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Role of ligand purity in separations of alkaline earth metals as arsenazo I complexes by capillary zone electrophoresis

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Abstract

In the separation of metal ions by capillary electrophoresis in the form of kinetically labile complexes formed through the incorporation of an auxiliary complexing ligand into the background electrolyte (BGE), it has been shown that the purity of this auxiliary complexing ligand can play a crucial role in the selectivity and efficiency of the resultant separations. Using the separation of alkaline earth metals as complexes with the metallochromic ligand arsenazo I as a model system, the effects of the addition of low concentrations of simulated impurities, in the form of various metal ions and competing ligands, were studied. Additions of Fe^{III} at low micromolar levels to a BGE containing 1 m*M* arsenazo I resulted in severe peak tailing. The addition of the competing ligands diethylenetriaminepentaacetic acid (DTPA) or arsenazo III, at a molar ratio as low as 1:1000 to arsenazo I, also caused substantial peak broadening and altered the separation selectivity. The practical implications of the above results for the separation of metals as labile complexes, using capillary zone electrophoresis (CZE) systems similar to the above, are discussed. © 1998 Elsevier Science B.V.

Keywords: Complexation; Ligand purity; Alkaline earth metals; Metal complexes; Arsenazo I; Metal cations

1. Introduction

Auxiliary ligands have been used extensively in separations of metal ions, both to facilitate the detection and to manipulate separation selectivity, in liquid chromatography (LC) [1–3] and increasingly also in capillary electrophoresis (CE) and related electromigration separation methods, such as micellar electrokinetic chromatography (MEKC) [4–6]. Despite the number of studies and applications carried out using LC or CE, the role that the purity of the auxiliary ligand plays in the separation of metal ions has not been investigated or discussed in any great detail. This is in itself surprising since it is well known that commercial sources of many ligands, particularly metallochromic ligands, are notoriously impure even when these are supplied as ''analytical grade'' reagents.

It is true to say that if any effects of ligand purity on the separation of metal ions were to be observed, they are likely to be more easily identified when using CE because of the generally higher separation efficiencies which can be obtained in CE compared to LC. In CE, the use of an auxiliary ligand for the separation of metal ions usually involves either pre-capillary complexation or on-capillary complexation [4,5]. When pre-capillary complexation is used, stable and often kinetically inert complexes of the metal ions are formed prior to injection and are separated as charged (CE) or neutral (MEKC)

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complexes [4,5,7]. In this case, the total complexation with the ligand can be regarded as a derivatisation of the analyte metal ion and any impurity in the ligand is likely to show itself as an extra peak for each analyte, provided the impurity is capable of metal complexation and can be detected in the same way as the main analyte complex [1].

A totally different situation exists if the separation of metal ions is carried out as kinetically labile complexes using an auxiliary ligand which has been included in the background electrolyte (BGE) [4,5]. Here the complexation equilibria govern the separation, especially when the metal ions are only partially complexed with the auxiliary ligand and the effective mobility of a metal ion analyte is a weighted average of the contributions of the mobilities of both the metal cation and the metal complex [4,5]. When the kinetics of complexation equilibria are insufficiently fast to permit attainment of equilibrium during migration in the electric field, additional zone broadening (called electrodiffusion) results [8]. Therefore the presence of minor concentrations of impurities in the BGE which can exert an effect on these equilibria can, in principle, change the separation selectivity and/or the efficiency. These impurities can be (i) complexing ligands which compete with the main auxiliary ligand for the analyte metal ions, or (ii) metal ion impurities in the BGE which undergo complexation equilibria with the auxiliary ligand. In this type of separation system, the role of the purity of the auxiliary ligand (and other chemicals) can therefore be anticipated as having the potential to influence both separation selectivity and efficiency.

Metallochromic ligands form a special group of auxiliary ligands which have the advantage of offering very sensitive direct detection in the visible spectral range [4,5,9–31]. In the majority of cases they form stable complexes and pre-capillary complexation is used. However, on capillary complexation has also been reported in some cases [12,30,31]. We have recently shown separations of metal ions complexed with metallochromic ligands when partial complexation [4,5] was applied [31–35], in which case the ligand purity can be expected to exert an effect on the separation.

The aim of the present study was to investigate the role of ligand purity on the separation of metal ions as kinetically labile complexes by CE with a metallochromic ligand contained within the BGE. The study was carried out using the separation of alkaline earth metal ions as complexes with arsenazo I as a model system.

2. Experimental

2.1. Instrumentation

A Polymicro Technologies (Phoenix, AZ, USA) fused-silica capillary, 0.60 m (0.52 m to detector)× 75 μm I.D. was used. This capillary was initially flushed with the acetate–diethanolamine (DEA) BGE for 10 min and left overnight before use. The instrument used was a Quanta 4000 (Waters, Milford, MA, USA) interfaced to a Maxima 820 data station (Waters). Additional to the original mercury lamp, a light-emitting diode (LED) using a 568 nm LED (HLMP8509, Farnell Electronic Components, NSW, Australia) was fitted to the instrument, as described elsewhere [30].

2.2. Reagents

Arsenazo I [2-(4,5-dihydroxy-2,7-disulfo-3-naphthylazo) phenylarsonic acid], was purchased, (i) as the trisodium salt (indicator quality) from Aldrich (Milwaukee, WI, USA), subsequently referred to as sample (A), and (ii) as the disodium salt (analytical-reagent grade) from Fluka (Buchs, Switzerland), sample (B). The arsenazo I (A) and (B) samples were purified twice by an acid precipitation as described in [36] resulting in purified samples of the two commercial batches. Elemental analysis results confirmed a composition for the free acid of the ligand with four molecules of water. Relative purity of the four arsenazo I samples was characterised by CE using detection at 254 nm and at 563 nm as described earlier [37], and results are shown in Table 1. The content of metal impurities was first determined semi-quantitatively using inductively coupled plasma (ICP)-MS: the only metals present in appreciable amounts were iron and calcium [apart from sodium in the case of sodium salts, arsenic as a part of the molecule, and approx. 0.1% Sb in the sample (B), which may have been an impurity of

Table 1
Relative purity of arsenazo I from peak areas (internal normalisation) at 254 nm and 568 nm and content of metal impurities

	Relative purity (%)		Content of metal impurities (mol%)	
	254 nm	568 nm	Ca	Fe
A	81.1	87.4	0.18	0.36
B	71.5	74.8	0.04	0.04
AP	97.3	98.7	0.15	0.06
BP	92.5	93.8	0.06	0.04

As]. Atomic absorption spectrometry (AAS) determinations were then carried out for iron and calcium and the results are given in Table 1.

DEA was purchased as the AR reagent from Fluka. All other chemicals were of analytical grade unless stated otherwise. Deionised water was obtained using a Millipore (Bedford, MA, USA) Milli-Q water purification apparatus. An acetate–DEA buffer was prepared by the appropriate dilution of acetic acid to give a final concentration of 20 m*M* and of DEA to give a pH of approx. 9.5 (final concentration of approx. 50 m*M*). A BGE containing arsenazo I (1.0 m*M*) was prepared from the acetate–DEA buffer and a 10 m*M* aqueous arsenazo I stock solution. A formate–DEA buffer was prepared by appropriate dilution of formic acid and adjustment to pH 9.6 with DEA, to give final concentrations of 20 m*M* formate and approximately 50 m*M* DEA. The BGE was prepared by adding appropriate amounts of EDTA and Z1-Methyl (Waters) to the formate–DEA buffer. Before use the BGEs were degassed by vacuum and filtered with a Millex-HA 0.45 μm disc filter (Millipore).

2.3. Procedures

Injection was performed hydrostatically by elevating the sample at 100 mm for 20 s at the anodic side of the capillary. Instead of the standard 20 ml buffer reservoir at the anodic side, a plastic sample vial of approximately 0.6 ml was installed inside the standard 20 ml container. At the cathodic (detector) side, a buffer reservoir of 4 ml was used. The running voltage was +30 kV.

3. Results and discussion

3.1. Choice of the model system

Arsenazo I is a metallochromic ligand which forms coloured 1:1 complexes with a number of metals. The ligand has found use as an indicator in complexometric and precipitation titrations and as a photometric reagent for metal determinations [36]. It is known to form 1:1 metal–ligand complexes (designated as ML) with the alkaline earth metals (at pH 9.5), for which stability constants have been reported [36] and are shown in Table 2. The relatively simple complex equilibria of the ML complexes can be of advantage for studies in metal separations by CE using the metallochromic ligand contained within the BGE, especially if the separated metals form kinetically labile complexes and consequently the complexation equilibria play a significant role in the separation.

In this work, evidence that the arsenazo I complexes formed with alkaline earth metal ions are kinetically labile was obtained by injections of pre-

Table 2
Conditional stability constants for Ba, Sr, Ca and Mg complexes (ML) of arsenazo I, DTPA and citrate at pH 9.5

Metal	Conditional stability constant×ligand concentration [stability constant [36], concentration (mmol/l), α_L]		
	AI	DTPA	Citrate
pK_a	8.2, 11.6	8.60, 10.55	3.12, 4.76, 6.40
Ba	2.05 (4.15, 1.0, 0.079)	4.60 (8.63, 0.001, 0.11)	2.73 (2.73, 1.0, 1.0)
Sr	2.11 (4.41, 1.0, 0.079)	5.65 (9.68, 0.001, 0.11)	3.02 (3.02, 1.0, 1.0)
Ca	2.99 (5.09, 1.0, 0.079)	6.71 (10.74, 0.001, 0.11)	3.5 (3.5, 1.0, 1.0)
Mg	3.48 (5.58, 1.0, 0.079)	5.27 (9.3, 0.001, 0.11)	3.5 (3.5, 1.0, 1.0)

capillary formed complexes into a BGE (acetate–DEA pH 9.5, see Section 2.2) which did not contain arsenazo I: no peaks for the complexes of the metal ion analytes were obtained. This was due to dissociation of the complexes before reaching the detector. Consequently, it was necessary for arsenazo I to be added to the BGE to ensure that the metal ion analytes remained in the complexed form (or at least a proportion thereof, according to the complex equilibria in the BGE under the given conditions) as they passed through the capillary.

3.2. Influence of ligand purity on the separation of alkaline earth metals

Interestingly, quite different separations of alkaline earth metals were obtained when using different commercial batches of arsenazo I added to the BGE: namely batches (A) and (B) and the two purified batches (AP) and (BP) (see Section 2.2). The differences in both separation efficiency and selectivity (Fig. 1) were most noticeable with barium and strontium, although calcium and magnesium also showed increased peak tailing.

In an effort to explain the observed differences, the effect of the concentration of the auxiliary ligand (arsenazo I) was first investigated as this is a parameter which is known to change the separation selectivity and/or efficiency [3,4]. It can be expected that due to the varying purities of the batches of arsenazo I, the BGE concentration of arsenazo I, when made up from each batch, was likely to vary (Table 1). Therefore it was necessary to determine whether such variations in concentration could be causing the observed effects on the separation. For this investigation the batch of arsenazo I producing the most efficient separation was taken (batch BP) and the BGE concentration was varied between 0.04 and 5.12 m*M*. As expected, for all four metal ions their effective mobilities changed according to the ligand concentration in the BGE, following a trend from relatively positive effective mobilities (cationic metal ions) at low ligand concentration, to relatively negative effective mobilities (anionic metal complexes) at high ligand concentration (Fig. 2). At the same time, at any particular ligand concentration the mobilities of the four metal ions followed the trend in stability constants for these metals with arsenazo I

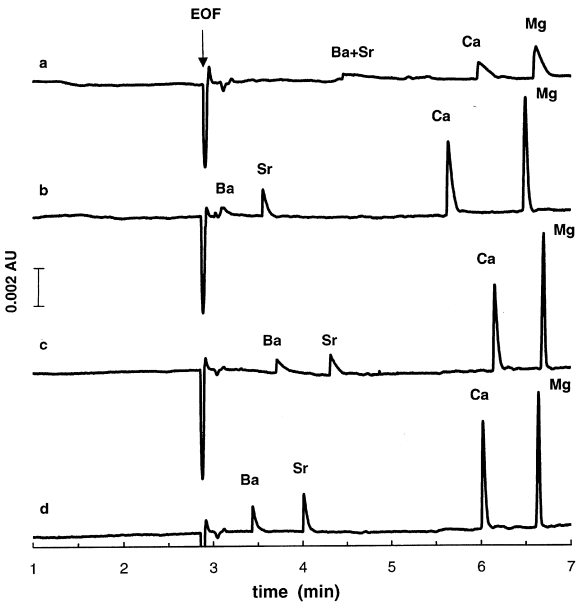

Fig. 1. Electropherograms of separations of Ba^{II}, Sr^{II}, Ca^{II} and Mg^{II} using different batches of arsenazo I within the BGE: (a) A, (b) B, (c) AP and (d) BP. Capillary: fused-silica 0.60 m (0.52 m to detector)×75 μm I.D.; BGE: 1.0 m*M* AI in 17 m*M* acetate–DEA BGE (pH 9.5); separation voltage: +30 kV (37 mA); temperature: 25°C; detection: 568 nm (LED); injection: hydrostatic (5 s); for other conditions see Section 2.

(Table 2). However, as is evident from, the changes in arsenazo I concentration did not explain the detrimental effects on the selectivity and efficiency of the separation seen in Fig. 1.

These results suggested that some impurities present in the ligand may have exerted a detrimental effect on the separation. Fig. 3 shows the electropherograms obtained for each batch of arsenazo I using an earlier published method [37]. Several impurity peaks are evident, particularly in the unpurified batches (A and B). Somewhat surprisingly, the differences shown in do not correlate with the overall batch purity determined by CE according to the above method (see Fig. 3 and Table 1). Consequently, the impurities that produced the detrimental effects on the separations shown in Table 1 were likely to be minor components only. The following two possibilities have to be considered:

(i) Metal ions present in the arsenazo I as impurities could have a detrimental effect on the separation through adsorption onto the capillary

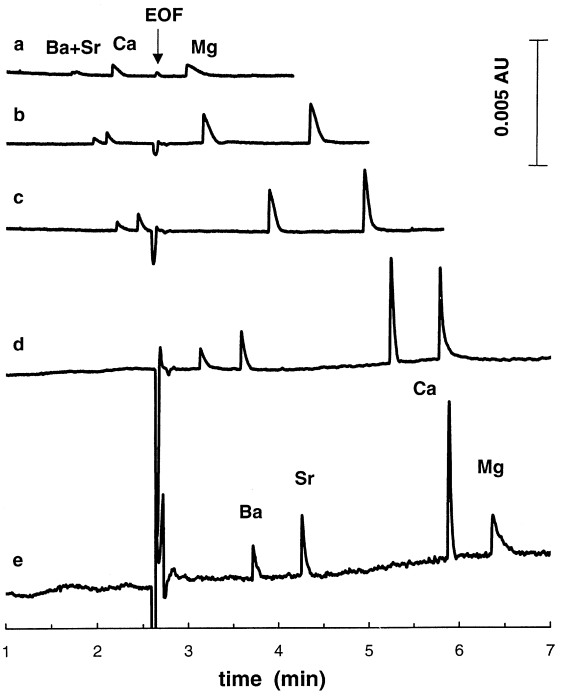

Fig. 2. Electropherograms of separations of Ba^{II}, Sr^{II}, Ca^{II} and Mg^{II} using different concentrations of arsenazo I BP within the BGE: (a) 0.04 m*M*, (b) 0.16 m*M*, (c) 0.32 m*M*, (d) 1.28 m*M* and (e) 5.12 m*M*. Capillary: fused-silica 0.60 m (0.52 m to detector)× 75 μm I.D.; BGE: 0.04 to 5.12 m*M* AI BP in 17 m*M* acetate–DEA BGE (pH 9.5); separation voltage: +30 kV (37 mA); temperature: 25°C; detection: 568 nm (LED); injection: hydrostatic (5 s); for other conditions see Section 2.

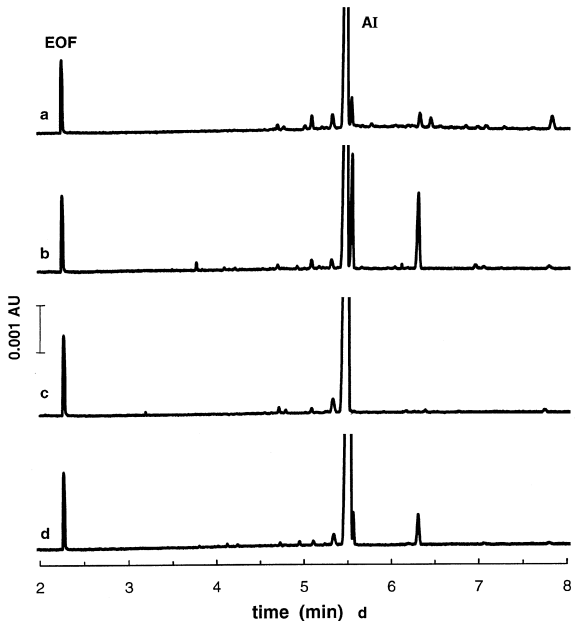

Fig. 3. Electropherograms at 254 nm of different batches of arsenazo I: (a) A, (b) B, (c) AP and (d) BP. Capillary: fused-silica 0.60 m (0.52 m to detector)×75 μm I.D.; BGE: 1.0 m*M* EDTA in 20 m*M* formate–DEA BGE (pH 9.6); separation voltage: +30 kV (41 μA); temperature: 25°C; detection: 254 nm (mercury lamp); injection: hydrostatic (5 s); for other conditions see Section 2.

walls, in a manner similar to the effect observed by Gassner et al. [38]. The analyses shown in Table 1 reveal that low levels of iron and calcium were found in all four arsenazo I batches.

(ii) Alternatively, impurities which form more thermodynamically stable complexes with the metal ion analytes, compared to arsenazo I itself, could act as competing ligands in the BGE. These would therefore complex a portion of the analyte ions, and if the complex of this competing ligand had a different mobility from that of the main auxiliary ligand (arsenazo I) and slow complexation kinetics, this would result in zone broadening of the analyte. Since the thermodynamic stability of the arsenazo I complexes with alkaline earth metal ions decrease from magnesium to barium, with barium in particular being only partially complexed (as is evident from the dependence of mobilities on arsenazo I concentration shown in Fig. 2), it is likely that barium (followed by strontium) would be most affected by a competing ligand in the BGE.

3.3. Simulations of arsenazo I impurities by additions of metal ions or competing ligands into the BGE

For the above reasons the effects of additions of metal ions and some competing ligands into the BGE containing arsenazo I were investigated. For these experiments the arsenazo I batch (BP) giving the best separation efficiencies (Fig. 1d) was again used. The following additions, over a range of concentrations, to the BGE were made: Fe^{III} (Fig. 4), Ca^{II} (not shown), arsenazo III (Fig. 5), DTPA (Fig. 6) and citrate (Fig. 7).

The concentration range studied for additions of metal ions into the BGE was 1–100 μ*M*. Additions of Fe^{III} at low μ*M* levels resulted in peak broadening

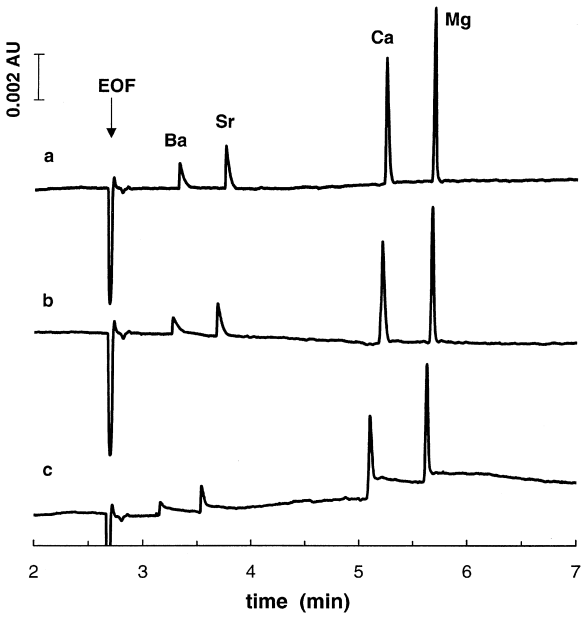

Fig. 4. Electropherograms of separations of Ba^{II}, Sr^{II}, Ca^{II} and Mg^{II} using arsenazo I within the BGE with additions of Fe(III): (a) 0 μ*M*, (b) 5 μ*M* and (c) 20 μ*M*. Capillary: fused-silica 0.60 m (0.52 m to detector)×75 μm I.D.; BGE: 0 to 20 μ*M* Fe(III) and 1.0 m*M* AI in 17 m*M* acetate–DEA BGE (pH 9.5); separation voltage: +30 kV (37 mA); temperature: 25°C; detection: 568 nm (LED); injection: hydrostatic (5 s); for other conditions see Section 2.

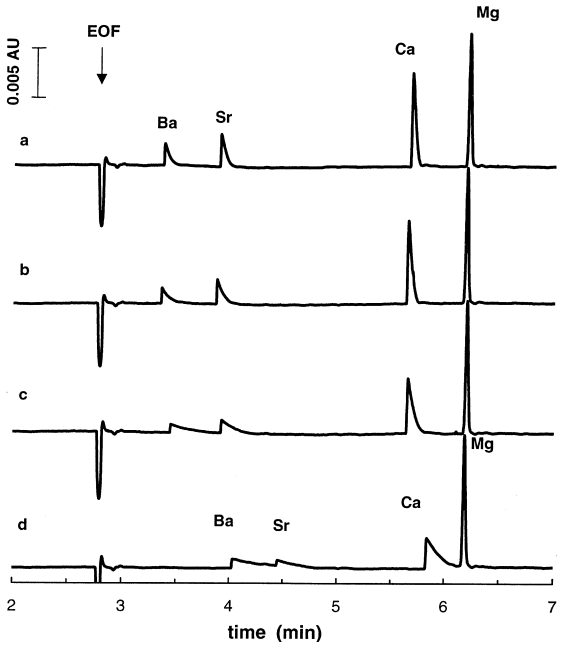

Fig. 5. Electropherograms of separations of Ba^{II}, Sr^{II}, Ca^{II} and Mg^{II} using arsenazo I within the BGE with additions of arsenazo III: (a) 0 μ*M*, (b) 1 μ*M*, (c) 5 μ*M* and (d) 20 μ*M*. Capillary: fused-silica 0.60 m (0.52 m to detector)×75 μm I.D.; BGE: 0 to 20 m*M* arsenazo III and 1.0 m*M* AI in 17 m*M* acetate–DEA BGE (pH 9.5); separation voltage: +30 kV (37 mA); temperature: 25°C; detection: 568 nm (LED); injection: hydrostatic (5 s); for other conditions see Section 2.

(see 1, 5 and 20 μ*M* Fe^{III} additions in Fig. 4a–c). At higher concentrations the resulting electropherograms (not shown) were similar to that with 20 μ*M* Fe^{III}, although the background was much noisier. The observed effect of Fe^{III} additions on the peak shapes of separated metals can be caused by either or both of the following two phenomena:

(i) Slow kinetics of complexation equilibria of the arsenazo I with the added Fe^{III}, which may influence the complex equilibria of arsenazo I with the alkali earth metal ions, or (ii) sorption of the metal ion on the capillary walls and secondary interactions with the ligand and metal complexes in the BGE. Some degree of sorption of the Fe^{III} is likely as the peak shape observed for barium and strontium deteriorated permanently after the addition of Fe^{III}, even when it was then removed from the BGE. A new capillary was necessary to restore the original peak shapes. Addition of calcium in the range of 1–100 μ*M* (not shown) did not have such pronounced effects as for Fe^{III} and caused only slight broadening of peak shapes and changes in migration times.

When looking at the concentrations found for iron and calcium in the four batches of arsenazo I (Table 1), the presence of these species cannot entirely explain the detrimental effects observed using the unpurified arsenazo I batch (A) in Fig. 1a. The additions of Fe^{III} (Fig. 4a–c) caused pronounced peak tailing, but not peak broadening and a change in selectivity as observed in Fig. 1. It was therefore likely that some other impurities were also exerting an influence.

Additions of several competing ligands, each exhibiting different degrees of complexation with the alkaline earth metal ions (Table 2) were studied. The ligands used were arsenazo III, DTPA and citrate and the resultant electropherograms are shown in Figs. 5–7. Arsenazo III and DTPA interfered with the separation and caused peak broadening at concen-

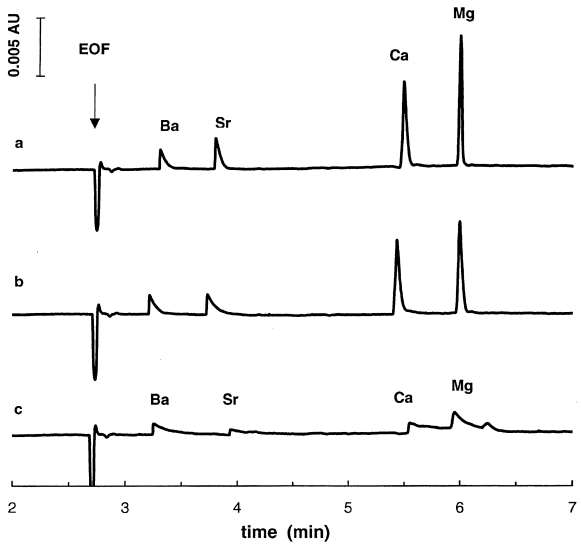

Fig. 6. Electropherograms of separations of Ba^{II}, Sr^{II}, Ca^{II} and Mg^{II} using arsenazo I within the BGE with additions of DTPA: (a) 0 μ*M*, (b) 1 μ*M* and (c) 5 μ*M*. Capillary: fused-silica 0.60 m (0.52 m to detector)×75 μm I.D.; BGE: 0 to 5 m*M* DTPA and 1.0 m*M* AI in 17 m*M* acetate–DEA BGE (pH 9.5); separation voltage: +30 kV (37 mA); temperature: 25°C; detection: 568 nm (LED); injection: hydrostatic (5 s); for other conditions see Section 2.

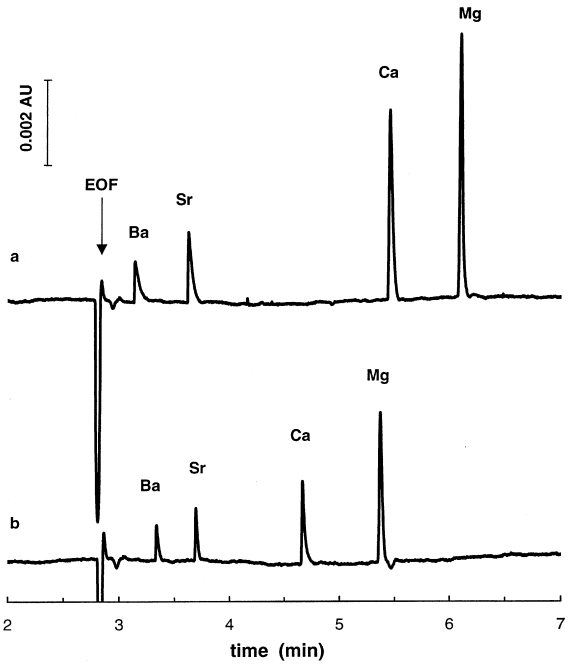

Fig. 7. Electropherograms of separations of Ba^{II}, Sr^{II}, Ca^{II} and Mg^{II} using arsenazo I within the BGE with additions of citrate: (a) 0 μ*M* and (b) 1 m*M*. Capillary: fused-silica 0.60 m (0.52 m to detector)×75 μm I.D.; BGE: 0 or 1 m*M* citrate and 1.0 m*M* AI in 17 m*M* acetate–DEA BGE (pH 9.5); separation voltage: +30 kV (37 mA); temperature: 25°C; detection: 568 nm (LED); injection: hydrostatic (5 s); for other conditions see Section 2.

trations as low as 1 μ*M*, which is 1:1000 (molar ratio) relative to arsenazo I. There are several conditions that must be fulfilled to make possible an interference by a competing ligand at such a low level:

(i) From a thermodynamic viewpoint, the interfering ligand must form a more stable complex with a particular metal than does arsenazo I. This is known to be the case for DTPA (see Table 2). Species distribution diagrams calculated for the conditions in the BGE used, shown in Fig. 8, also illustrate the feasibility of the competing complexation for the case of DTPA. Although there are no precise data on stability constants for individual alkaline earth metals with arsenazo III, a $K(ML)\sim6$ is given for calcium and arsenazo III is known to form complexes with this group of metals [36].

(ii) The metal complexes of such a competing ligand must have a different mobility from that of the arsenazo I complex, which is quite probable for the ligands used.

(iii) The kinetics of at least some of these competing complexation equilibria must be slow so that they result in additional peak broadening. The last condition is obviously not fulfilled for citrate, which when added even at a relatively high concentration (1 m*M*, Fig. 7) exhibited only an influence on the migration times and somewhat decreased the detection sensitivity (since only the portion of the metal ion analyte complexed with arsenazo I was detected), but the peak shapes of all four metals were not affected in any negative way.

3.4. Practical implications

Firstly, the ligand and other reagents used must be free from any other ligands that would, under the conditions used, form comparatively stable (with respect to the main auxiliary ligand) complexes with the metals to be separated, particularly if these complexes are to some extent kinetically inert.

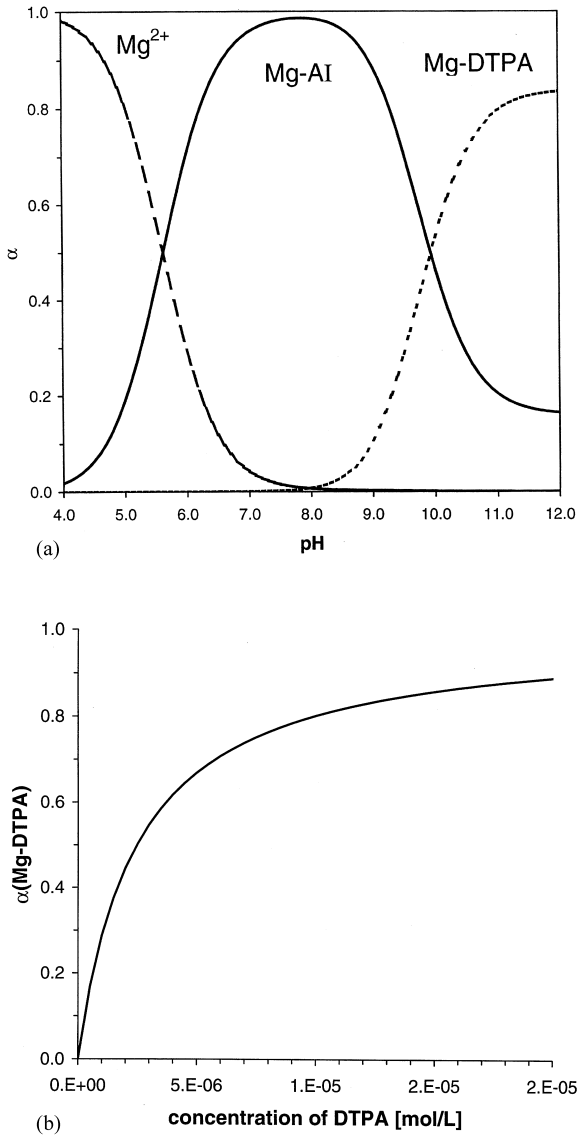

Fig. 8. Distribution diagrams (a) for Mg^{2+} ions and complexes Mg–arsenazo I and Mg–DTPA as a function of pH for 1 m*M* arsenazo I and 1 m*M* DTPA (a) and distribution coefficient for the Mg–DTPA complex as a function of DTPA concentration for 1 m*M* arsenazo I and pH 9.5 (b).

Secondly, the ligand and other reagents used should be free of metals (especially tri- or higher valency) which form complexes with the auxiliary ligand, since these metals can not only participate in the complexation equilibria and increase the background absorbance and baseline noise, but may also adsorb on the fused-silica capillary wall. This may occur even in a BGE with an excess of ligand and will establish a secondary influence on the peak shapes of the separated metal ions through adsorption of the ligand.

4. Conclusions

When performing separations of metals complexed with an auxiliary ligand in which the complexes are not kinetically inert, i.e., they are in equilibrium with other components of the solution, the purity of the auxiliary ligand can have a major effect on the separation achieved. The presence of complexing impurities that form stable complexes with relatively slow kinetics of complex formation/dissociation can cause deterioration of peak shapes at concentrations as low as 1:1000 relative to the auxiliary ligand. Consequently, ligand purity is crucial if efficient and reproducible separations are to be achieved. Furthermore, it is clear that both the ligand and other chemicals used should also be free of metals capable of complex formation with the ligand. The findings of these investigations carried out using a metallochromic ligand as a model system can also be expected to be valid for other auxiliary ligands used in similar systems.

Acknowledgements

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