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Review

Comparison of ion chromatography and capillary electrophoresis for the determination of inorganic ions

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Abstract

The techniques of ion chromatography and capillary electrophoresis are compared as analytical methods for the determination of inorganic anions and cations. Comparison is made in the areas of stage of development, separation efficiency, separation selectivity, analytical performance parameters, method development procedures, applications, strengths and weaknesses, and future directions. It is shown that the two techniques are complementary rather than competitive, especially with regard to their separation selectivities and the type of applications to which they are most suited.

Keywords: Reviews; Inorganic anions; Inorganic cations

Contents

1. Introduction

Ion chromatography (IC) has been in use since 1975 [1] and has become a routine analytical method for the determination of inorganic ions, especially anions. The theory and applications of IC have been discussed in detail in a number of reference texts [2–7]. Capillary electrophoresis (CE) was first demonstrated as an inorganic analytical technique in 1990 [8], and since that time there has been increasing attention paid to this method [9–13]. Many IC users are considering either replacement of their IC

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methods with CE, or using CE as a supplementary method. It is the purpose of this review to explore some of the relative advantages and disadvantages of the two techniques and to suggest the future roles that they will play in inorganic analysis.

2. Definitions

Before embarking on any comparisons, it is first necessary to define each technique. IC should not be considered as a single chromatographic technique, since the term refers to the use of a variety of liquid chromatographic methods applied to the determination of a reasonably specific group of analytes [2]. The separation techniques embraced by IC include ion-exchange chromatography, ion-exclusion chromatography and reversed-phase ion-interaction chromatography. Detection methods include conductivity (both suppressed and non-suppressed), spectrophotometry (both direct and indirect), electrochemistry (amperometry and potentiometry) and post-column reaction detection. The analytes considered to be the domain of IC include inorganic anions and cations and low-molecular-mass organic acids and bases (that is, those acids and bases in which the ionic functionality exerts the predominant influence on retention).

Discussion of all the above separation and detection methods and analytes is impractical for a review of this type, so for simplicity we will confine the definition of IC to what can be considered to be its mainstream activity, namely: *the separation of inorganic anions and cations by ion exchange, with detection by suppressed conductivity.*

Representative chromatograms for inorganic anions and cations are shown in Figs. 1 and 2, respectively.

The precise meaning of CE also needs some clarification. The separation variables are more limited than in IC, but several distinct approaches can be identified, including capillary zone electrophoresis, micellar electrokinetic capillary chromatography, capillary electrochromatography, etc. Again a variety of detection methods exists, such as direct or indirect spectrophotometry, suppressed or non-suppressed conductivity, amperometry, laser-induced fluorescence and mass spectrometry. Moreover, one

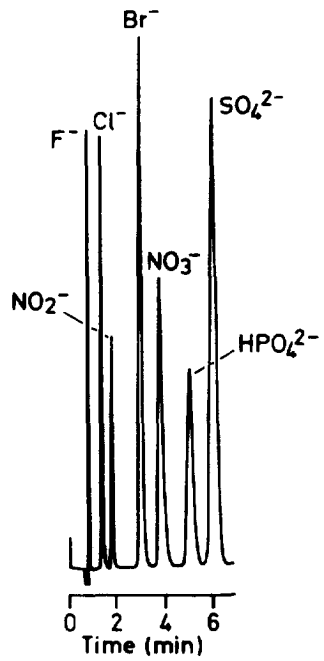

Fig. 1. Separation of anions by ion chromatography with suppressed conductivity detection. A Dionex IonPac-AS4A agglomerated anion-exchange column was used with a 1.8 m*M* sodium carbonate–1.7 m*M* sodium bicarbonate eluent. Chromatogram courtesy of Dionex.

must specify whether the separation is performed with the electrophoretic migration of the ions being in the same direction as the electroosmotic flow (the co-EOF mode) or in the opposite direction to the electroosmotic flow (the counter-EOF mode). The range of analytes applicable to CE is very broad, but if one considers only those analytes which overlap with IC it can be seen that CE has been applied to all such solutes, namely inorganic anions and cations and low-molecular-mass acids and bases.

Taking the same approach used earlier for IC and identifying the mainstream application of CE, the following focus for CE can be defined as a basis for discussion: *the separation of inorganic anions and cations by capillary zone electrophoresis using the co-EOF mode and indirect spectrophotometric detection.*

Representative electropherograms obtained for inorganic anions and cations are shown in Fig. 3 and Fig. 4, respectively.

Armed with the above definitions, a meaningful

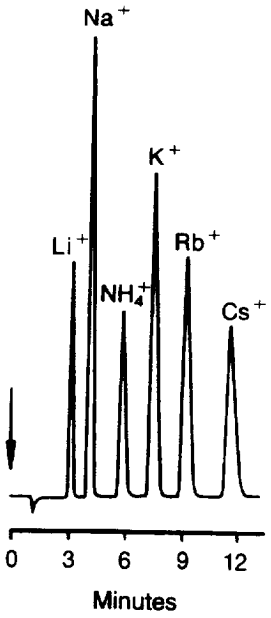

Fig. 2. Separation of alkali metal cations by ion chromatography with suppressed conductivity detection. A Dionex Fast-Sep Cation I column was used with 30 m*M* hydrochloric acid as eluent. Chromatogram courtesy of Dionex.

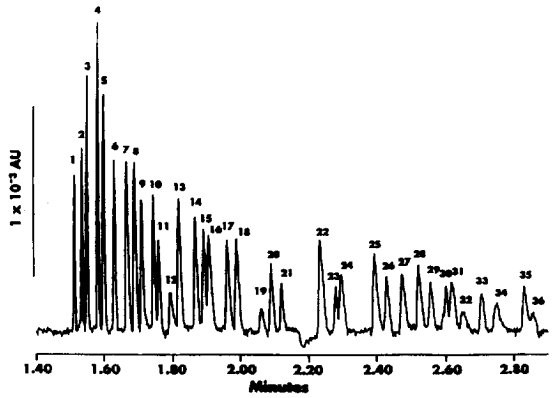

Fig. 3. Separation of anions by co-EOF capillary electrophoresis using indirect spectrophotometric detection. A 5 m*M* chromate electrolyte containing Waters OFM Anion-BT at pH 8.0 was used. Injection was by electromigration at 1 kV for 15 s. Solute identities: 1=thiosulfate, 2=bromide, 3=chloride, 4=sulfate, 5=nitrite, 6=nitrate, 7=molybdate, 8=azide, 9=tungstate, 10=monofluorophosphate, 11=chlorate, 12=citrate, 13=fluoride, 14=formate, 15=phosphate, 16=phosphite, 17=chlorite, 18=glutarate, 19=*o*-phthalate, 20=galactarate, 21=carbonate, 22=acetate, 23=chloroacetate, 24=ethanesulfonate, 25=propionate, 26=propanesulfonate, 27=*dl*-aspartate, 28=crotonate, 29=butyrate, 30=butanesulfonate, 31=valerate, 32=benzoate, 33=*l*-glutamate, 34=pentanesulfonate, 35=*d*-gluconate, 36=*d*-galacturonate. Reproduced with permission from Ref. [10].

comparison of IC and CE can now be undertaken. This comparison will include the areas of developmental overview, separation efficiency, separation selectivity, analytical performance parameters, method development procedures, applications, relative strengths and weaknesses, and future directions.

3. Developmental overview

The most readily apparent aspect which emerges from a comparison of IC and CE is that the two techniques are at very different stages of development. In a recent review, Lucy [14] used Laitinen's "Seven Ages of an Analytical Method" [15] in order to classify the stage of development of IC. In brief, these ages are (i) initiation phase, (ii) validation phase, (iii) development of instrumentation, (iv) development of principles and mechanisms, (v) applications, (vi) inclusion in standard methods and (vii) senescent phase. Lucy placed IC in the sixth age and gave evidence to support this contention. He also noted that in order for IC to move into the seventh age, a technique offering advantages in speed, economy, convenience, sensitivity and reliability must emerge. This review will address whether CE is such a technique.

The same seven ages can be used to classify the stage of development of CE. At the present time the literature suggests that there is quite strong activity in ages (ii)–(v) inclusive. Publications on validation [16], instrumentation [17], principles and mechanisms [18] and applications [19] have appeared recently. Irrespective of the precise stage of development of CE, it is readily apparent that CE is much less developed than IC.

4. Separation efficiency

The most striking difference between the IC chromatograms in Figs. 1 and 2 and the electropherograms in Figs. 3 and 4 is the separation efficiency. If one uses the number of theoretical plates to quantify the efficiency (remembering that

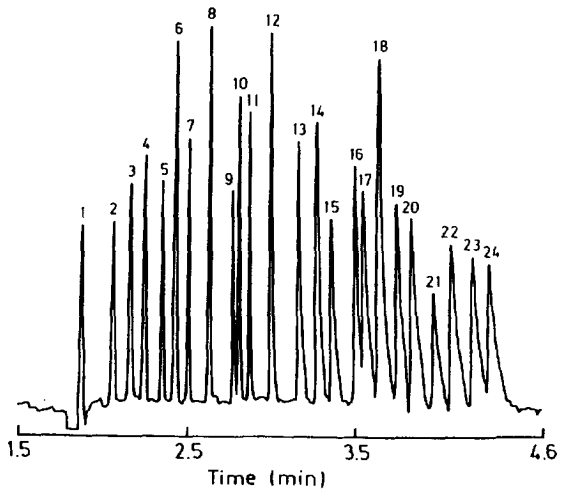

Fig. 4. Separation of cations by co-EOF capillary electrophoresis with indirect spectrophotometric detection. The electrolyte was 15 m*M* lactic acid and 10 m*M* 4-methylbenzylamine at pH 4.3. Peak identities: 1=K^+, 2=Ba^{2+}, 3=Sr^{2+}, 4=Ca^{2+}, 5=Mg^{2+}, 6=Mn^{2+}, 7=Cd^{2+}, 8=Co^{2+}, 9=Pb^{2+}, 10=Ni^{2+}, 11=Zn^{2+}, 12=La^{3+}, 13=Ce^{3+}, 14=Pr^{3+}, 15=Nd^{3+}, 16=Sm^{3+}, 17=Gd^{3+}, 18=Cu^{2+}, 19=Dy^{3+}, 20=Ho^{3+}, 21=Er^{3+}, 22=Tm^{3+}, 23=Yb^{3+}, 24=Lu^{3+}. Electropherogram courtesy of J.S. Fritz.

the concept of a theoretical plate is not strictly applicable to CE) then there is an approximate 50-fold increase in efficiency between the two techniques (4000 plates for sulfate in Fig. 1 and 250 000 plates for sulfate in Fig. 3.

At first glance this increase in efficiency would appear to endow CE with an enormous advantage over IC. However, it is instructive to examine the peak capacities (that is, the number of peaks which can be baseline resolved in a given time window) of each technique. To do this one must first consider the manner in which efficiency varies over the specified separation time. Fig. 5 shows the variation of the number of theoretical plates with the capacity factor of the peak used for the calculation of efficiency, for each of the peaks shown in the chromatogram forming part of the figure. It can be seen that the efficiency reaches a fairly constant level for peaks with capacity factors in the range 2–10. The peak capacity calculated for the time window of 1–20 min is aproximately 32. This process can be repeated for a typical electropherogram for CE and Fig. 6 shows the results obtained. Here, the electropherogram shows a separation of some inorganic anions and some aliphatic sulfonic acids using chromate as the electrolyte. It can be seen that the plot of the number of theoretical plates (N) versus analyte mobility reaches a maximum when the analyte mobility is close to that of the electrolyte. When there is a difference between the mobilities of the analyte and

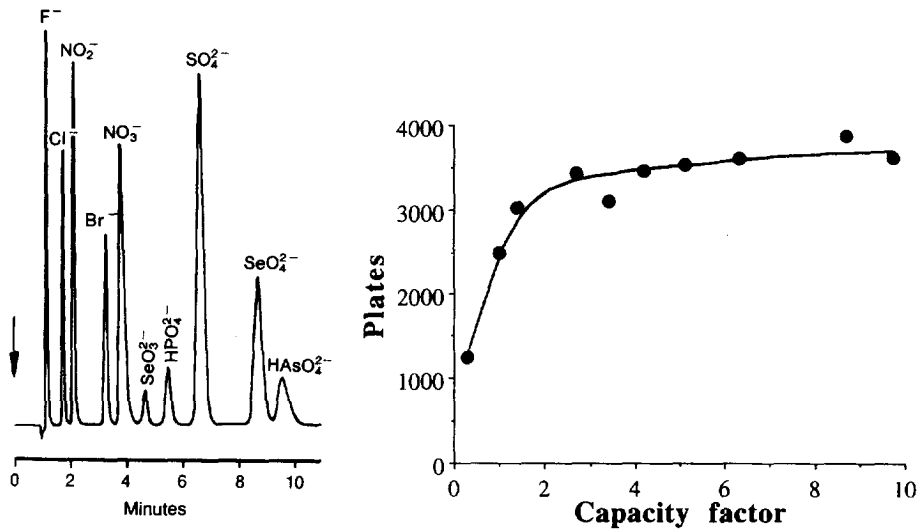

Fig. 5. Variation of the number of theoretical plates calculated for each peak in the chromatogram shown. The chromatogram was obtained using a Dionex IonPac AS4A column with 7.5 m*M* sodium bicarbonate and 2 m*M* sodium carbonate as eluent. The number of theoretical plates was calculated using the 5σ method. Chromatogram courtesy of Dionex.

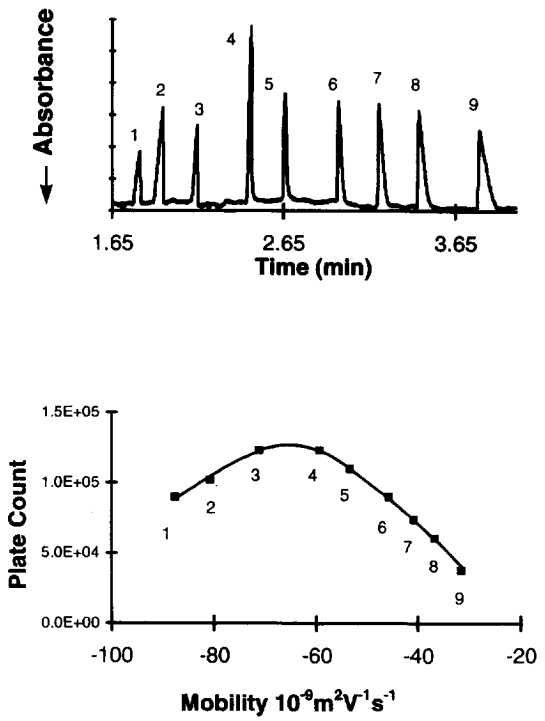

Fig. 6. Variation of the number of theoretical plates calculated for each peak in the electropherogram shown. The electropherogram was obtained using 10 m*M* phthalate, 40 m*M* diethanolamine and 0.5 m*M* cetyltrimethylammonium hydroxide at pH 9.3 as electrolyte. The number of theoretical plates was calculated using the 5σ method. Peak identities: 1=chloride, 2=sulfate, 3=chlorate, 4=phosphate, 5=carbonate, 6=ethanesulfonate, 7=propanesulfonate, 8=butanesulfonate, 9=pentanesulfonate. Electropherogram courtesy of P. Doble.

electrolyte, the peak shape is distorted, leading to reduced values of *N*. This peak distortion is clearly evident in the electropherogram in Fig. 6. The wide variation in *N* over the electropherogram is in direct contrast to that observed for IC and leads to the peak capacity for CE being considerably less than one would expect by consideration of the maximum value of *N* alone. Choosing a time window of 5 min for a co-EOF system, the peak capacity is approximately 88.

5. Separation selectivity

The selectivity of a separatory system is reflected in the sequence in which analytes emerge from the system. In the case of IC, the observed selectivity results from a complex interplay of interactions between the analyte, the stationary phase and the eluent. This interplay is governed by characteristics such as the charge, polarisability, size and hydrophobicity of the analyte, and the pH and nature of competing ion in the eluent. The selectivity effects exerted by the stationary phase are derived predominantly from the nature of the (usually polymeric) substrate and the type of functional group. Many stationary phases for IC having differing selectivities have been developed and there are more than 150 stationary phases available commercially. Eluent parameters also exert major effects on selectivity and a review of the literature shows that more than 2000 eluents comprising different combinations of competing ions, pH, organic modifiers, etc., have been employed. The outcome of such a variety of stationary phases and eluents means that a wide range of separation selectivities is possible in IC and manipulation of selectivity can be accomplished using effects deriving from numerous experimental parameters.

Separation selectivity in CE is governed by the electrophoretic mobilities of the analytes, which in turn are dependent predominantly on the zeta potentials of the analyte ions. Manipulation of zeta potential can be achieved by variation of the size or charge of the analyte, generally by altering electrolyte parameters such as pH, addition of complexing agents, organic modifier content, addition of surfactants, etc.. Unlike IC, the selectivity of CE arises essentially from the analyte and the electrolyte, with very little influence being exerted by the capillary itself. CE therefore lacks the major influence on selectivity which is exerted in IC by the stationary phase, so that manipulation of selectivity becomes limited to changes in analyte zeta potential which can be created by variation of the composition of the electrolyte. It is therefore much more difficult to manipulate selectivity in CE than in IC [20,21].

It is instructive to examine the separation selectivities achieved by IC and CE under standard conditions, as revealed by the elution or migration order of common analytes. In the case of IC, anions are eluted in the following order of retention times: $F^- < Cl^- < NO_2^- < Br^- < NO_3^- < PO_4^{3-} < SO_4^{2-} < I^-$. However, the migration times of these species in

co-EOF CE are $Br^- < Cl^- < SO_4^{2-} < NO_2^- < I^- < NO_3^- < F^- < PO_4^{3-}$ and in counter-EOF CE are $F^- < PO_4^{3-} < NO_3^- < I^- < NO_2^- < SO_4^{2-} < Cl^- < Br^-$. These sequences show that the selectivities of each of the three techniques are quite different, suggesting that these separatory methods provide complementary selectivities which can be used to advantage in solving analytical problems. Thus, separation of fluoride from a difficult matrix is likely to be problematic in IC and counter-EOF CE because of the early elution/migration of this species, but should be possible using co-EOF CE. The reverse situation would be expected to apply for bromide. A similar pattern emerges when the same comparison is made for inorganic cations for which the IC retention times follow the order $Li^+ < Na^+ < NH_4^+ < K^+ < Mg^{2+} < Ca^{2+} < Sr^{2+} < Ba^{2+}$, whilst migration times for co-EOF CE are $NH_4^+ < K^+ < Ba^{2+} < Sr^{2+} < Ca^{2+} < Na^+ < Mg^{2+} < Li^+$ and for counter-EOF CE are $Li^+ < Mg^{2+} < Na^+ < Ca^{2+} < Sr^{2+} < Ba^{2+} < K^+ < NH_4^+$. Complementary selectivities are again apparent and one would expect co-EOF CE to be best for analytes like lithium and sodium in complex matrices, but IC or counter-EOF CE to be better for analytes like potassium.

6. Analytical performance parameters

A simple comparison of two analytical techniques in terms of their analytical performance parameters is fraught with danger because of the many variables which must be considered. However, if one confines the discussion to the mainstream usage of IC and CE as specified in Section 2, a meaningful comparison can be made. Table 1 summarises the detection limits and precision of suppressed IC with conductimetric detection and co-EOF CE with indirect spectrophotometric detection. It can be seen that under routine conditions, IC is considerably more sensitive than CE, but the techniques display similar sensitivities if preconcentration (for IC) or electromigration injection (for CE) are employed. It should be noted that on-line sample preconcentration methods for IC require additional hardware in the form of at least one switching valve and sometimes also an additional pump, whereas electromigration injection in CE can be performed with the standard hardware. On the other hand, preconcentration in IC usually also results in the achievement of some selective sample clean-up which can permit it to be applied to more complex sample matrices than is the case for electromigration injection in CE.

The precision of IC and CE in terms of elution/migration times and peak areas merits comment. As shown in Table 1, relative standard deviations (R.S.D.s) of 1% or less are routinely achievable in IC, but precision is somewhat worse for CE. Two factors contribute to this. The first is that the migration time of an analyte in CE is governed not only by the electrophoretic mobility of the analyte but also by the mobility of the electroosmotic flow (EOF). The EOF arises from the zeta potential on the capillary wall which in turn is dependent on the composition of the electrolyte. A stable EOF (and hence stable migration times) results only when the capillary wall reaches equilibrium with the electrolyte and its zeta potential is stabilised. Such equilibration may take some time, especially if a cationic surfactant is used as an electrolyte additive to coat the wall to reverse the EOF. Even after equilibrium has been reached, injection of a sample may alter the equilibrium if the sample contains components which may adsorb to the wall. The second factor, which leads to reduced precision of peak area in CE, is the small magnitude of the detection signal resulting from the short light path

Table 1
Some analytical performance parameters for IC and CE

Parameter	IC[a]	CE[b]
Detection limit (direct sample injection)	10 ppb, 50 μl	200 ppb, 30 s
Detection limit (sample preconcentration)	<1 ppb	<1 ppb
Precision	1% R.S.D.	3–5% R.S.D.

[a] Suppressed IC system, conductivity detection.
[b] Indirect UV detection.

used in spectrophotometric detection. Signal-to-noise ratios are therefore smaller in CE than in IC for similar analyte concentrations, leading to the possibility for increased error in measurement of peak areas.

7. Method development

In developing an IC method for the determination of particular analytes in a specified sample matrix, a number of key decisions must be made. These include the type of separation procedure (ion exchange, ion interaction, ion exclusion, etc.), the particular column providing the desired separation selectivity, and which of the numerous detection methods is most appropriate. Literature resources in IC are quite extensive (in excess of 5000 papers have been published since 1975) and detailed reviews, tabulations and electronic databases are available to assist in selection of the most important parameters. The task of method development in IC therefore is normally reduced to fine-tuning of the eluent composition in order to achieve the desired separation, generally using a trial-and-error process.

The technique of IC is sufficiently well developed that quite extensive research has been performed on computer-assisted method development. This research has taken two forms, the first of which is the use of expert systems to configure an IC method (that is, to select the separation method, the column, eluent type and detector) [22], whilst the second is the use of an optimisation algorithm to formulate the best eluent composition [23]. The latter approach is underpinned by a considerable volume of research on retention mechanisms and mathematical retention models [24,25], which enables the optimisation to be performed using a limited amount of experimentation. Conceptually, the stages of literature searching, expert systems and optimisation can be combined into a single computer package, as represented schematically in Fig. 7. Here, the computer first consults an electronic database and seeks a suitable method. If identified, such a method can be either used exactly as presented in the literature or subjected to further optimisation. If no prior method is available, an expert system configures the separation method, column, eluent type and detector and the final eluent composition is then determined using the optimisation algorithm. Development of such a computer package is in progress.

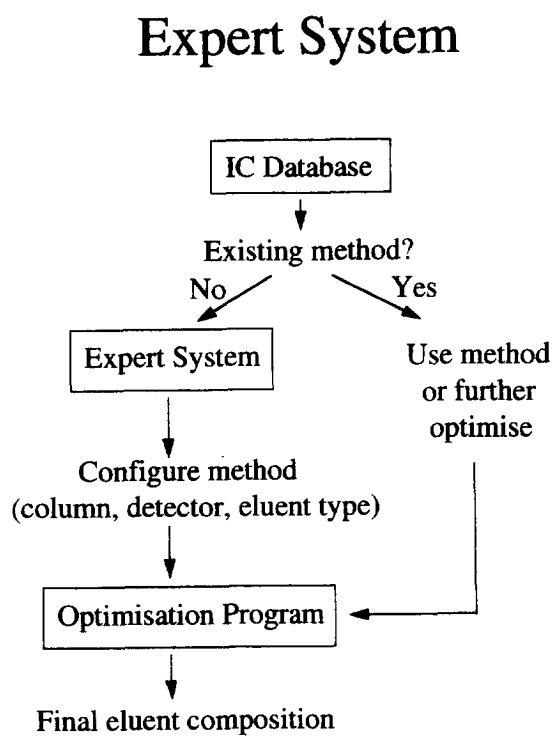

Fig. 7. Schematic representation of a computer package for ion chromatography which enables systematic literature searching of a database, use of an expert system to configure a method, and use of an optimisation program to fine-tune the composition of the eluent.

Method development in CE is a more difficult exercise than in IC because of the limited amount of publications available. In formulating the parameters of a CE method, decisions must be made regarding the required magnitude and direction of the EOF, and the nature of the electrolyte composition (e.g., the electrolyte ion, UV-absorbing probe ion, surfactant and ligand). The range of electrolytes is currently limited to a few standard systems and method development is often restricted to trial-and-error manipulation of these systems. However, it has already been pointed out that the opportunities are limited for control of separation selectivity in CE, so adaptation of an existing electrolyte system to a particular analytical problem may not always be successful. Computer-assisted procedures for method

development in CE are not yet available, but some research in this area is being undertaken [26–28] and further progress can be expected in the near future.

8. Applications

IC has been applied to a much wider range of samples than has CE, so a direct comparison of these application areas will simply show that IC enjoys a higher level of routine usage than CE. However, it is interesting to look at the rate of growth of new applications for each technique and the types of samples to which they are applied. Table 2 shows the numbers of IC and CE applications papers published in the Proceedings of the International Ion Chromatography Symposia (IICS) series for the years 1990–1995 [29–34], with the corresponding numbers of papers dealing with fundamentals being shown in parentheses. Both IC and CE applications have remained at fairly constant levels since 1992 and there is little evidence from this source of either a decline in IC applications (which might be expected for a method if it was entering its senescent phase) or an increase in CE applications (which might be expected for a method if it was emerging as the potential replacement for IC). Even after acknowledging that the data in Table 2 are limited in scope, it should be recognised that the IICS meetings are the prime annual forum for presentation of results in this field and can therefore be considered to provide a useful overview of publication trends.

Closer examination of the types of CE applications papers reveals an interesting trend. These papers commonly address a sample or analyte type which is difficult to analyse using IC. Samples of high pH or high ionic strength, or those which are limited in sample volume, fall into this category. Thus, CE has been applied to the determination of oxalate and fluoride in Bayer liquor [21], anions in pulp and paper samples [35], ions in atmospheric aerosols [36], analysis of single raindrops [37] and sulfide in leather treatment liquors [38], all of which can be considered to complement the types of analyses routinely performed by IC.

Table 2
Application papers published in the Proceedings of the International Ion Chromatography Symposia series for 1990–1995

Year	IC	CE
1990	22 (16)	2 (3)
1991	20 (9)	4 (2)
1992	29 (16)	6 (6)
1993	28 (22)	4 (6)
1994	40 (18)	5 (6)
1995	30 (13)	4 (5)

The number of papers dealing with fundamentals of the techniques is shown in parentheses.

9. Relative strengths and weaknesses

The preceding discussion has indicated some of the relative strengths and weaknesses of IC and CE and these are summarised in Table 3. It can be seen that the strengths of IC lie in the highly developed nature of the technique, its broad applications base and the relative ease with which it can be applied to demanding analyses. Its weaknesses relate to considerations of speed, separation efficiency and costs of consumables, which are the same areas in which the strengths of CE lie. Similarly, the weaknesses of CE are generally in the aspects where IC is strong. Table 3 demonstrates clearly the complementarity of the two techniques, which was also evident in the earlier discussion of separation selectivity and applications.

10. Future directions

Prediction of the future directions of an analytical technique is a challenging task, but some insight can be gained by close examination of the recent literature. This suggests that future developments in IC might include further advances in sample handling to extend the application of IC to more complex samples, the development of new stationary phases offering different separation selectivities to those available currently, and the increased usage of IC in hyphenated techniques. Miniaturisation and increased portability of IC instrumentation will occur and it is possible that research in building analytical instruments on microchips will include work on IC. Finally, further development of expert systems and computer-assisted optimisation packages for IC can

Table 3
Strengths and weaknesses of IC and CE

Technique	Strengths	Weaknesses
IC	Broad range of applications	Moderate speed
	Well-developed hardware	Moderate separation efficiency
	Many detection options	Intolerance to some sample matrices (e.g., high ionic strength)
	Reliability (good accuracy, precision)	High cost of consumables
	Accepted as standard methodology	
	Manipulation of separation selectivity is simple	
	High sensitivity	
CE	High speed	Instability and irreproducibility of migration times and peak areas
	High separation efficiency	Moderate sensitivity
	Good tolerance to sample matrices (especially high pH)	Manipulation of separation selectivity is difficult
	Low cost of consumables	Detection options are limited
		Routine applications are limited

be expected, leading to commercial availability of such software.

Future directions in CE will address the weaknesses listed in Table 3. Stability and ruggedness of the technique, especially for anions, will be improved through the use of stable, bonded-phase capillaries. Detection options will increase, particularly in the areas of electrochemical detection (both amperometry and potentiometry), suppressed conductivity detection and post-column reaction detection. The hardware technology can be expected to improve and the range of applications will increase as more scientists adopt CE as a routine tool for inorganic analysis. Microchip technology for CE is already a reality and is likely to be examined as a tool for inorganic analysis. Finally, capillary electrochromatography, which can be considered to be a hybrid of IC and CE and which combines the advantages of both techniques, can be expected to be applied to the ions of interest.

11. Conclusions

IC is a mature analytical technique supported by well-developed hardware, a strong user base and a broad range of published applications. The emphasis of research in IC is on applications rather than fundamentals, although there continues to be substantial progress in the latter area. CE of inorganic ions can be considered to be an emerging technique in which current research carries an emphasis on fundamentals rather than applications. However, rapid development of CE can be expected.

Comparison of IC and CE in most of the aspects considered in this paper has shown that the two techniques can be considered to be complementary rather than competitive. This is particularly true in the area of separation selectivity, where it is readily apparent that a separation which is problematic in one technique should often be relatively straightforward in the other. The question of whether CE will ultimately replace IC therefore becomes less important since there are clear possibilities for the two techniques to co-exist and to together offer to the analytical chemist the means to solve most practical problems in the determination of inorganic anions and cations.

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