

Studies on eluents suitable for use with simultaneous conductivity and direct UV detection in non-suppressed ion chromatography

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ABSTRACT

Simultaneous conductivity and direct UV detection is frequently employed in ion chromatography for the analysis of complex matrices or when samples contain disparate levels of anions. A number of eluents suitable for use with both detection modes were evaluated in terms of their general utility as anion screening eluents for non-suppressed ion chromatography. Octanesulphonate–borate was perhaps the most versatile of the eluents investigated as it had good separation selectivity, gave no system peaks and chloride response could be eliminated when using direct UV detection. Both borate–gluconate and carbonate–hydrogencarbonate also proved to be very useful screening eluents for use with direct UV and non-suppressed and suppressed conductivity detection respectively. Hydroxyde and tartrate–borate were of less utility as general purpose eluents, however both have unique characteristics which make them ideal for selected applications.

INTRODUCTION

Conductivity is the most commonly used detection mode in ion chromatography (IC); however, the tandem combination of conductivity and direct UV detection is frequently used for the analysis of complex matrices or when samples contain disparate levels of anions [1]. Many of the eluents commonly employed with conductivity detection, such as carbonate–hydrogencarbonate [2,3] or borate–gluconate [4,5], are also applicable for use with direct UV detection. Other species which are suitable for use as eluents with simultaneous conductivity and direct UV detection include alkylsulfonates, such as methane- [6], chloromethane [7] and octanesulfonate [8] and UV-transparent inorganic anions, such as hydroxide [9] and phosphate [10]. Non-suppressed IC offers an advantage over suppressed IC

in terms of the wide range of eluents that are applicable with the technique; however, in many cases it is not necessarily clear which eluent is most appropriate for a particular application. This can frequently lead to the chromatographer having to “try” a number of eluents in order to achieve suitable results.

In this paper, several eluents which can be used with simultaneous conductivity and direct UV detection were evaluated with a view toward making the selection of an eluent for a particular application more straightforward. The eluents studied were the commonly used borate–gluconate and carbonate–hydrogencarbonate; hydroxide, tartrate–borate, which gives similar elution characteristics to borate/gluconate eluent [11]; and also an octanesulfonate–borate eluent. The elution characteristics and detection properties of these eluents are discussed and examples of optimal practical applications of each eluent are presented.

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EXPERIMENTAL

Instrumentation

The liquid chromatograph consisted of a Waters Chromatography Division of Millipore (Milford, MA, USA) Model 510 pump, either a U6K injector or WISP 712 autoinjector, Model 431 conductivity detector, Model 486 UV detector and either an 820 Maxima data station or Model 730 data module. A Waters reagent delivery module (RDM) was added to the system for solid-phase reagent (SPR) conductivity detection. Three methacrylate-based, anion-exchange analytical columns from the Waters IC-Pak Anion range were used; an IC-Pak Anion (50 × 4.6 mm I.D.), an IC-Pak Anion HC (150 × 4.6 mm I.D.) and an IC-Pak Anion HR (75 × 4.6 mm I.D.) column. UV-absorbance spectra were recorded on a Varian DMS 100 scanning spectrophotometer (Palo Alto, CA, USA).

Eluents

The five eluents used in the preliminary evaluations were initially selected to give a similar separation of a mixture of carbonate, chloride, nitrite, nitrate, phosphate and sulfate. The eluents were: borate–gluconate (1.3 mM tetraborate, 5.8 mM boric acid, 1.3 mM gluconate, 5 g/l glycerin, 120 ml/l acetonitrile, 20 ml/l *n*-butanol, pH 8.5), carbonate–hydrogencarbonate (1.2 mM carbonate, 1.2 mM hydrogencarbonate, pH 10.0), hydroxide (6.0 mM hydroxide, pH 11.7), tartrate–borate (3.0 mM tartaric acid, 0.4 M boric acid, adjusted to pH 4.5 with lithium hydroxide) and octanesulfonate–borate (3.0 mM octanesulfonate, 5 mM boric acid, adjusted to pH 8.5 with lithium hydroxide). Eluents were prepared daily, filtered and degassed with a Waters solvent clarification kit.

Reagents

Water purified (18 M Ω) using a Millipore Milli-Q water purification system (Bedford, MA, USA) was used for all solutions. Sodium tetraborate, lithium hydroxide and boric acid (all analytical-reagent grade) and glycerin and tartaric acid (both laboratory-reagent grade) were obtained from Ajax Chemicals (Sydney, Australia), as were the analytical-grade sodium salts used for the preparation of all the anion standards. Sodium gluconate (laboratory-reagent grade) was obtained from Fluka

(Buchs, Switzerland). Sodium octanesulfonate was obtained from BDH (Poole, UK). Acetonitrile and *n*-butanol (both HPLC grade) were obtained from Waters.

RESULTS AND DISCUSSION

Preliminary investigations

In considering the requirements of an eluent to be suitable for general purpose use with dual conductivity and direct UV detection, three important criteria emerge. These are sensitivity, selectivity and eluent pH and buffering capacity. The eluent chosen by the chromatographer must firstly permit sensitive detection, *i.e.* have a large difference between the equivalent conductances of the solute and eluent anions in the case of non-suppressed conductivity detection [12] and be (essentially) transparent at the detection wavelength in the case of direct UV detection. An eluent must then have appropriate selectivity for a particular application with the column of choice. Finally, the eluent should be able to be operated at a suitable pH to allow the determination of common weak acid anions, such as phosphate, and also possess sufficient buffering capacity to allow samples with pH values appreciably different to the eluent to be successfully chromatographed.

The eluents chosen for this study include three commonly used IC eluents, namely borate–gluconate, carbonate–hydrogencarbonate and hydroxide as well as the less commonly used tartrate–borate [11] and a novel eluent, octanesulfonate–borate. All eluents permit relatively sensitive detection with both non-suppressed conductivity and direct UV detection, with the exception of the combination of carbonate–hydrogencarbonate eluent and conductivity detection. While this eluent is typically used with suppressed conductivity detection [2], for the purposes of comparison, it was used in the non-suppressed mode in the preliminary investigations. The five eluents were initially selected to give a separation of a mixture of carbonate, chloride, nitrite, nitrate, phosphate and sulfate within a reasonable elution time in order to evaluate the selectivity of the eluents. The UV spectrum of each of the eluents was then measured, along with the spectrum of a solution of 100 ppm chloride, as dual conductivity–direct UV detection is frequently employed when

samples contain high levels of chloride. The final stage of the preliminary investigations was to use each eluent for an application for which dual conductivity-direct UV detection IC would be typically employed, the determination of anions in a sewage plant effluent to bay discharge sample.

Fig. 1 shows the UV spectra in the range 190–256 nm for each of the five eluents described in the Experimental section, along with the spectra of a solution of 1000 ppm chloride. These results indicate that octanesulfonate–borate should probably provide the most sensitive detection of UV-absorbing anions, such as the commonly analyzed nitrite and nitrate, in the 200–220 nm region where these anion show appreciable absorbance. Carbonate–hydrogencarbonate, hydroxide and tartrate–borate all

appear as though they will permit similar detection sensitivity in the 200–220 nm range, although the hydroxide spectrum was probably effected by the adsorption of carbon dioxide into the solution. The UV spectra also indicate that it should be possible to eliminate any UV response for chloride by appropriate wavelength selection when using an octanesulfonate–borate eluent.

Fig. 2 shows chromatograms of a standard mixture containing 10 ppm chloride, 20 ppm nitrite, 20 ppm nitrate, 30 ppm phosphate and 20 ppm sulfate using each of the five eluents described in the Experimental and an IC-Pak Anion HC column with conductivity and direct UV absorption detection at 214 nm. A review of the chromatograms in Fig. 2 suggests that each of the different eluents has a number

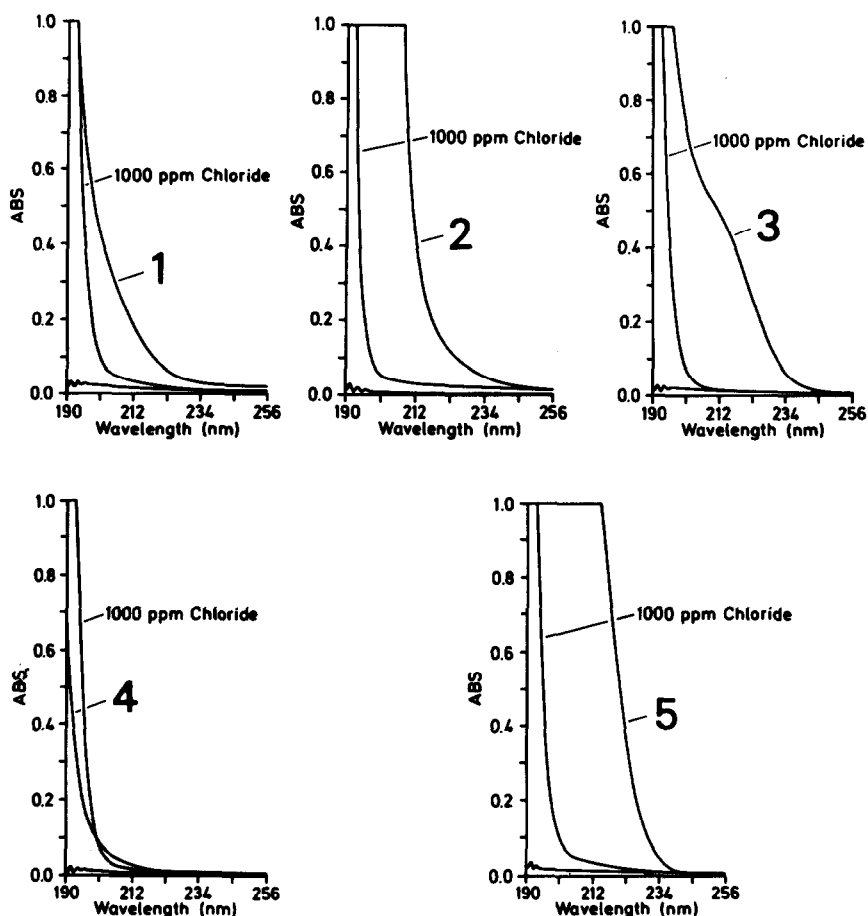


Fig. 1. UV spectra in the range 190–256 nm for the five eluents and 1000 ppm chloride. Eluents (see Experimental): 1 = borate–gluconate; 2 = hydroxide; 3 = tartrate–borate; 4 = octanesulfonate–borate; 5 = carbonate–hydrogencarbonate.

of unique characteristics: (a) Borate–gluconate gave the best overall selectivity of the five eluents for the standard mixture with the methacrylate-based anion exchanger. Hydrogencarbonate and chloride, and to a lesser extent phosphate and sulfate gave negative peaks when using direct UV detection and also the UV response for nitrate was significantly greater than for nitrite with this eluent. (b) Carbonate–hydrogencarbonate was a poor eluent for non-suppressed conductivity detection, as was expected and system peaks [13,14] were observed with both detection modes. Also, phosphate did not elute under these conditions, however this eluent gave excellent sensitivity for nitrite and nitrate with UV detection at 214 nm. (c) Phosphate also did not elute when using an hydroxide eluent and carbonate co-eluted with sulfate under most eluent conditions that allowed a reasonable total run time with the IC-Pak Anion HC column. Good response was obtained for nitrite and nitrate; and as was the case

with the other two eluents above, chloride appeared as a negative peak with UV detection at 214 nm. (d) Tartrate–borate gave the least sensitive response for nitrate when using direct UV detection and was the only eluent where the UV response for nitrite was significantly greater than for nitrate. The phosphate peak gave a negative response with conductivity detection and no carbonate peak appeared at this low eluent pH. Also, nitrate eluted later than sulfate under these eluent conditions. (e) Octanesulfonate–borate gave reasonable selectivity for the anions of interest, but most significantly, it gave good UV response for nitrite and nitrate and virtually no response for the other anions at 214 nm. All the eluents, with the exception of carbonate–hydrogencarbonate, gave similar conductivity response for the anions of interest, although hydroxide (in the indirect mode) gave slightly better sensitivity with conductivity detection than the other eluents.

The five eluents were then used for an application

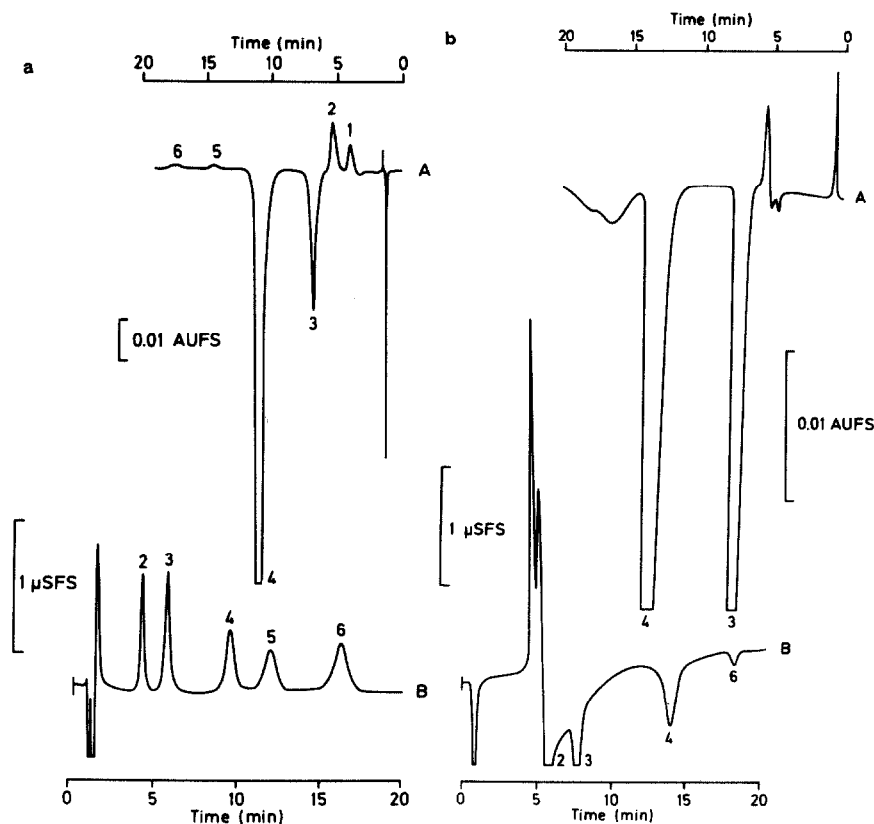


Fig. 2.

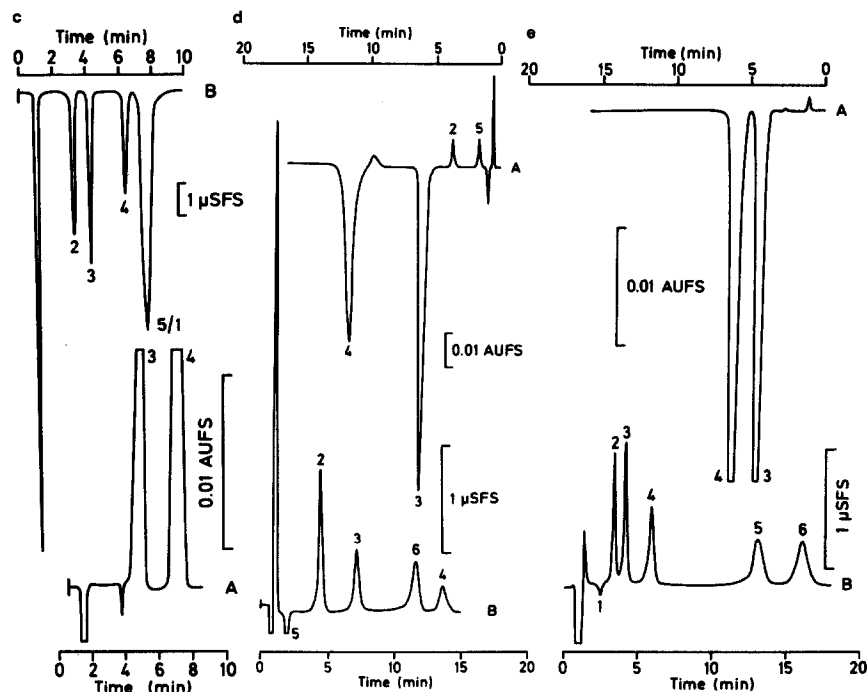


Fig. 2. Chromatograms of a standard anion mixture for the five eluents with conductivity and direct UV absorption detection at 214 nm. Conditions: eluents (details as in Experimental), (a) borate–gluconate; (b) carbonate–hydrogencarbonate; (c) hydroxide; (d) tartrate–borate; (e) octanesulfonate–borate; column, Waters IC-Pak Anion HC; flow-rate, 2.0 ml/min; injection volume, 25 μ l; detection, A = UV at 214 nm, 0.05 AUFS and B = conductivity, 5 μ SFS. Solutes: 1 = carbonate; 2 = chloride (10 ppm); 3 = nitrite (20 ppm); 4 = nitrate (20 ppm); 5 = phosphate (30 ppm); 6 = sulfate (20 ppm).

for which dual conductivity–direct UV detection would be typically employed, *i.e.* the analysis of anions in a sewage plant effluent to bay discharge sample using the same conditions as described for the standard chromatograms above. The use of dual detectors was important in this application as it allowed the quantitation of nitrite (and nitrate to a lesser extent) by UV at 214 nm and the remaining anions in the sample by conductivity. As would be expected, the same characteristics for each of the eluents as described in the previous paragraph were evident. Fig. 3 shows an example of a typical chromatogram of the discharge sample obtained using an octanesulfonate–borate eluent and dual conductivity and direct UV absorption detection. The higher level of chloride present in the sample (compared to the standards shown in Fig. 2) resulted in a negative chloride peak which interfered with the determination of nitrite by direct UV at 214 nm when using an hydroxide eluent. Also, a negative sulfate

peak interfered with the determination of nitrate by direct UV at 214 nm in the sample when using a tartrate–borate eluent. The borate–gluconate and octanesulfonate–borate eluents were the most suitable eluents for this analysis as they allowed the determination of all the anions of interest in the sample, *i.e.* chloride, nitrite, nitrate, phosphate and sulfate with no interferences for any peak using either detection mode.

Optimal applications of each eluent

The preliminary investigations above indicated that each eluent had both useful and deleterious features which would influence whether it would be suitable for a particular application. Table I lists the advantages and disadvantages of each of the eluents and Figs. 4–8 show examples of optimal applications for each of the five eluents. These examples were selected to highlight the merits of each of the eluents. Fig. 4 shows a chromatogram of a sanita-

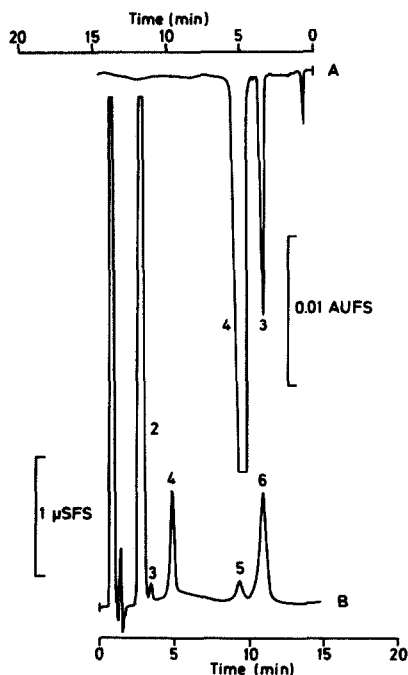


Fig. 3. Chromatogram of anions in a sewage plant effluent to bay discharge sample using octanesulfonate–borate eluent and dual conductivity and direct UV absorption detection at 214 nm. Conditions as for Fig. 2. Solutes: 1 = carbonate; 2 = chloride; 3 = nitrite; 4 = nitrate; 5 = phosphate; 6 = sulfate.

tion effluent sample using borate–gluconate eluent with an IC-Pak Anion HC column and dual conductivity–direct UV detection. Borate–gluconate had the best overall selectivity (at least with methacrylate-based anion-exchangers) of any eluent and as such, was ideally suited to anion screening in samples such as effluents, drinking waters, wastewaters, etc. Fluoride can also be quantitated using these eluent conditions. Extreme pH samples and also samples high in calcium/magnesium may cause system peaks [15] and high levels of bicarbonate may interfere with chloride when using this eluent. As mentioned previously, carbonate–hydrogencarbonate was a poor eluent for use with non-suppressed conductivity detection, however it is the most versatile eluent when used with suppressed (post-column enhancement) conductivity detection [1]. Fig. 5 shows a chromatogram of an Antarctic ice melt sample using carbonate–hydrogencarbonate eluent with an IC-Pak Anion HR column and post-column “enhancement” SPR conductivity de-

tection [16]. This selectivity of this eluent was particularly useful for the analysis of samples containing alkylsulfonates in the presence of chloride and carboxylic acids [17], as demonstrated in Fig. 5. System peaks can be a problem with this eluent and excessive phosphate retention was a problem with many column and eluent concentration combinations. An additional advantage of carbonate–hydrogencarbonate eluents is that their use masks sample carbonate to a large degree, making this eluent particularly suited for the analysis of samples high in carbonate, *e.g.* alkaline fusion or trap samples.

Borate–gluconate and carbonate–hydrogencarbonate were both very versatile for anion screening applications and are the most widely used eluents with non-suppressed and suppressed conductivity detection respectively [1]. Both can be used with direct UV detection, although high levels of chloride can interfere with the determination of nitrite when using this detection mode. Hydroxide was by far the most “difficult” to use of the five eluents studied, hence was of less utility as a general purpose eluent. The high pH means that eluent protection with helium blanketing/sparging or the use of an Ascarite trap was necessary as carbon dioxide adsorption can lead to baseline drift, cycling and also retention time instability. However, this eluent did permit the most sensitive conductivity detection when operated in either the non-suppressed or suppressed mode and it also allowed very low detection limits for nitrite and nitrate when using direct UV detection at 214 nm. Undoubtedly, the greatest advantage of hydroxide is that the high eluent pH allows very weak acids ($pK_a > 7$) to be chromatographed using an anion-exchange column. This eluent is often used in conjunction with other detection modes, such as amperometry, for the analysis of weak acid anions, *e.g.* sulfide and cyanide [18]. Another important application of hydroxide eluents is for the analysis strongly acidic samples, such as acid preserved drinking waters. Fig. 6 shows a chromatogram of nitrite-N and nitrate-N (at low $\mu\text{g/l}$ levels) in a sulfuric acid preserved, chlorinated drinking water sample with an IC-Pak Anion column and direct UV detection at 214 nm.

Tartrate–borate was of limited utility as a general purpose eluent as phosphate eluted as a negative peak when using conductivity detection and could

TABLE I
SUMMARY OF THE ADVANTAGES AND DISADVANTAGES FOR THE FIVE ELUENTS

Eluent	Advantages	Disadvantages
Borate–gluconate	(1) Excellent overall selectivity (2) pH allows determination of most common anions (3) Good buffer	(1) Chloride may interfere with nitrite at high levels on UV detection (2) Carbonate may interfere with chloride at high levels on conductivity (3) System peaks may be a problem
Carbonate–hydrogencarbonate	(1) Reasonable selectivity, especially for alkylsulfonate and carboxylic acid mixtures (2) Good “suppressed” eluent (3) Good buffer (4) No sample carbonate peak	(1) Chloride may interfere with nitrite at high levels on UV detection (2) System peaks may be a problem (3) Phosphate retention can be excessive with some column/eluent combinations
Hydroxide	(1) High eluent pH allows for determination of weak acid anions, <i>i.e.</i> cyanide (2) Most sensitive eluent with conductivity detection (3) Good sensitivity with UV detection at 214 nm	(1) Chloride may interfere with nitrite at high levels on UV detection (2) Eluent must be helium sparged (3) Baseline drift/cycling a problem (4) Retention time stability a problem (5) CO ₃ /SO ₄ resolution can be a problem (6) No buffering capacity
Tartrate–borate	(1) Low eluent pH discriminates against organic acid retention (2) Good low-pH buffer (3) No sample carbonate peak	(1) Sulfate may interfere with nitrate at high levels on UV detection (2) Phosphate cannot be quantitated (3) System peaks may be a problem
Octanesulfonate–borate	(1) Reasonable overall selectivity (2) pH allows determination of most common anions (3) No chloride interference with UV detection at 214 nm	(1) Carbonate may interfere with chloride at high levels on conductivity

not be quantitated, however this eluent was particularly suited to the analysis of inorganic anions in samples containing high levels of organic acid anions. Divalent organic acid anions, *e.g.* tartrate, succinate, malate and oxalate, often co-elute with inorganic anions such nitrate and sulfate when using neutral to alkaline eluents. Similarly, short-chained carboxylic acids, such as formate and acetate, are typically weakly retained and can interfere with anions such as fluoride and chloride. These organic acids all eluted at the void volume with tartrate–borate as a result of the low affinity of these species for the column exchange sites combined with the fact that low eluent pH protonates the organic acids, further decreasing their retention. The presence of a high concentration of a weak eluting species such as boric acid also discriminates against the retention of organic acids, hence this eluent was applicable to the analysis of inorganic anions in

samples containing high levels of organic acids, such as Bayer liquors, soil and plant extracts. Fig. 7 shows a chromatogram of well resolved chloride and sulfate peaks in a very complex sample matrix, a diluted Bayer liquor (which typically contains up to 30 g/l total organic carbon present as various organic acids at a pH of *ca.* 14) using a tartrate–borate eluent with an IC-Pak Anion HC column and conductivity detection. Lowering of the eluent pH would further decrease the retention of sample organic acids and also increase the sensitivity of conductivity detection [19], however the run times also become much longer with decreasing eluent pH.

A variety of alkylsulfonates have been used previously with dual conductivity–direct UV detection in IC [6–8]; however, these eluents had no buffering capacity and the pH was typically not appropriate to permit phosphate analysis. The addition of a bo-

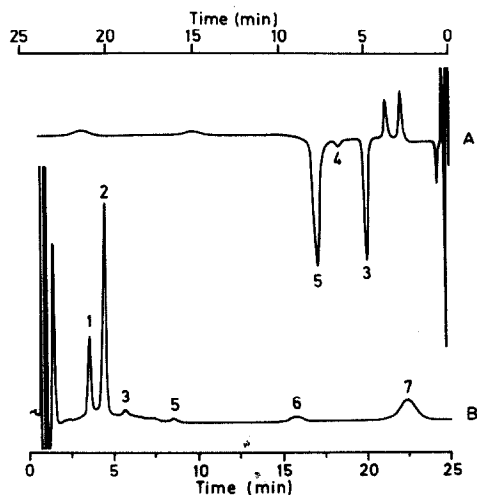


Fig. 4. Chromatogram of a sanitation effluent sample using borate-gluconate eluent and dual conductivity-direct UV detection. Conditions as for Fig. 2, except eluent, 1.3 mM tetraborate, 5.8 mM boric acid, 1.3 mM gluconate, 5 g/l glycerin, 120 ml/l acetonitrile, 20 ml/l *n*-butanol, pH 8.5; injection volume, 100 μ l; detection, A = UV at 214 nm and B = conductivity. Solutes: 1 = carbonate; 2 = chloride (10.5 ppm); 3 = nitrite (0.4 ppm); 4 = bromide (0.2 ppm); 5 = nitrate (0.5 ppm); 6 = phosphate (2.5 ppm); 7 = sulfate (7.3 ppm).

rate buffer to octanesulfonate creates a very versatile, anion-screening eluent for IC. Borate itself is a very weak eluent [11] and a concentration of approximately 50 mM was required to elute the

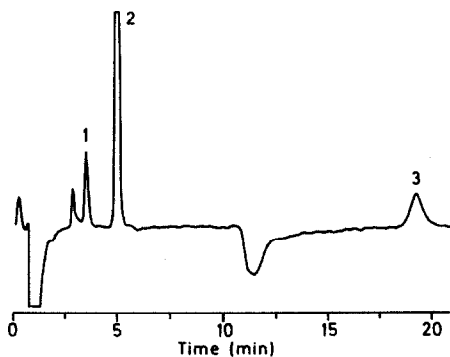


Fig. 5. Chromatogram of an Antarctic ice melt sample using carbonate-hydrogencarbonate eluent and SPR conductivity detection. Conditions as for Fig. 2, except column, Waters IC-Pak Anion HR; eluent, 1.2 mM carbonate, 1.2 mM hydrogencarbonate; flow-rate, 1.0 ml/min; injection volume, 100 μ l; detection, SPR conductivity. Solutes: 1 = methanesulfonate (0.07 ppm); 2 = chloride (0.12 ppm); 3 = sulfate (0.04 ppm).

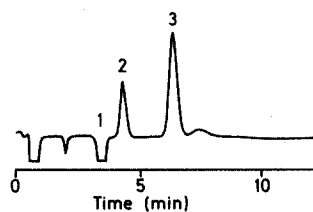


Fig. 6. Chromatogram of nitrite-N and nitrate-N in a sulfuric acid preserved, chlorinated drinking water sample using hydroxide eluent and direct UV detection at 214 nm. Conditions as for Fig. 2, except column, Waters IC-Pak Anion; eluent, 2.5 mM hydroxide; flow-rate, 1.2 ml/min; injection volume, 100 μ l; detection, UV at 214 nm. Solutes: 1 = chloride; 2 = nitrite-N (0.03 ppm); 3 = nitrate-N (0.07 ppm).

anions with approximately the same retention times as shown in Fig. 2e, hence it buffers the octanesulfonate eluent without significantly effecting its eluting strength. The utility of the octanesulfonate-borate eluent is demonstrated by the analysis of a relatively high chloride matrix, a grass filtration (preliminary treatment) sewage sample, as shown in Fig. 8. The chloride peak, present at approximately 500 ppm, did not interfere with the determination of nitrite, present at 0.5 ppm, by direct UV detection. This eluent has good overall selectivity for the common anions and the fact that chloride has no UV response allows the determination of nitrite by direct UV detection with no interference from as much as a 10 000-fold excess of chloride.

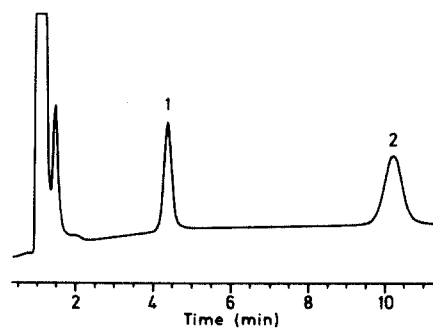


Fig. 7. Chromatogram of a Bayer liquor sample using tartrate-borate eluent and conductivity detection. Conditions as for Fig. 2, except eluent, 3.0 mM tartaric acid, 0.4 M boric acid adjusted to pH 4.5 with hydroxide; injection volume, 100 μ l; detection, conductivity; sample preparation, 1:500 dilution with water. Solutes: 1 = chloride (31 ppm); 2 = sulfate (43 ppm).

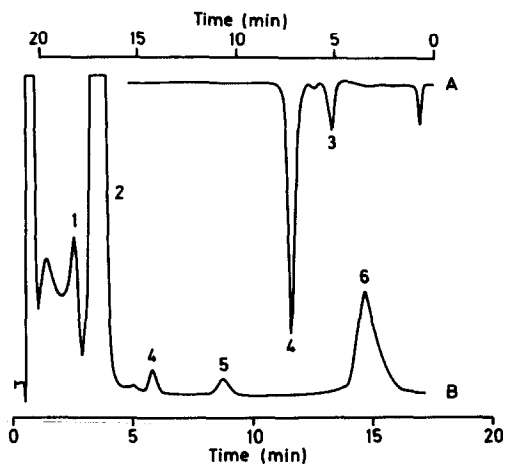


Fig. 8. Chromatogram of a grass filtration (preliminary treatment) sewage sample using octanesulfonate–borate eluent and dual conductivity–direct UV detection. Conditions as for Fig. 2, except eluent, 3.0 mM octanesulfonic acid, 5 mM boric acid adjusted to pH 8.5 with hydroxide; injection volume, 100 μ l; detection, A = UV at 210 nm and B = conductivity. Solutes: 1 = carbonate; 2 = chloride (523 ppm); 3 = nitrite (0.5 ppm); 4 = nitrate (3.1 ppm); 5 = phosphate (58 ppm); 6 = sulfate (124 ppm).

CONCLUSIONS

The use of dual conductivity and direct UV detection is a very versatile approach for the analysis of complex samples in IC. While no single eluent is appropriate for all applications, octanesulfonate–borate is perhaps the most versatile, general purpose anion screening eluent for non-suppressed IC. This eluent has good separation selectivity, gives no system peaks and chloride response can be eliminated when using direct UV detection. Both borate–gluconate and carbonate–hydrogencarbonate are also very good general purpose eluents for use with direct UV and non-suppressed and suppressed conductivity detection, respectively. The difficulties of using hydroxide eluents detract from their general utility, however the high pH makes it the only eluent suitable for the analysis of weak acid anions such as cyanide and silicate when using anion-ex-

change separations. The use of tartrate–borate eluents is restricted to analyses not requiring phosphate quantitation, however the eluent selectivity and low pH strongly discriminate against the retention of organic acids, making it ideal for the analysis of inorganic anions in samples containing high levels of organic acids.

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REFERENCES

- 1 P. R. Haddad and P. E. Jackson, *Ion Chromatography: Principles and Applications (Journal of Chromatography Library, Vol. 46)*, Elsevier, Amsterdam, 1990.
- 2 H. Small, T. S. Stevens and W. C. Bauman, *Anal. Chem.*, 47 (1975) 1801.
- 3 R. J. Williams, *Anal. Chem.*, 55 (1983) 851.
- 4 G. Schmuckler, A. L. Jagoe, J. E. Girard and P. E. Buell, *J. Chromatogr.*, 356 (1986) 413.
- 5 A. L. Heckenberg, W. R. Jones, W. J. Wildman, J. A. Krol and P. Alden, in P. Jandik and R. M. Cassidy (Editors), *Advances in Ion Chromatography*, Vol. I, Century International, Franklin, MA, 1990, p. 333.
- 6 J. P. Ivey, *J. Chromatogr.*, 267 (1983) 218.
- 7 P. E. Jackson, P. R. Haddad and S. Dilli, *J. Chromatogr.*, 295 (1984) 471.
- 8 *Ion Chromatography Laboratory Report No. 236*, Waters Chromatography Division of Millipore, Milford, MA, 1986.
- 9 T. Okada and T. Kuwamoto, *Anal. Chem.*, 55 (1983) 1001.
- 10 M. E. Miller and C. J. Capon, *Clin. Chem.*, 30 (1984) 781.
- 11 T. Okada and T. Kuwamoto, *J. Chromatogr.*, 403 (1987) 35.
- 12 D. T. Gjerde, J. S. Fritz and G. Schmuckler, *J. Chromatogr.*, 186 (1979) 509.
- 13 P. E. Jackson and P. R. Haddad, *J. Chromatogr.*, 346 (1985) 125.
- 14 W. Ishibashi, R. Kikuchi and K. Yamamoto, *Bunseki Kagaku*, 31 (1982) 207.
- 15 C. Erkelens; H. A. H. Billiet, L. de Galan and E. W. B. de Leer, *J. Chromatogr.*, 186 (1979) 509.
- 16 P. E. Jackson, P. Jandik, J. Li, J. Krol, G. Bondoux and D. T. Gjerde, *J. Chromatogr.*, 546 (1991) 189.
- 17 D. M. Davis and J. P. Ivey, *Anal. Chim. Acta*, 194 (1987) 275.
- 18 A. M. Bond, I. D. Heritage, G. G. Wallace and M. J. McCormick, *Anal. Chem.*, 54 (1983) 582.
- 19 J. S. Fritz, D. L. DuVal and R. E. Barron, *Anal. Chem.*, 56 (1984) 1177.