable and resolution of the five metal ions becomes worse due to higher concentration of CTAB (0.02

mM). Thus, CTAB concentration of 0.015 mM seemed to be a suitable compromise and was selected as the optimized concentration.

3.1.4. Effect of 18-Crown-6 Ether on Electromigration

Under the tested experimental conditions discussed above, a phenomenon was observed that Sr^{2+} and Mg^{2+} could not be separated completely by using only Cit buffer and CTAB. The separation of some comigrating cations can be fine-tuned by the addition of 18-crown-6 ether to the BGE. 18-Crown-6 ether is known to form inclusion complexes with several inorganic cations, such as K^+ and Sr^{2+} . The complex formation depends on the sizes of both the cation and the crown ether cavity. **Figure 4** illustrates the influence of the addition of 18-crown-6 ether to the electrolyte with a constant concentration of Cit at pH 3.5. The crown ether concentration was varied in the range 0 - 0.3 mM. The migration times of some ions did not change significantly. However, the migration time of K^+ and Sr^{2+} ions was significantly influenced. Consequently, **Figure 4** exhibits that the electrolyte containing 0.2 mM 18-crown-6 ether concentration provides a complete baseline separation of Sr^{2+} and Mg^{2+} ions.

3.1.5. Effect of Injection Time

Furthermore, effect of the injection time ranging from 2 to 10 s was investigated. Variation of the injection time from 2 - 10 s at 17 kV led to an increase in the peak heights of these five cations. However, simultaneously the band broadening of signals and low separation efficiency were also observed. When the injection time exceeded 6 s, the problem related to peak broadening in conjunction with peak distortion became more obvious (results not shown). Thus, the abovementioned discussion indicated that 6 s in-

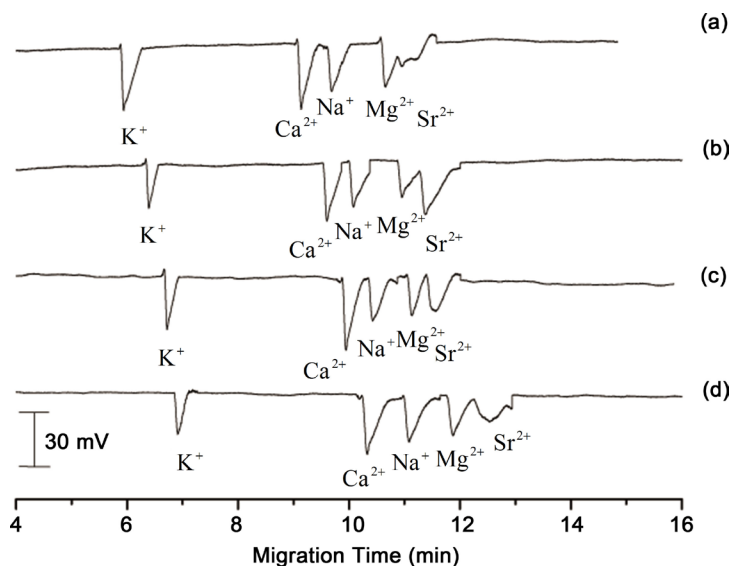


Figure 4. Influence of 18-crown-6 ether concentration on separation of metal ions. (a) 12 mM Cit + 0.015 mM CTAB; (b) 12 mM Cit + 0.015 mM CTAB + 0.1 mM 18-crown-6 ether; (c) 12 mM Cit + 0.015 mM CTAB + 0.2 mM 18-crown-6 ether; and (d) 12 mM Cit + 0.015 mM CTAB + 0.3 mM 18-crown-6 ether; pH 3.5; other conditions were similar to those mentioned in **Figure 3**.

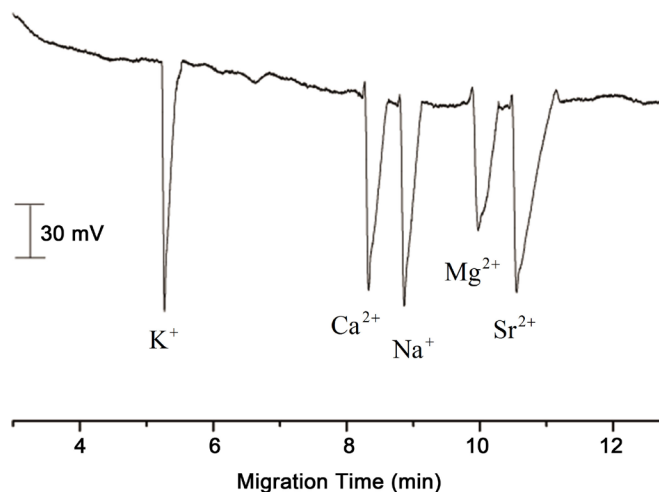


Figure 5. Electropherogram for separation of five metal ions. Conditions: capillary column: 65 cm \times 75 μ m; separation voltage: 17 kV; Electrolyte: 12 mM Cit + 0.015 mM CTAB + 0.2 mM 18-crown-6 ether; pH: 3.5; The concentration of K^+ , Ca^{2+} , Na^+ , Mg^{2+} , Sr^{2+} ions were 4, 8, 6, 4, and 18 $mg \cdot L^{-1}$, respectively. Samples were dissolved in running buffer; electrokinetic injection using a voltage of 17 kV for 6 s.

jection time could be selected as the optimal condition.

Above parameters related to BGEs showed that simultaneous determination of five metal ions in the real samples was possible. The electrolyte solution consisted of 12 mM Cit, 0.015 mM CTAB, and 0.2 mM 18-crown-6 ether at pH 3.5 (monitored by a PHS-3C Acidometer). The optimal injection time and separation voltage were 6 s and 17 kV, respectively. The separation was achieved in less than 12 min. The total capillary length used was 75 cm and the effective capillary length was 65 cm. The resulting electropherograms of the simultaneous determination of five metal ions are depicted in **Figure 5**.

3.2. Evaluation of the Developed Method

In order to evaluate the developed method for the quantitative purpose, linearity figures and limits of detection (LOD) were determined. The linearity of the method was determined by constructing a calibration curve with different concentrations of the five metal ions. The linear regression coefficients were always higher than 0.9950. Repeatability of the relative area and migration time, expressed as relative standard deviations (RSD), was better than 5.1% and 2.3% ($n = 8$), respectively, indicating a high degree of precision. All the validation data are listed in **Table 1**.

The detection limits (the concentrations providing peak heights which are 3 times as tall as the baseline noise) for K^+ , Na^+ , Mg^{2+} , Sr^{2+} , Ca^{2+} ions were determined to be below 0.2 $mg \cdot L^{-1}$ for all but one of the ions for this experimental system, which is well below the requirements for the application envisaged.

3.3. Sample Analyses

The methodology based on the CE- C^4D was applied to the analysis of five metal ions in six different drinking matrices. The quantitative results (obtained using a calibration

Table 1. The results of linearity, LOD, and precision.

Compound	Linear regression ^a	Linearity (mg·L ⁻¹)	Correlation (R ²)	LOD ^b (mg·L ⁻¹)	RSD% (time)	RSD% (area)
K ⁺	y = 10.387x + 1.146	0.05 - 20	0.9973	0.02	1.3	2.7
Ca ²⁺	y = 3.747x + 0.151	0.25 - 20	0.9968	0.1	2.1	3.6
Na ⁺	y = 4.837x + 0.766	0.1 - 15	0.9984	0.05	1.7	4.8
Sr ²⁺	y = 1.671x + 0.058	0.4 - 15	0.9955	0.2	2.3	5.1
Mg ²⁺	y = 5.076x + 0.612	0.1 - 20	0.9972	0.05	1.4	4.2

^aLinear regression based on peak area (mV s) vs. concentration (mg·L⁻¹).

^bEstimated on the basis of S/N = 3.

Table 2. Quantitative analysis of real samples^a. ND: not detected.

Sample	Cation concentration (mg·L ⁻¹) ^b				
	K ⁺	Ca ²⁺	Na ⁺	Sr ²⁺	Mg ²⁺
1	8.2	34.8	5.7	0.5	36.3
2	ND	69.2	3.8	0.8	10.9
3	3.1	2.7	4.5	0.6	ND
4	2.9	54.2	43.9	0.6	9.4
5	4.3	21.9	15.5	ND	8.2
6	0.61	51.7	2.1	ND	5.4

^aDetermination was performed in triplicate and experimental conditions were similar to those mentioned in **Figures 3-5**.

^bRSD (n = 3): 3% - 10%.

curve method) are listed in **Table 2**. The values listed in **Table 2** indicate the presence and quantification of the metal ions Ca²⁺ and Na⁺ ions in all the samples, and existence of K⁺, Sr²⁺, and Mg²⁺ ions in some samples.

4. Conclusion

The perfect separation of metal ions K⁺, Na⁺, Mg²⁺, Sr²⁺, and Ca²⁺ in drinking water samples could be achieved by CE-C⁴D using a Cit buffer solution containing CTAB and 18-crown-6 ether as complexing reagents. The proposed method did not require any sample retreatment, except dilution with running buffer, and thus did not significantly change the composition of the original sample. It was easy to optimize the composition of the running buffer by using a conductivity detector. Under the optimal conditions, the developed method exhibited a very good quantitative performance in terms of accuracy and precision with an analysis time of less than 12 min for all the ions.

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