Determination of metal cations by capillary electrophoresis
Effect of background carrier and complexing agents

Y.-H. Lee, T.-I. Lin*

Department of Chemistry, National Taiwan University, Taipei 106, Taiwan

Abstract

The determination of trace metals in aqueous samples can be readily accomplished by capillary electrophoresis (CE) via indirect absorbance detection. Methods for simultaneously determining alkali, alkaline earth and transition metal ions and Group IB, IIB and IVA metals ions were developed. Imidazole, benzylamine, ephedrine or pyridine was used as the carrier buffer and the background absorbance provider. Glycolic acid, α-hydroxyisobutyric acid or succinic acid was used as the complexing agent. The elements determined were Li, Na, K, Cs, Mg, Ca, Sr, Ba, Cr, Mn, Fe, Co, Ni, Cu, Zn, Cd, Ag, Al and Pb. All ions could be separated in less than 15 min. In most instances reported here, more than a dozen ions could be separated in 5–10 min. All peaks were well separated and baseline resolved (*i.e.*, no peaks overlapped), except Ag and Al, which required a separate and additional analysis. The detection limit was in the range 0.02 (Na)–208 ppb (Cr) with the electrokinetic injection mode (10 kV, 5 s). The reproducibility was 1% for the migration time and better than 5% for the peak height for most metal ions. The calibration graphs were linear for most ions in the concentration range 10^{-5}–10^{-3} M ($R^2 = 0.9995$–0.9999) using the hydrodynamic injection mode. Concentrations lower than 10^{-5} M can be determined using the electrokinetic injection mode, but the calibration graph is not linear. The methods developed here are well suited for determining metal ions in a variety of real samples.

1. Introduction

The determination of metal ions in various samples is important for obvious reasons. Reliable and rapid techniques are needed for the determination of metal ions in medicines, soil samples, drinking water, etc. Metal ions can be determined by a number of techniques, including various atomic spectroscopic methods, electrochemical methods and ion chromatography [1,2]. A separation tool which has been rapidly developing is capillary electrophoresis (CE). Since a UV absorption detector is readily available in

all commercial capillary electrophoresis systems, the development of indirect UV absorption detection has made metal ion determination by CE an attractive alternative.

Foret et al. [3] utilized indirect UV detection to demonstrate the separation of fourteen lanthanide cations by CE with the aid of a complexing agent, α-hydroxyisobutyric acid (HIBA), and creatinine as a UV-absorbing co-ion. Wildman et al. [4] and Weston and co-workers [5–7] investigated the factors affecting the separation of metal cations and optimized the detection sensitivity for metal cations using indirect photometric detection. They showed that a mixture of ninetten alkali, alkaline earth and lanthanide

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* Corresponding author.

metal cations can be baseline resolved in less than 2 min. Beck and Engelhardt [8] investigated several background carrier electrolytes (BCE) for indirect UV detection and found imidazole to be suitable for the separation of metal ions, amines and amino alcohols. They recently reported [9] a new buffer system consisting of *p*-aminopyridine and 2-hydroxybutyric acid for the separation of some transition metals. Timerbaev *et al.* [10] used 8-hydroxyquinoline-5-sulphonic acid for the CE of the transition and alkaline earth metals as precolumn-formed chelates with direct UV detection. Aguilar *et al.* [11] and Buchberger *et al.* [12] reported a detection scheme for the CE determination of Fe, Cu, Ni, Cr, Hg, Pd, Ag, Cd, Zn and Co cations using cyanide complexes by direct UV detection. Chen and Cassidy [13] evaluated several experimental parameters, *e.g.*, indirect detection reagents, pH, complexing agents and type of capillary surfaces, that would affect the separation of metal ions by CE and optimized the conditions for the separation of 26 metal ions. Shi and Fritz [14] described the use of several complexing agents, including phthalate, tartrate, HIBA and lactate, for the separation of 27 metal ions in only 6 min. Recently, we have investigated the role of complexing agents and pH on the CE separation of alkali and alkaline earth metal ions [15]. Using various mono-, di- and tricarboxylic and hydroxycarboxylic acids as complexing agents and imidazole as the background carrier for indirect UV absorbance detection, we showed that the metal ions could be completely separated in less than 2 min with a maximum resolution of 15 and a number of theoretical plates as high as 750 000 per metre.

We report here CE methods for analyzing a mixture of nineteen metal ions (Li, Na, K, Cs, Mg, Ca, Sr, Ba, Cr, Mn, Fe, Co, Ni, Cu, Zn, Cd, Ag, Al and Pb) (alkali metals and Ag ions are monovalent, Al and Cr ions are trivalent, all other ions are divalent; charges are omitted for brevity). Detection of metal ions was accomplished by indirect UV absorbance measurement. Imidazole, benzylamine, ephedrine and pyridine were investigated as carrier buffers and background absorbance providers. Glycolic and suc-

cinic acid and HIBA were employed as metal-complexing agents.

2. Experimental

2.1. Chemicals

All metal ion solutions were prepared from their nitrate salts, except Fe(II) (chloride salt). Stock standard solutions (1.0×10^{-2} M) were prepared, mixed and diluted to the specified concentrations used in different CE runs. All chemicals used, including various BCE and complexing agents, were of analytical-reagent grade from several vendors. Doubly deionized water prepared with a Milli-Q system (Millipore, Bedford, MA, USA) or doubly deionized distilled water was used exclusively for all solutions. The water blank was routinely checked for contamination by trace amounts of alkali and alkaline earth metal ions.

2.2. Buffers and pH adjustment

The running buffer contained 5 or 10 mM of BCE. The pH was varied as specified in the figures, being adjusted by adding a 1 M stock solution of complexing agent to the desired pH of 4.0 or 4.5 depending on the experiments. The concentration of the complexing acid varied from 5.7 to 16 mM (calculated by the volume added) as specified in the figures.

2.3. Apparatus

CE experiments were carried out in a fully automated Spectra Phoresis Model 1000 instrument (Spectra-Physics, San Jose, CA, USA). The system was equipped with a rapid scanning UV–Vis detector with 5-nm wavelength resolution. In most instances reported here, the detector wavelength was fixed at 254 nm (for pyridine) or 210 nm (all others) in order to obtain a more stable and less noisy baseline. The instrument was also equipped with an autosampler, a capillary cartridge and a solid-state Peltier temperature control unit. A personal computer (486 IBM

AT-compatible PC) was used to control the instrument settings, data acquisition and analysis with the vendor-provided software. The separation capillaries (bare fused silica) from Poly-micro Technologies (Phoenix, AZ USA) were 75 μm I.D. (365 μm O.D.) × 70 cm (63 cm to the detector) for the determination of the mobilities of background carriers and 75 μm I.D. (365 μm O.D.) × 90 cm (83 cm to the detector) for the separation of mixtures of metal ions. UV–Vis absorption spectra of BCE and complexing agents were measured with a Hitachi (Tokyo, Japan) U-2000 double-beam scanning spectrophotometer.

2.4. Electrophoretic procedures

Prior to first use, a new capillary was subjected to a standard wash cycle, and subsequent runs were carried out according to the established procedure [15]. Sample injection was carried out in either the electrokinetic (EK) or hydrodynamic (HD) mode as specified in the figures and tables. The separation run was at a constant voltage of +25 kV at a constant temperature of 25°C and with a current of 4–12 μA. The capillary was also washed with 0.1 M NaOH and deionized water as a daily routine. All buffer solutions were freshly prepared, filtered through 0.20-μm membranes and degassed under vacuum for 10 min.

2.5. Electrophoretic mobility determination

Benzyl alcohol was added to samples as a neutral marker for the electrophoretic mobility determination. The mobilities of the various BCE under the specified CE conditions were determined in 10 mM sodium acetate buffer (pH 4.0 or 4.5). Samples were injected in the HD mode for 1 s. Detection was made by rapidly scanning the UV absorbance over the range 200–300 nm, which allowed a positive identification of the background provider. Electroosmotic mobility, μ_{eo}, was calculated by the following equation:

$$\mu_{eo} = l_d l_t / t_m V \ (\text{cm}^2 \ \text{V}^{-1} \ \text{s}^{-1})$$

$$\mu_e = \mu_{obs} - \mu_{eo}$$

where l_d and l_t are the length of the capillary to the detector and the total length of the capillary, respectively, V is the running voltage and t_m is the migration time of the neutral marker (benzyl alcohol). The electrophoretic mobility of the background carrier electrolyte, μ_e, was obtained by subtracting μ_{eo} from the observed mobility μ_{obs}.

2.6. Identification of metal peaks in electropherograms

Peak identification for a specific metal ion was carried out after a complete baseline separation of all peaks had been established under the given conditions specified in the figures. Standards that contained only a single metal ion were run under the same conditions. The migration time of the known ion was compared with those in the mixture of metals. Since the reproducibility of migration time was about 0.2% in the HD injection mode and about 1% in the EK injection mode (see the Results and Discussion), this approach was fairly reliable. Also, trace amounts of K and Na were sometimes present in the diluent, the retention of which served as an internal marker for adjusting small discrepancies in the migration time. Alternatively, samples were spiked with standards containing only single metal species and the peak with increased height was identified.

3. Results and discussion

3.1. Separation of metal ions in different background electrolytes

Seventeen metal ions could be separated in CE using imidazole, pyridine or benzylamine as the BCE with the addition of glycolic acid as the complexing agent. Previous studies have shown that in order to achieve a good separation of analytes, the mobilities of the analyte ions must match that of the BCE [7,8]. The mobilities of the BCE used in this work were determined and

Table 1
Mobilities and pK_a values of various BCE

BCE	pK_a[a]	Mobility[b] (cm^2 kV^{-1} s^{-1})	
		pH 4.0	pH 4.5
Imidazole	6.95	0.511	0.507
Pyridine	5.25	0.472	0.416
Benzylamine	9.33	0.373	0.352
Ephedrine	10.14	0.283	0.280

[a] From ref. 16.
[b] See Experimental for details.

are listed in Table 1 along with their pK_a values. The positions where these background carriers appear in the electrophoregram are indicated by upward arrows in the figures. The mobility of imidazole matched best with the alkali and alkaline earth metal ions (they have higher mobilities), hence in this buffer these metal ions were better separated than the other ions (see Fig. 1). The migration order of the seventeen metal ions is as follows: (1) Cs, (2) K, (3) Ba, (4) Sr, (5) Na, (6) Ca, (7) Mg, (8) Mn, (9) Cr, (10) Fe, (11) Cd, (12) Li, (13) Co, (14) Ni, (15) Pb, (16) Zn, (17) Cu. In all of the following

figures, these peaks will be referred to by arabic numerals 1–17. In imidazole, peaks 10 and 11 (Fe, Cd) and peaks 12 and 13 (Li, Co) were not baseline resolved. Ni, Pb and Zn ions (peaks 14–16) that migrated more slowly showed peak trailing and had broader peak widths. Cu ion migrated the slowest and had the highest retention (about 13 min *vs.* 5.4–10.4 min for other peaks) with considerable peak trailing. Presumably the last four ions formed complexes with glycolic acid that have higher stability constants.

The migration order of these seventeen ions was the same in all three BCE. However, the resolution depended on the mobility of the BCE used. In imidazole the resolution for the last four peaks was poor because the mobility of BCE was too fast, and hence incompatible with these four metals. Pyridine, which had a lower mobility than imidazole, provided a better separation for the transition metal ions than the other BCE that were tried. Comparing the CE performed in imidazole, the most noticeable difference was the better separation between Fe and Cd peaks, and a complete baseline resolution of Li and Co
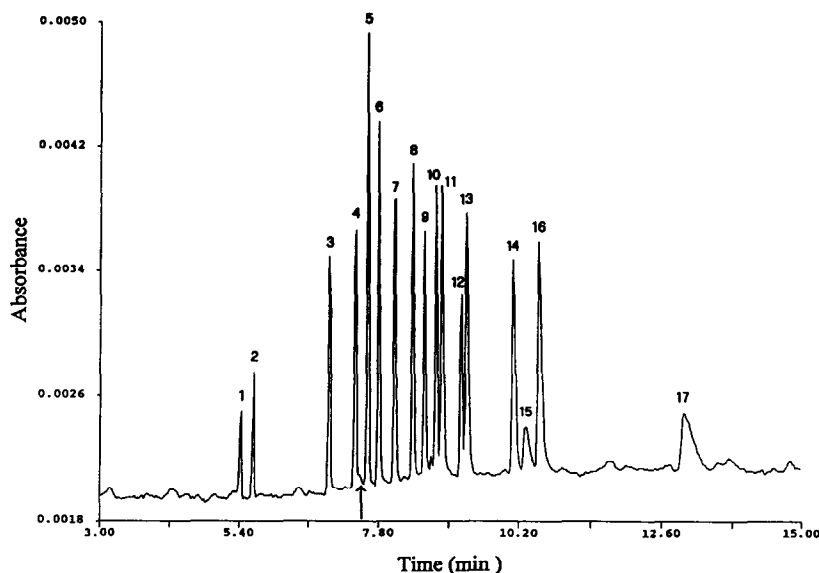

Fig. 1. Electrophoretic separation of seventeen metal ions using 10 mM imidazole–13 mM glycolic acid (pH 4.0). 1 = Cs; 2 = K; 3 = Ba; 4 = Sr; 5 = Na; 6 = Ca; 7 = Mg, 8 = Mn; 9 = Cr; 10 = Fe; 11 = Cd; 12 = Li; 13 = Co; 14 = Ni; 15 = Pb; 16 = Zn; 17 = Cu. Metal ions migrate in the same order in all figures. The upward arrow indicates the migration position of imidazole.
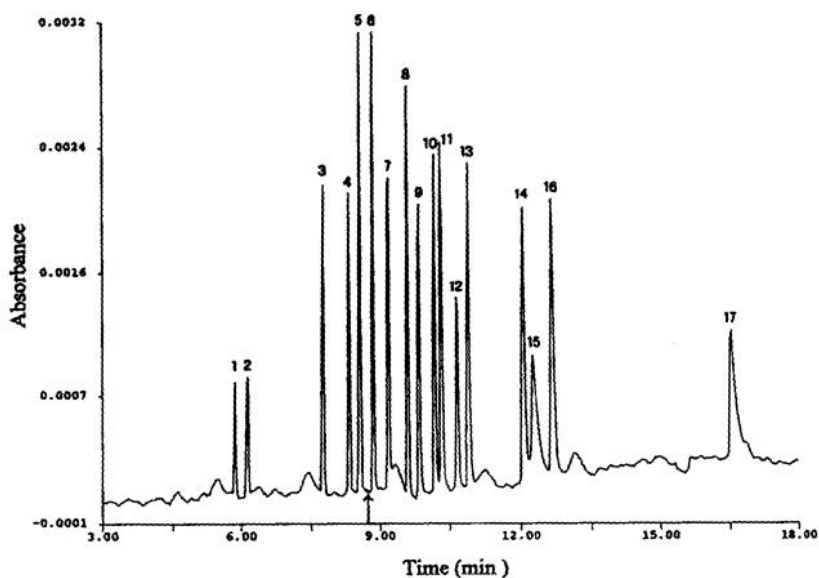
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Fig. 2. Electrophoretic separation of seventeen metal ions using 10 mM pyridine–12 mM glycolic acid (pH 4.0). The upward arrow indicates the migration position of pyridine. Peaks as in Fig. 1.

peaks (Fig. 2). The Pb and Cu peaks were also higher. The number of theoretical plates, N, in pyridine was higher for the last four ions than in the imidazole buffer. In benzylamine the five slower migrating ions all had sharper peaks (and hence higher N); however, Cd and Li were poorly separated (Fig. 3).

We thought that adding ephedrine (lowest mobility among the four) might improve the resolution of the last four peaks. A mixed BCE
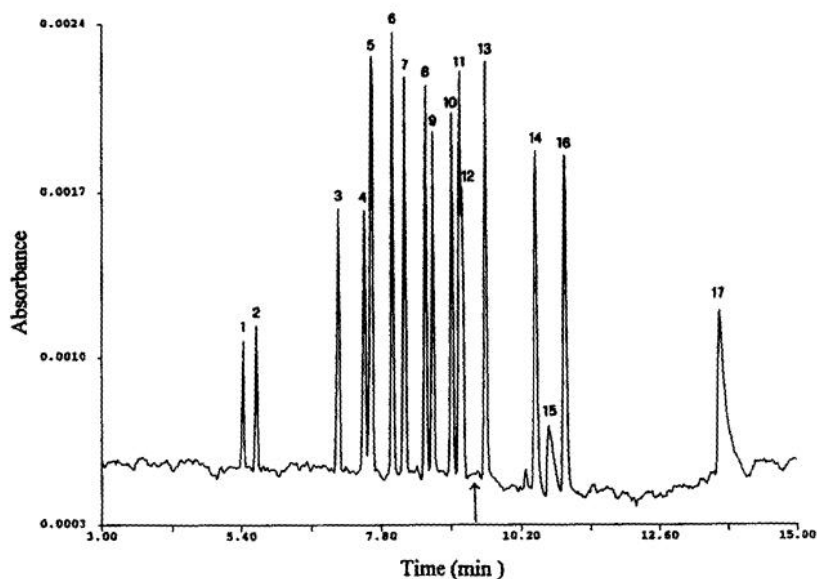

Fig. 3. Electrophoretic separation of seventeen metal ions using 10 mM benzylamine–16 mM glycolic acid (pH 4.0). The upward arrow indicates the migration position of benzylamine. Peaks as in Fig. 1.

containing 5 mM imidazole and 5 mM ephedrine with 12.2 mM glycolic acid (pH 4.0) was investigated, since in imidazole the migration time is shorter while the addition of ephedrine (smallest mobility among the four) may improve the performance of the last four peaks. Indeed, the Pb, Zn and Cu peaks (migration time unchanged as in 10 mM imidazole) were all considerably sharper in the mixed BCE than in the absence of ephedrine (Fig. 4). However, the separations between Fe and Cd and between Li and Co were poor. Thus, for samples in which analysis for the last four ions is more important, running CE in mixed BCE will improve their determination. The electroosmotic flow increased as the pH increased from 4.0 to 4.5, hence the CE separation time could be shortened at higher pH. However, we found that at pH 4.5 the resolution was inferior to that at pH 4.0 when monoprotic acids were used as the complexing agents. The resolution of the electropherogram was very sensitive to pH, as reported previously for alkali and alkaline earth metal ions [15]. Overall, running CE in 10 mM pyridine combined with 12 mM glycolic acid at pH 4.0 seems to give the best performance.

3.2. Effect of the complexing agent on CE separation

Several previous studies [3,6,15] have shown that HIBA is an effective complexing agent for separating alkali and alkaline earth metal cations by CE. We showed that succinic acid and several other dicarboxylic acids are also useful complexing agents for similar applications [15]. Previously, we tried CE for the seventeen cations using 10 mM imidazole and 14.6 mM HIBA at pH 4.0. Under such conditions, the Na–Ca, Cr–Fe, Cd–Li and Ni–Pb peaks could not be completely resolved [17]. The effects of imidazole and HIBA were investigated by lowering their concentrations to 5 and 6.75 mM (pH 4.0), respectively. The separation of certain metal cations was improved; in particular, the Ca, Na, Cr, Fe and Pb peaks were all well separated. However, the separation of Mn–Cd was poor and the Ni peak became merged with the Li peak (Fig. 5). The migration order of these seventeen ions also changes, indicating that the complexing agent has a strong effect particularly on the mobility of the divalent cations. The effect of succinic acid as a complexing agent was investigated. CE was
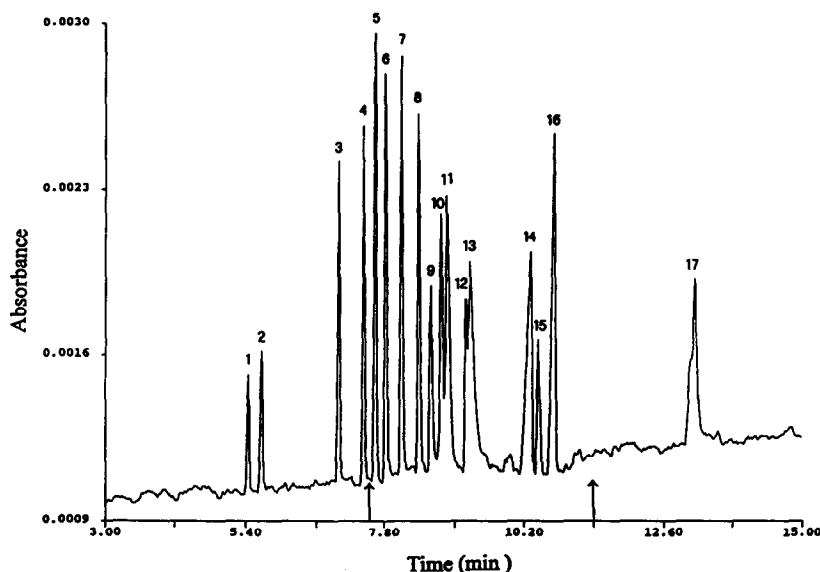

Fig. 4. Electrophoretic separation of seventeen metal ions using 5 mM imidazole–5 mM ephedrine–12.2 mM glycolic acid (pH 4.0). The left arrow indicates the migration position of imidazole and the right arrow that of ephedrine. Peaks as in Fig. 1.
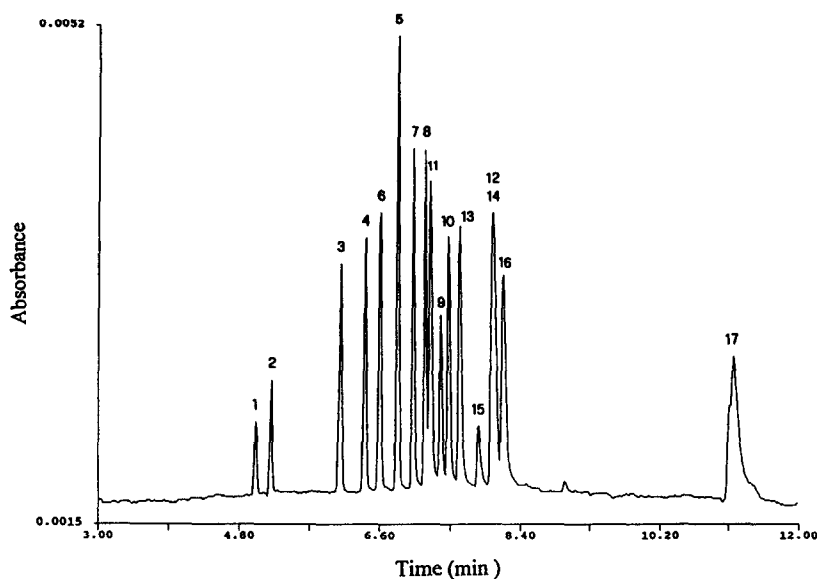
Fig. 5. Electrophoretic separation of seventeen metal ions using 5 m*M* imidazole-6.75 m*M* HIBA (pH 4.0). Peak 14 is Ni merged with the Li peak. Peaks as in Fig. 1.

performed using 10 m*M* imidazole and 9.7 m*M* succinic acid (pH 4.5), using 10 m*M* pyridine and 7.5 m*M* succinic acid (pH 4.5) and using 5 m*M* benzylamine and 5.75 m*M* succinic acid (pH 4.5). In all three buffers, which contained succinic acid as the complexing agent, the separations of metal cations were inferior to those obtained using glycolic acid. The results support the previous notion that the complexing agent plays a more important role than the background carrier in resolving ions whose mobilities were close and therefore could not be separated without the addition of a particular complexing agent.

3.3. CE determination of silver and aluminium cations

The determination of Ag and Al when mixed with the alkali and alkaline earth metal ions is a challenging problem. Numerous combinations of different compositions of BCE and complexing agents have been tried without complete success. Al and Ag cations could be separated from the seventeen ions in the previous analysis only in pyridine (5 m*M*), with the pH adjusted to 3.2 by

sulphuric acid, which also acted as a complexing agent [15]. However, the electropherogram of the mixture of nineteen metal ions showed that the transition metal and Pb ions (peaks 8-11 and 13-17 in Fig. 2) overlapped with the alkali and alkaline earth metal ions (peaks 3-7 and 12 in Fig. 2). For clarity, in Fig. 6, the electropherogram of Al mixed with Ag and eight other alkaline and alkaline earth metal is shown. Using sulphuric acid at pH 3.2, the Al peak trailed behind all other cations except Cr. Thus, under the above conditions, the trivalent ions were the slowest migrating ions. In the above analysis, adjustment of the pH to low acidity was necessary in order to prevent precipitation of Al(OH)$_3$. Sulphuric acid was most suitable as it formed complexes with divalent cations and retarded the migration of Ba (behind Na), and thus provided a window for Ag to migrate between K and Na. Separation of Al and Cr from other ions could be obtained at pH 3.5 but only at pH 3.2 could the two trivalent ion peaks be resolved (Cr trailed behind Al). Also, it should be noted that using any complexing agent other than sulphuric acid would cause Al peak to overlap with other divalent cation peaks. For
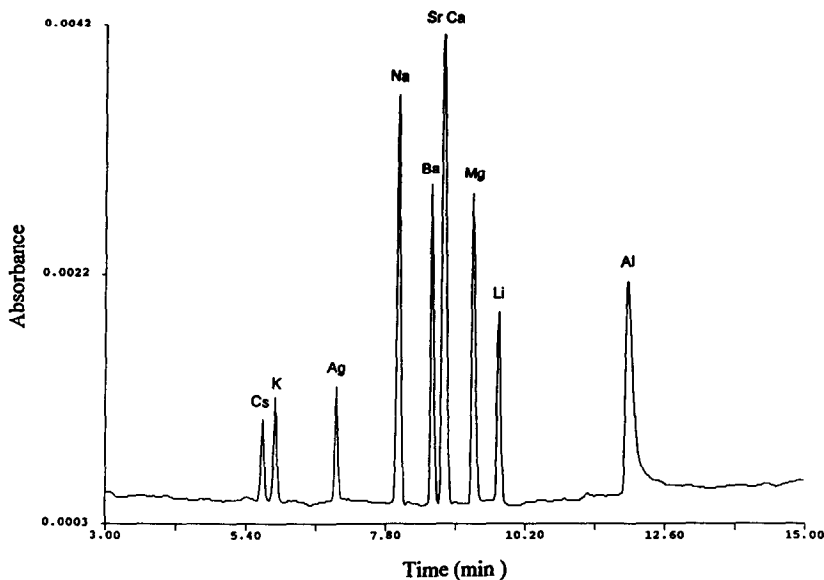
Fig. 6. Electrophoretic separation of Ag and Al from the mixtures with alkali and alkaline earth cations using 5 mM pyridine with the pH adjusted to 3.2 with sulphuric acid.

example, if glycolic acid (3-6 mM), which strongly affects the mobility of alkaline earth metal cations [15] and Al ion is used, several peaks would merge and the Al peak would be buried. Therefore, it is very difficult (if not impossible) to resolve all nineteen ions in a single CE analysis. For the determination of Ag and Al, an additional run is necessary.

3.4. Reproducibility, quantification, linearity and detection limit

The reproducibility of the CE method was studied by making five consecutive runs with all seventeen ions present at 10 mM with pyridine as the BCE (as in Fig. 2). Two injection modes were studied, a 3-s HD mode and a 3-s EK mode at +10 kV. The precisions in terms of relative standard deviation (R.S.D.) for the EK and ED modes are shown in Table 2. Both injection modes provided excellent precision for the migration time. Good precision could be obtained for peak height or area for most ions, with the exception of the ions where the mobilities were considerably different from the BCE, e.g.,

Cu in the EK mode and Cs and K in the HD mode. The precision in the HD mode was better than that in the EK mode. Also for quantification purposes it seems to be better to use peak height than peak area, as the former had better reproducibility for most ions. However, for plotting calibration graph, using peak area provided a larger linear scaling than using peak height.

The linearity of the calibration graphs expressed as peak area vs. metal ion concentration was evaluated in the concentration range 10 μequiv.-1 mequiv. for the HD mode and 1 μequiv.-1 mequiv. for the EK mode. Fig. 7A displays typical calibration lines for K, Ca, Zn and Cd. Note that the sensitivity varies greatly from ion to ion. For most ions, a good linearity ($R^2 = 0.9995-0.9999$) could be obtained in the HD mode over a concentration range where the highest and lowest standard differed by a factor of 100. However, in the EK mode, the calibration graphs were hyperbola-shaped (e.g., the Zn curve in Fig. 7B). In the EK mode the sensitivity was much better in the lower concentration range. Therefore, for quantitative analysis, the sample should be serially diluted

Table 2
Precision: comparison between HD and EK injection modes

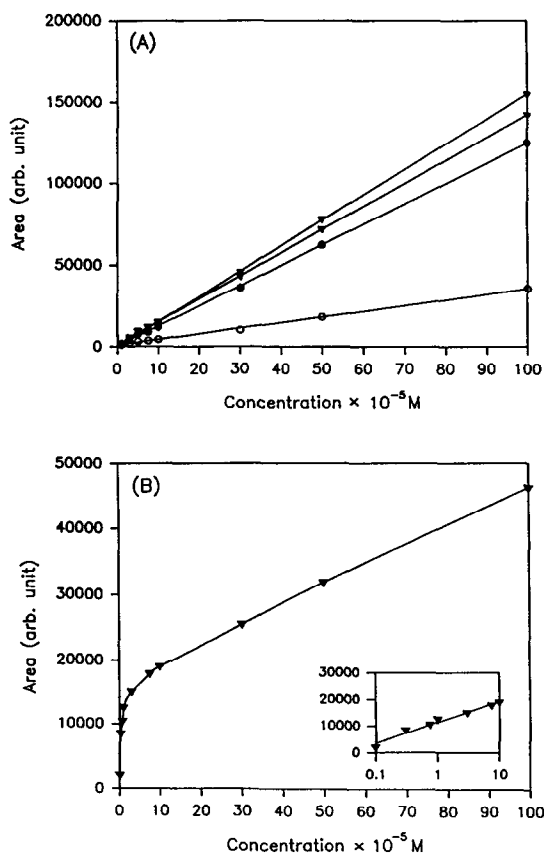| Parameter	R.S.D. (%)[a]	
	Hydrodynamic injection	Electrokinetic injection
Migration time	0.29–0.76	0.62–2.24
Peak height	1.1–4.0, except for K, Cs, 5.0–6.4	1.2–4.3, except for Cu, 7.6
Peak area	Ni, Pb, Mg, Zn, Ba, Co, Fe, Cr, Sr, Mn, 1.7–4.8 Ca, Li, Cd, Cu, 5.3–6.3 Na, K, Cs, 6.5–8	Ca, Mg, Co, Cr, Sr, Cs, Fe, Na, Ba, 1.6–4.5 K, Li, Ni, Pb, Cd, 5.5–8.0 Cu, 21

[a] Electrophoretic conditions as in Fig. 2. $n = 5$.

Fig. 7. Comparison of calibration graphs for selected metal ions in the (A) HD and (B) EK modes. O = K; ● = Ca; ▽ = Cd; ▼ = Zn. Only the Zn ion graph is shown in the EK mode. The inset is an enlargement for concentrations below 0.1 mM. Electrophoretic conditions as in Fig. 2.

such that the concentration of the analyte falls into the range where the calibration graph is linear.

The detection limit (DL) for the various metals was studied by diluting the standard solution serially and comparing the appearance of the peaks of each species in the electropherograms between the diluted standards and the diluent. The concentration that was one level higher in the series than that in which the S/N ratio fell below 3 was defined as the DL. The DL values for the various metals using the EK mode (which gave better sensitivity than the HD mode) are compared in Table 3. The better detection sensitivity in the EK mode probably was due to the "stacking" effect. The stacking effect occurred when the ion concentration in the sample plug was considerably lower than the leading electrolyte in the separation buffer. This phenomenon led to concentration of the analyte ions. Thus, the non-linearity of the calibration graph run in the EK mode could also be attributed to the stacking effect.

4. Conclusions

We have developed a rapid and reliable capillary electrophoretic method for the determination of seventeen metal ions. A complete separation of these metal ions can be accomplished in

Table 3
Detection limits of various metal ions

Metal	Detection limit		Metal	Detection limit	
	μM	ppb		μM	ppb
Na	0.001	0.02	Cd	0.1	11.2
Mg	0.01	0.2	Fe	0.3	17
K	0.01	0.4	Ni	0.5	29
Ca	0.01	0.4	Cs	0.5	66
Li	0.1	0.7	Ba	0.5	69
Mn	0.1	5.5	Pb	0.5	104
Co	0.1	5.9	Cu	2	127
Zn	0.1	6.5	Cr	4	208
Sr	0.1	9			

Minimum detectable concentration at $S/N > 3$; 5 s EK injection at 10 kV; separation conditions as in Fig. 2.

15 min with pyridine or imidazole as the background electrolyte and glycolic acid as the metal-complexing agent (at pH 4.0). Peak detection was done by indirect UV absorbance measurements at 210 or 254 nm. This method has excellent reproducibility for migration time and good precision for peak height and peak area in order to determine metal ion species and their concentrations accurately. The calibration linearity is good over a 100-fold range of concentration. With successive serial dilutions, real samples can be analysed at the sub-ppb to sub-ppm level. The results obtained so far show that the CE methods developed here are well suited for both qualitative and quantitative analyses for metals in water, food and soil samples. A CE method has also been developed for determining Ag and Al, but must be performed separately using 5 mM pyridine with the pH adjusted to 3.2 with sulphuric acid.

5. Acknowledgements

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