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## Enhancing the anion separations on a polydivinylbenzene-based anion stationary phase

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### Abstract

A new anion-exchange stationary phase is described for the separation of inorganic anions by ion chromatography. This stationary phase is made of polydivinylbenzene (P-DVB) based resin with dimethylethanolamine functional groups. The highly cross-linked P-DVB backbone makes this material chemically and mechanically stable under extreme chromatographic conditions. It withstands organic solvents and is stable at a wide range of pH, temperature and pressure. The separation of anions on this stationary phase can be enhanced by simple modification of the chromatographic conditions. The resolution and peak shape can be improved by adding organic additives to the eluent. These improvements can also be achieved by changing the column operating temperature. This stationary phase is useful for the separation of inorganic anions by suppressor-based and single-column ion chromatography methods.

*Keywords:* Stationary phases, LC; Mobile phase composition; Inorganic anions; Polydivinylbenzene

### 1. Introduction

Anion-exchange chromatography is the classical method for the separation of inorganic and small organic anions. Anion-exchange resins can be classified as either microporous or macroporous. Most of the classical ion-exchange resins are made of microporous substrates which are prone to strong shrinking and swelling due to its low degree of cross-linking [1]. Low cross-linkage also results in poor mechanical stability of the column. On the other hand, the macroporous substrates have high surface area and are highly cross-linked to produce remarkable mechanical stability. The material does not exhibit shrinkage and swelling when changing the polarity of the solvents. However, this material has the drawback of low mass transference due to

adsorptive effects on the high surface area resulting in poor column efficiency and poor ion-exchange kinetics [2].

A new type of anion-exchanger is described in this paper for the separation of anions by ion chromatography (IC) methods. This stationary phase is made of polydivinylbenzene (P-DVB) with dimethylethanolamine functional groups. The P-DVB stationary phase is developed for both single-column and suppressor-based IC applications. This resin has both micro- and macropores that are highly cross-linked to provide durability and high mass transference. The columns packed with the P-DVB anion-exchange material are stable at high temperature, wide range of pH, high pressure, and are compatible with a variety of organic solvents. The stability of the P-DVB stationary phase under these chromatographic conditions can be utilized to improve anion separation on this column. The objective of this report is to study the influence of some of these factors on the

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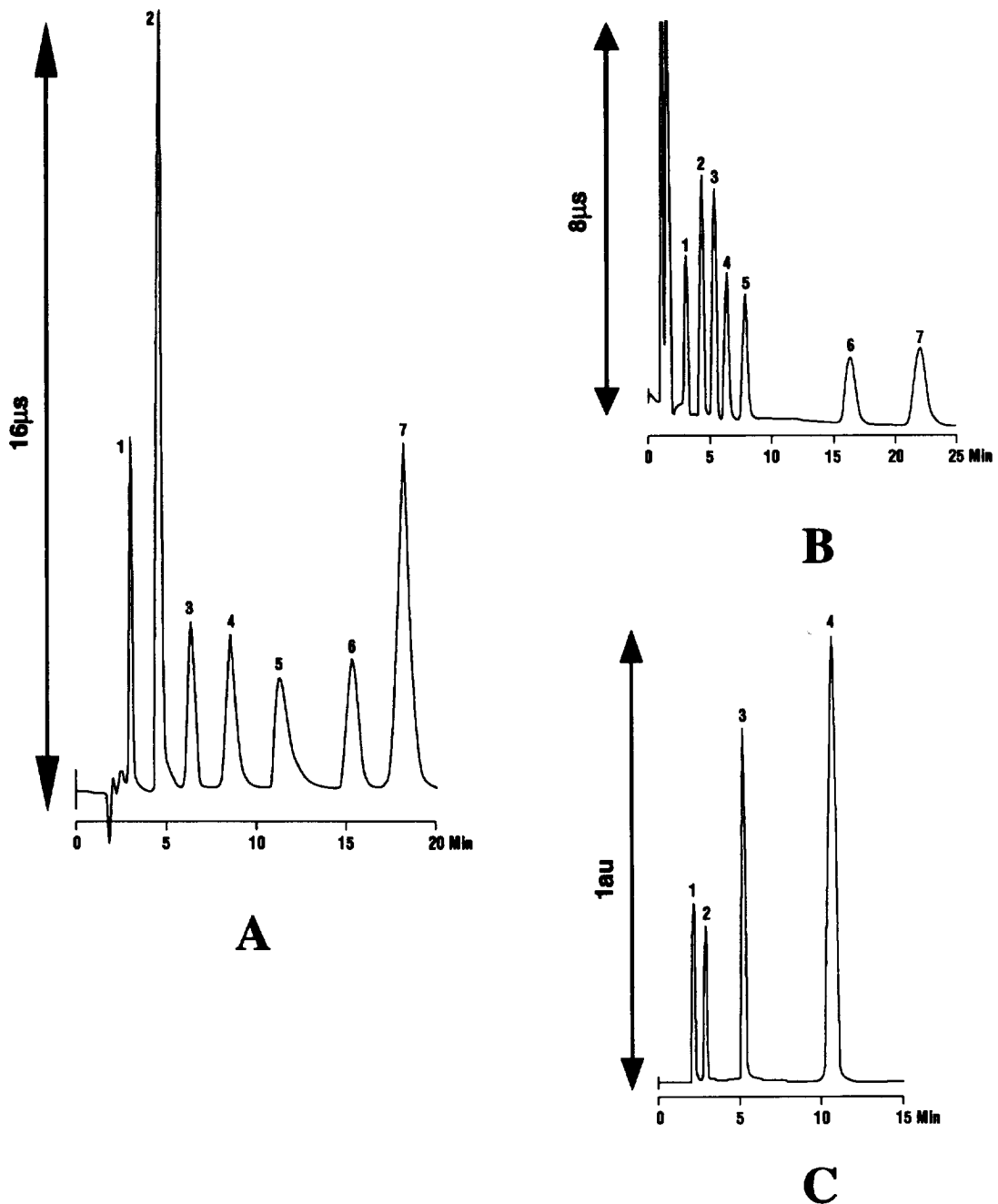


Fig. 1. (A) Anion separation by suppressor-based IC system. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate in 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity. Peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate (6 ppm); 7=sulfate (6 ppm). (B) Anion separation by single-column IC system. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 1.4 mM *p*-hydroxybenzoic acid in 5% methanol, pH 8.0 adjusted with lithium hydroxide, flow-rate: 1 ml/min, detector: conductivity. Peaks: 1=fluoride (5 ppm); 2=chloride (10 ppm); 3=nitrite (10 ppm); 4=bromide (10 ppm); 5=nitrate (10 ppm); 6=phosphate (15 ppm); 7=sulfate (15 ppm). (C) Anion separation by single-column IC system. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 10 mM sodium perchlorate in 10% methanol, flow-rate: 1 ml/min, detector: UV at 210 nm. Peaks: 1=iodate (50 ppm); 2=bromate (50 ppm); 3=thiosulfate (100 ppm); 4=thiocyanate (100 ppm).

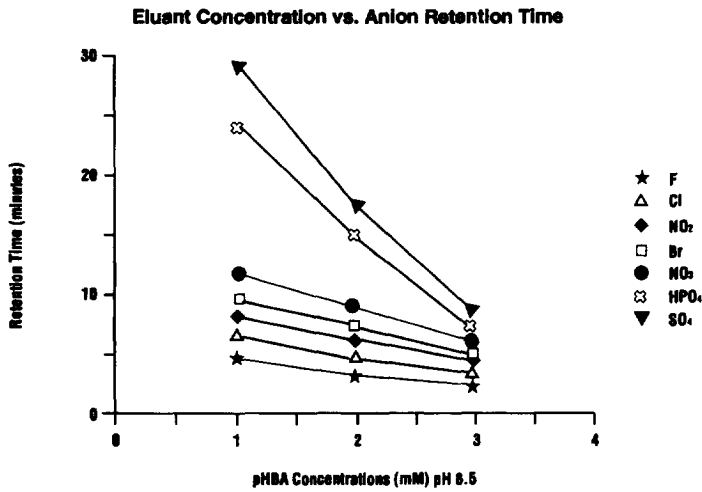


Fig. 2. Eluent concentration vs. anion retention time.

selectivity and peak shape of anions when the P-DVB stationary phase is employed.

## 2. Experimental

### 2.1. Chromatography system and columns

The Alltech Universal IC system (Alltech Associates, Deerfield, IL, USA) was used. It consists of the Model 526 metal-free HPLC pump, Model 350 conductivity detector, Model 335 suppressor module and the Alltech Model 570 autosampler. An HP

General Purpose DataStation (Hewlett-Packard, Wilmington, DE, USA) was used to collect and process data. The anion separation was performed on the Alltech Durasep A-1 (100 mm×4.6 mm) column, packed with P-DVB-based anion-exchange packing material.

### 2.2. Eluents and standards

All eluents and standards were prepared from analytical-reagent grade chemicals from Aldrich (Milwaukee, WI, USA) and deionized (18 M $\Omega$ ) water. A Barnstead Nanopure 11 system (Sybron

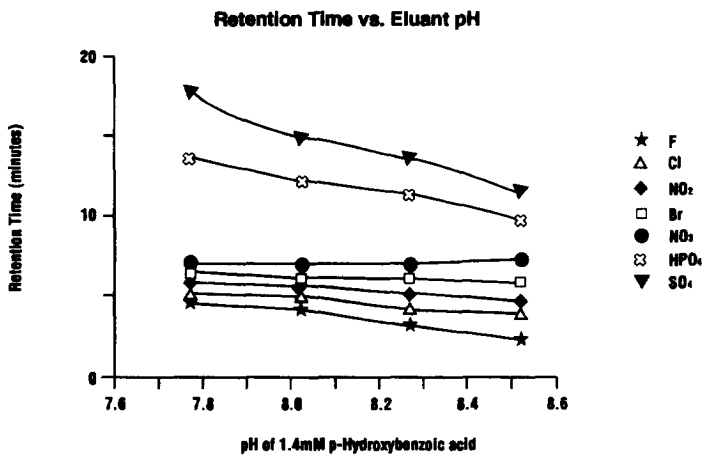


Fig. 3. Eluent pH vs. anion retention time.

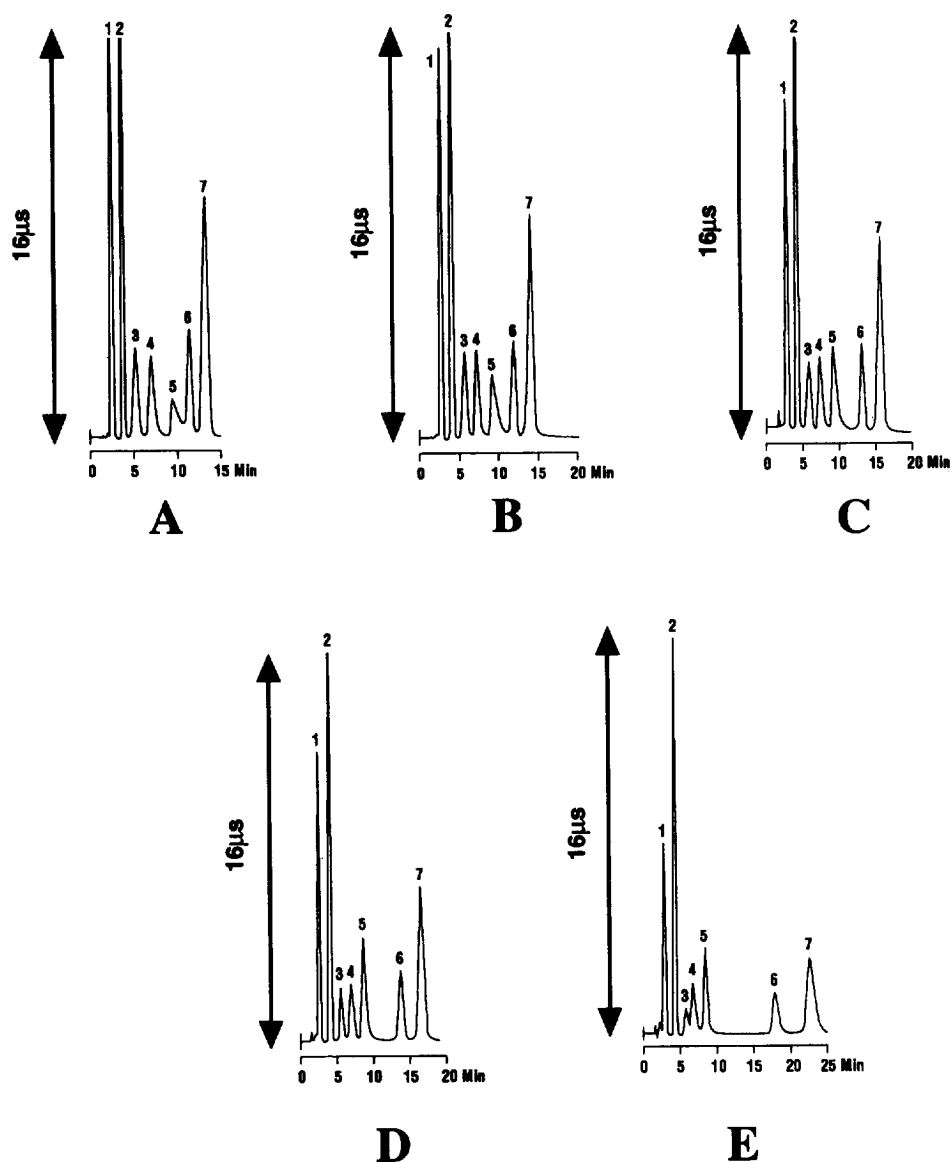


Fig. 4. Effect of methanol in bicarbonate-carbonate eluent. (A) 0% methanol. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 0% methanol flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate (6 ppm); 7=sulfate (6 ppm). (B) 5% methanol. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 5% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate (6 ppm); 7=sulfate (6 ppm). (C) 10% methanol. Column: Alltech Durasep A-1 column, 100×4.6 mm eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate (6 ppm); 7=sulfate (6 ppm). (D) 15% methanol. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 15% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate (6 ppm); 7=sulfate (6 ppm). (E) 20% Methanol. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 20% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate (6 ppm); 7=sulfate (6 ppm).

Barnstead, Boston, MA, USA) was used to produce the water for preparing eluents and samples. Stock solutions of 100 mM sodium bicarbonate, sodium carbonate, sodium hydroxide, *p*-hydroxybenzoic acid and sodium perchlorate were made, diluted with deionized water to make appropriate eluents.

### 2.3. Sample preparation

All water samples were filtered through 0.2  $\mu\text{m}$  Anotop IC (Alltech) syringe filters before injection. The silica sample was diluted 100 $\times$ , passed through Novo-Clean IC-H (Alltech) solid-phase extraction disk and filtered through a 2  $\mu\text{m}$  Anotop IC syringe filter. The Novo-Clean IC-H removed excess carbonate in the sample which would have co-eluted with the chloride in the sample.

### 3. Results and discussion

P-DVB resin is made of highly cross-linked polymer material that shows high mechanical stability. The columns packed with this material are stable at high temperature, high pressure, wide ranges of pH and exposure to organic solvents. The maximum operating temperature and pressure are 75°C and 6000 p.s.i (1 p.s.i.=6894.76 Pa), respectively. The P-DVB resin is functionalized with dimethylethanolamine to provide anion-exchange capabilities. The resulting material can separate inorganic anions using a variety of eluents by both suppressor-based and single-column IC methods. Among the eluents used in this study are sodium bicarbonate-carbonate, sodium hydroxide, *p*-hydroxybenzoic acid and sodium perchlorate. The P-DVB-based anion

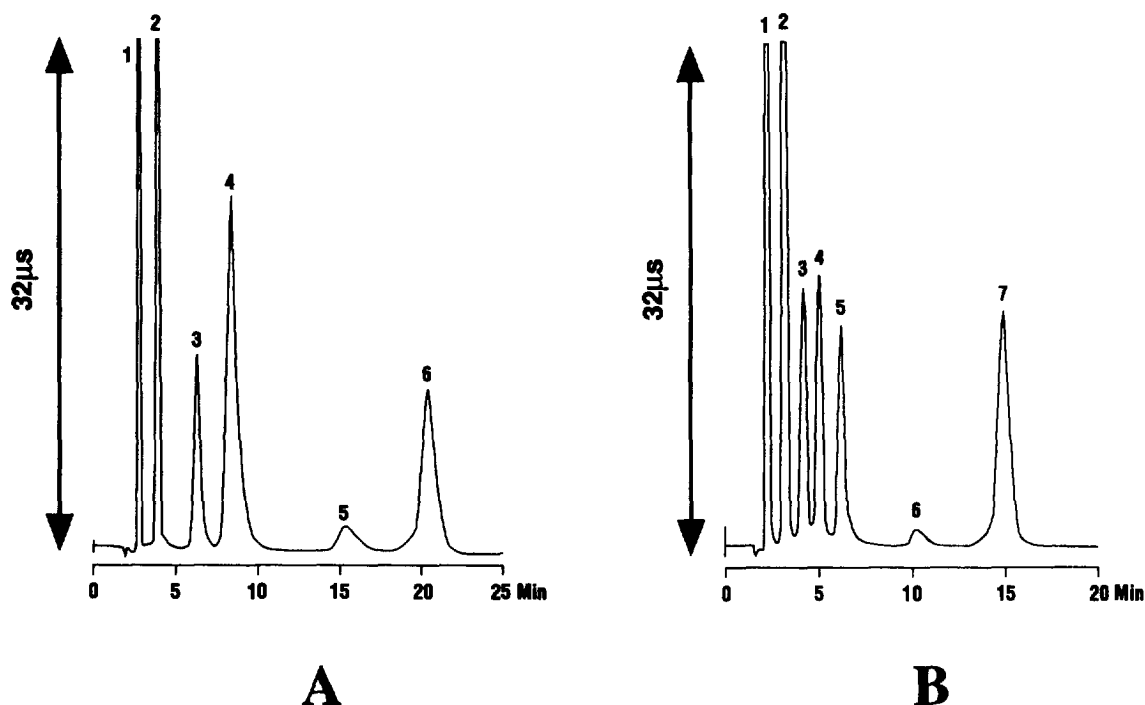


Fig. 5. Effect of methanol in sodium hydroxide eluent. (A) 0% methanol. Column: Alltech Durasep A-1 column, 100 $\times$ 4.6 mm, eluent: 2 mM sodium hydroxide, with 0% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm) and nitrate (4 ppm); 5=carbonate; 6=sulfate (6 ppm). (B) 10% methanol. Column: Alltech Durasep A-1 column, 100 $\times$ 4.6 mm, eluent: 2 mM sodium hydroxide, 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=carbonate; 7=sulfate (6 ppm).

columns are highly efficient, 100% organic compatible and stable at pH ranges from 1–14.

### 3.1. Separation of anions on the P-DVB anion-exchanger

The separation of seven common anions on the P-DVB-based anion column using a suppressor-based IC system is shown in Fig. 1A. An eluent containing 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate in 10% methanol was used for this application. The elution order is similar to the separation on other commercially available anion-exchange columns. Due to the low ion-exchange capacity of the packing, the eluent concentration required for the separation is very low. This feature has a great advantage when a solid-phase chemical suppressor (SPCS) [3] is used. The lower eluent concentration increases the suppressor cartridge lifetime, which is economically beneficial to the chro-

matographer. The average lifetime of the SPCS is approximately 16 h when this eluent is used.

Separation of anions by single-column IC methods using two different eluents are shown in Fig. 1B and C. *p*-Hydroxybenzoic acid eluent with 5% methanol, pH adjusted to 8.0, was used for Fig. 1B and 2 mM sodium perchlorate eluent was used for Fig. 1C. *p*-Hydroxybenzoic acid is one of the most common eluents used for anion analysis by single-column IC [4]. Sodium perchlorate eluent along with ultraviolet detection was used for the strongly-retained ionic species such as iodate, bromate, thiosulfate and thiocyanate. Ultraviolet detection was used instead of conductivity because of the high conductivity of the sodium perchlorate solution used for the separation.

### 3.2. Effect of eluent concentration

The influence of different eluent concentration on the retention of seven anions is shown in Fig. 2. As

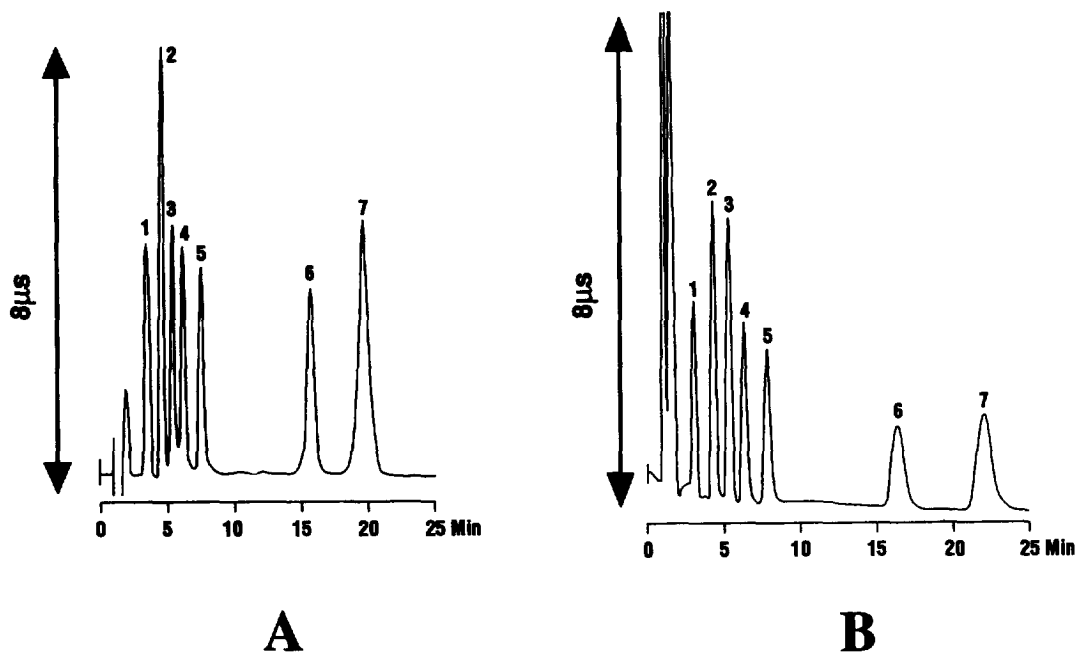


Fig. 6. Effect of methanol in *p*-hydroxybenzoic acid eluent. (A) 0% methanol. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 1.4 mM *p*-hydroxybenzoic acid, pH 8.0, 0% methanol, flow-rate: 1 ml/min, detector: conductivity; peaks: 1=fluoride (5 ppm); 2=chloride (10 ppm); 3=nitrite (10 ppm); 4=bromide (10 ppm); 5=nitrate (10 ppm); 6=phosphate (15 ppm); 7=sulfate (15 ppm). (B) 5% methanol. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 1.4 mM *p*-hydroxybenzoic acid, pH 8.0, 5% methanol, flow-rate: 1 ml/min, detector: conductivity; 1=fluoride (5 ppm); 2=chloride (10 ppm); 3=nitrite (10 ppm); 4=bromide (10 ppm); 5=nitrate (10 ppm); 6=phosphate (15 ppm); 7=sulfate (15 ppm).

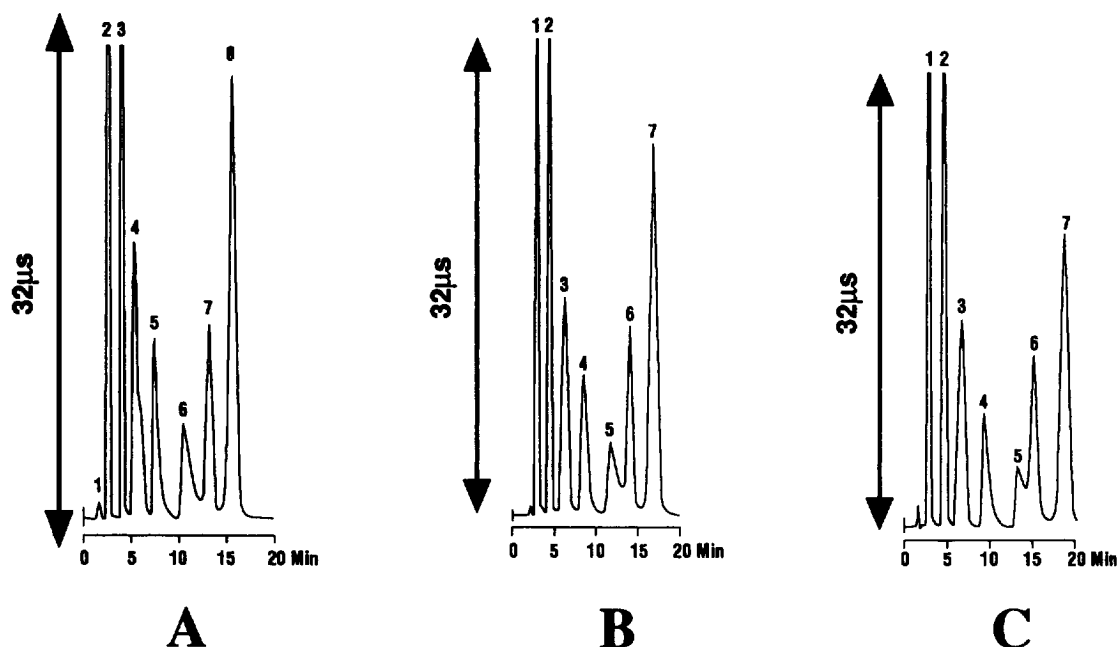


Fig. 7. Effect of adding acetonitrile to the eluent. (A) 0% acetonitrile. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 0% acetonitrile, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate; 7=sulfate (6 ppm). (B) 5% acetonitrile. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 5% acetonitrile, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate; 7=sulfate (6 ppm). (C) 10% acetonitrile. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 10 acetonitrile, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate; 7=sulfate (6 ppm).

expected, the retention of all seven anions decreased with increased eluent concentration. The retentions of fluoride, chloride, nitrite, phosphate and sulfate are purely based on ion-exchange mechanisms, which is why the retention times are reduced when the ionic strength of the eluent is increased. Bromide and nitrate retentions are affected by both ion-exchange and hydrophobic interaction mechanisms.

### 3.3. Effect of eluent pH

Since the P-DVB-based anion-exchanger is stable between pH values of 1 to 14, it can be used with very acidic or basic eluents. Variation of eluent pH at constant eluent concentrations and its effect on the retention time of anions is illustrated in Fig. 3. An eluent containing 1.4 mM *p*-hydroxybenzoic acid in 5% methanol is used for this study. The retention

times decreased with increase in eluent pH, as in most ion-exchange separations. The *p*-hydroxybenzoic acid is divalently charged at higher pH, becoming a stronger driving ion, eluting anions faster from the column.

### 3.4. Effect of organic additives in the eluent

The P-DVB-based anion-exchanger is an organic-compatible stationary phase which allows organic additives to alter the selectivity and improve the peak shape of anions. Three additives were chosen for this study with three different eluents using suppressor-based and single-column IC methods. The additives chosen were methanol, acetonitrile and uracil.

The effect of different concentrations of methanol in an eluent containing 0.68 mM sodium bicarbonate and 0.72 mM sodium carbonate is shown in Fig.

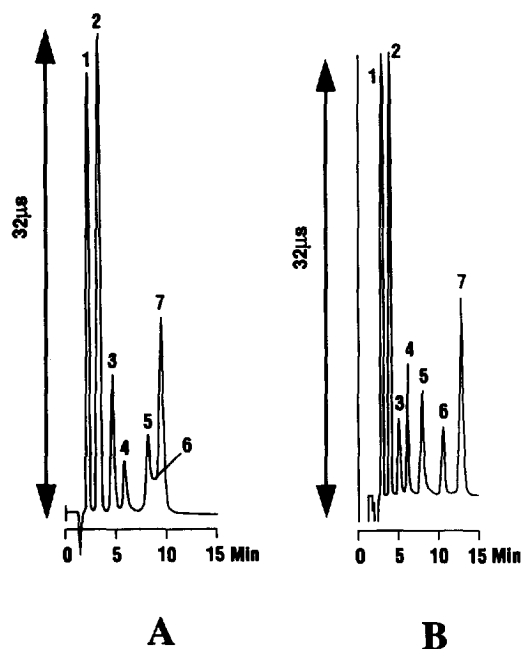


Fig. 8. (A) Effect of adding uracil to the eluent. (A) 0% uracil. Column: Alltech Durasep A-1 column,  $100 \times 4.6$  mm, eluent:  $1.02$  mM sodium bicarbonate– $1.08$  mM sodium carbonate, 0% uracil, flow-rate:  $1$  ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=phosphate (6 ppm); 6=nitrate (4 ppm); 7=sulfate (6 ppm). (B) 100 ppm uracil. Column: Alltech Durasep A-1 column,  $100 \times 4.6$  mm, eluent:  $1.06$  mM sodium bicarbonate– $1.08$  mM sodium carbonate, 100 ppm uracil, flow-rate:  $1$  ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate (6 ppm); 7=sulfate (6 ppm).

4A–E. These results show that too little or too much methanol is not good for the separation. When no methanol is used, the peak shape for nitrate is asymmetrical. This is due to the retention of nitrate by hydrophobic interaction on the hydrophobic P-DVB packing. When methanol is added, the hydrophobic interaction is reduced or eliminated, resulting in symmetrical peak shape for nitrate. The retention of phosphate and sulfate increased with increase in methanol concentration. This is probably due to the lower ion-exchange characteristics of the eluent when more methanol is added. No significant effect on other anions is observed. The optimized bicarbonate–carbonate eluent conditions for anion separation on the P-DVB stationary phase is  $0.68$  mM sodium

bicarbonate– $0.72$  mM sodium carbonate containing 10% methanol.

In suppressor-based IC sodium hydroxide is also a commonly used eluent for anion analysis. Adding methanol to sodium hydroxide eluent can improve the anion resolution on the P-DVB stationary phase. Fig. 5A and B are chromatograms of anion separation using  $2$  mM sodium hydroxide eluent with and without methanol. Without methanol, bromide and nitrate peaks are co-eluted. Addition of 10% methanol to the eluent resolved bromide and nitrate peaks. Phosphate, which exists as a trivalent anion at this pH, is not eluted within reasonable time with sodium hydroxide eluent.

The effect of methanol on peak shape and resolution when using single-column IC eluent such as *p*-hydroxybenzoic acid is shown in Fig. 6A and B. As shown in Fig. 6A, the resolution between monovalent anions is not very good without methanol. By adding methanol to the eluent, the resolution between all anions is improved, as shown in Fig. 6B. It is interesting to note that when no methanol is added to the *p*-hydroxybenzoic acid eluent, the peak shape for nitrate is comparatively better than the peak shape obtained using sodium bicarbonate–carbonate or sodium hydroxide eluent (when no methanol added). This may be due to the similar aromatic background of *p*-hydroxybenzoic acid and the packing, resulting in less hydrophobicity of the stationary phase.

The influence of acetonitrile on the anion selectivity was also investigated by injecting anion standards under optimized chromatographic conditions and adding different concentrations of acetonitrile to the eluent. Fig. 7A–C shows the chromatograms obtained with 0, 5 and 10% acetonitrile concentrations. These results are the opposite to the methanol effect. With increased acetonitrile content in the eluent, the retention time for nitrate increased, causing poor resolution between nitrate and phosphate peaks. Acetonitrile seems to increase the hydrophobicity of the column, hence the nitrate retention is increased.

Several other organic additives such as nitromethane and uracil were tried to improve the anion separation on the P-DVB column. Use of nitromethane as the additive had to be discontinued, even though great separation could be achieved, due to the hazardous nature of this compound. The effect of



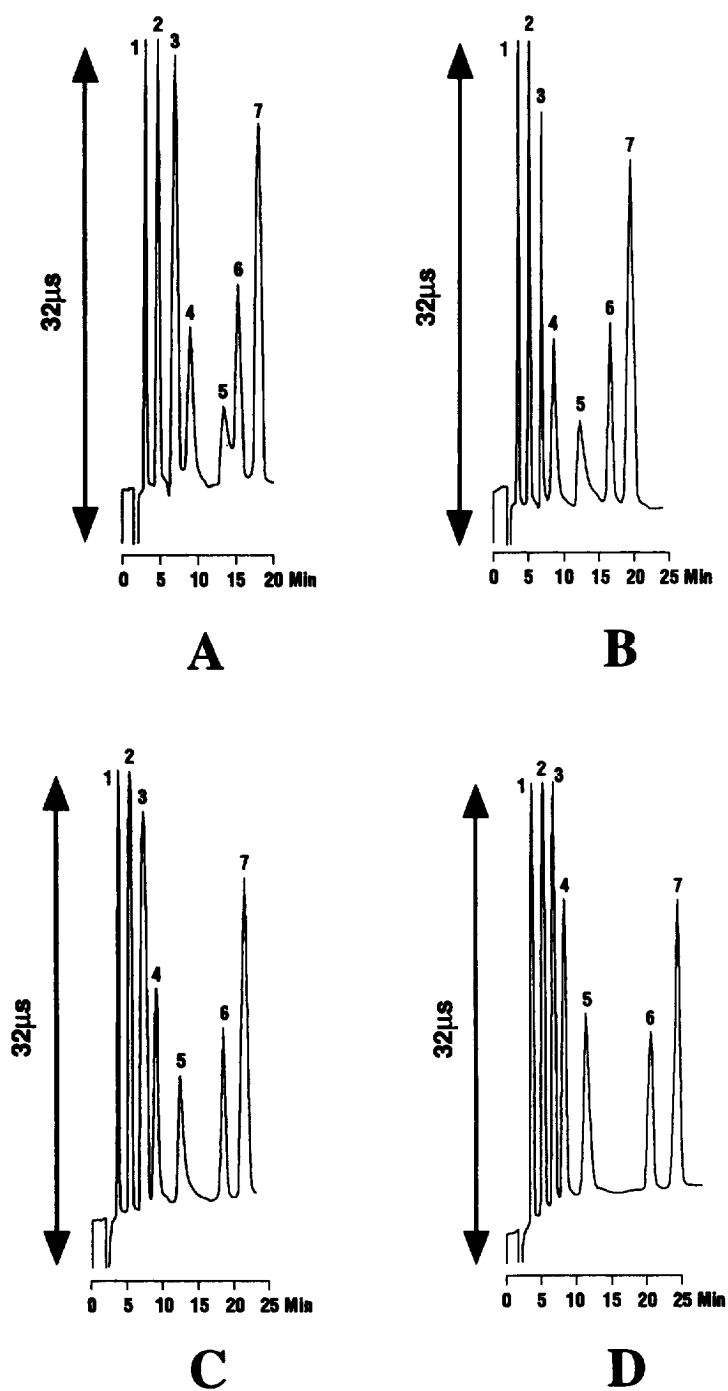


Fig. 9. Effect of temperature on the selectivity of anions. (A) At 25°C. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (1 ppm); 2=chloride (2 ppm); 3=nitrite (2 ppm); 4=bromide (2 ppm); 5=nitrate (2 ppm); 6=phosphate (3 ppm); 7=sulfate (3 ppm). (B) At 35°C. Conditions and peaks as in (A). (C) At 45°C. Conditions and peaks as in (A). (D) At 55°C conditions and peaks as in (A).

Table 1  
The efficiency of P-DVB-based column

Anion	Efficiency (100×4.6 mm column)
Nitrate	2693
Phosphate	5078
Sulfate	5447

The efficiency (number of theoretical plates per column) was calculated using the half-height method.

Column=100×4.6 mm, P-DVB column. Eluent=0.68 mM sodium bicarbonate–0.72 mM sodium carbonate in 10% methanol.

uracil in the eluent (1.02 mM bicarbonate–1.08 mM carbonate) is illustrated in Fig. 8A and B. Without uracil, nitrate is not resolved from phosphate. By adding 100 ppm uracil to the eluent all seven anions are well resolved, especially nitrate and phosphate. Uracil is a strong electron donor which can coat (by covalent bonding) the aromatic ring of the P-DVB resin, thus suppressing the hydrophobic interactions. This results in improved resolution and peak shape for bromide and nitrate ions.

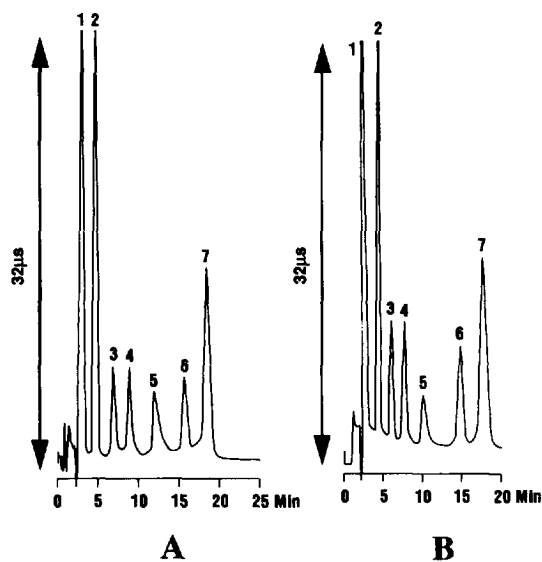


Fig. 10. Durability study. (A) First injection. Column: Alltech Durasep A-1 column, 100×4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate with 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride (2 ppm); 2=chloride (4 ppm); 3=nitrite (4 ppm); 4=bromide (4 ppm); 5=nitrate (4 ppm); 6=phosphate (6 ppm); 7=sulfate (6 ppm). (B) 2500th injection. Conditions and peaks as in (A).

### 3.5. Effect of temperature

Chromatographic separations can be manipulated using temperature. Since the P-DVB stationary phase can tolerate temperature up to 75°C, changing the column temperature was considered worth investigating. Separations of anions using bicarbonate–carbonate eluent without any additives, but varying column temperatures were performed to study the temperature effect. Fig. 9A–D show the chromatograms obtained at 25, 35, 45 and 55°C. At 25°C nitrate is only partially resolved from the phosphate peak. With increase in column temperature, the hydrophobic interaction becomes less prominent, resulting in improved peak shape for the nitrate ion. The retention of divalent anions increased slightly at higher temperature. These results show that temperature affects the separation the same way as the organic additives. Instead of using organic additives to improve the separation on the P-DVB based packing, one can manipulate the operating temperature to get the same results. The adsorptive effects of the P-DVB resin are greatly influenced by changes in temperature.

### 3.6. Chromatographic efficiency

Table 1 shows the efficiencies calculated for nitrate, phosphate and sulfate peaks on the P-DVB column. The efficiencies are reported as number of theoretical plates ( $N$ ) per column, calculated by the half height method ( $N=5.54 (t_R/w_{1/2})^2$ ). These efficiencies are comparable to the efficiencies reported for many commercially available columns [5]. The lower efficiency for the nitrate peak may be due to the hydrophobic nature of the packing material.

### 3.7. Lifetime study

The durability of P-DVB stationary phase was studied by injecting anion standards continuously, under optimized conditions. These experiments proved that after 2500 repeated injections, the column still maintained the same resolving power and reproducible retention as in the first injection. Fig. 10A and B are chromatograms of first and 2500th injections of anion standards on this column.

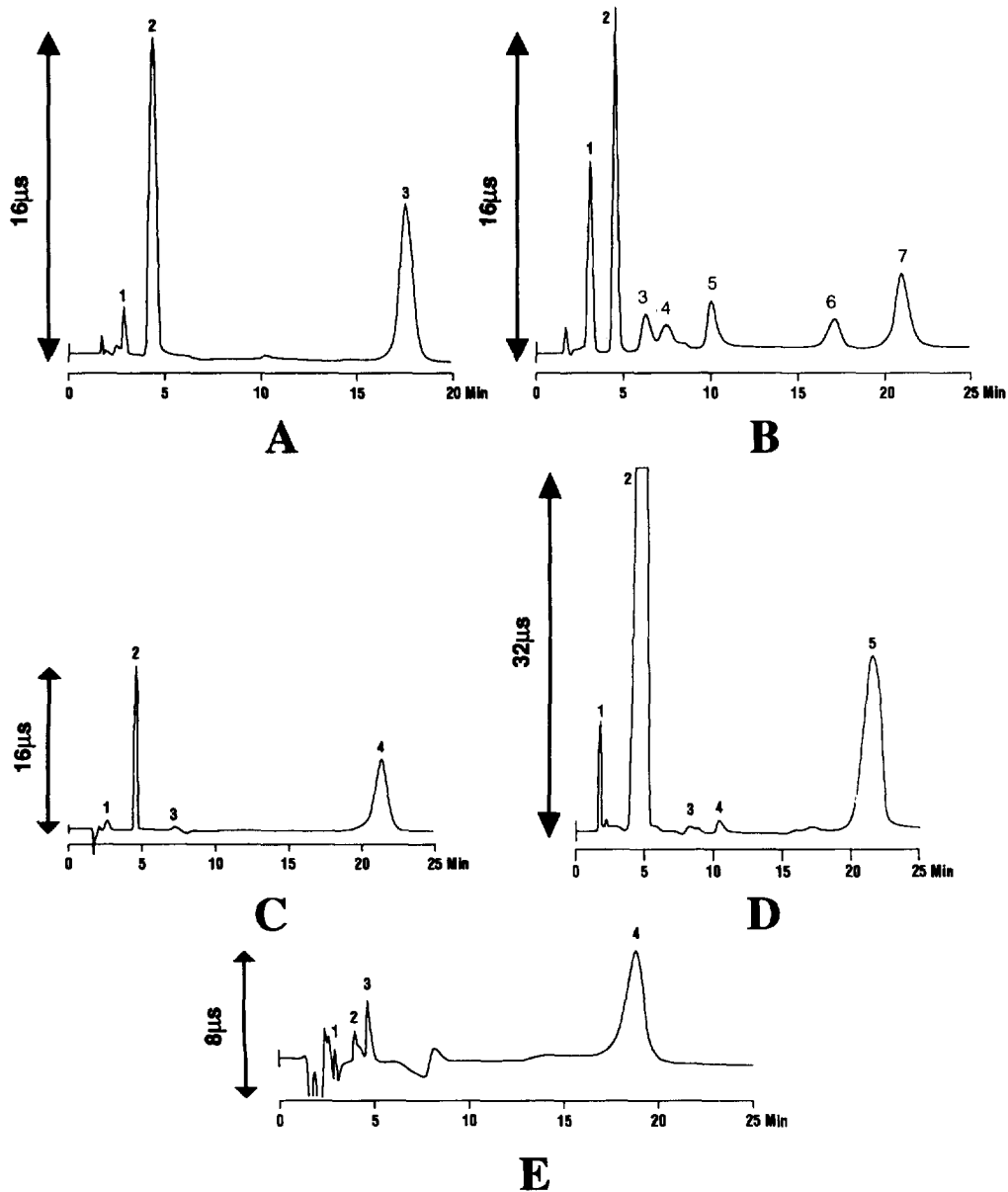


Fig. 11. Applications of real world samples. (A) Tap water. Column: Alltech Durasep A-1 column, 100 $\times$ 4.6 mm, eluent; 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride; 2=chloride; 3=sulfate. (B) Anions in fertilizer. Column: Alltech Durasep A-1 column, 100 $\times$ 4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride; 2=chloride; 3=nitrite; 4=unknown; 5=nitrate; 6=phosphate; 7=sulfate. (C) Power plant water. Column: Alltech Durasep A-1 column, 100 $\times$ 4.6 mm, eluent; 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride; 2=chloride; 3=nitrite; 4=sulfate. (D) Swimming pool water. Column: Alltech Durasep A-1 column, 100 $\times$ 4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride; 2=chloride; 3=nitrite; 4=nitrate; 5=sulfate. (E) Anions in colloidal silica. Column: Alltech Durasep A-1 column, 100 $\times$ 4.6 mm, eluent: 0.68 mM sodium bicarbonate–0.72 mM sodium carbonate, 10% methanol, flow-rate: 1 ml/min, detector: suppressed conductivity; peaks: 1=fluoride; 2=carbonate; 3=chloride; 4=sulfate.

### 3.8. Applications

The suitability of P-DVB stationary phase for real world sample analyses are shown in Fig. 11A–E. No chromatographic interferences were observed when water samples were injected. The colloidal silica sample had carbonate that interfered with the chloride peak. By treating the sample with Novo-Clean IC-H disk, this problem was eliminated.

### 4. Conclusion

The anion-exchange stationary phase made of P-DVB material is highly efficient and durable for anion analysis by IC. The column is useful for both suppressor-based or single-column IC applications. Eluents such as sodium bicarbonate–carbonate, sodium hydroxide, *p*-hydroxybenzoic acid and sodium perchlorate can be successfully used for this purpose. The separation of the anions can be improved by slight modifications of the chromatographic conditions. Adding organic additives to the eluent or

increasing the column temperature improves the resolution between all anions and peak shape for hydrophobic anions. The organic compatibility of the column also offer other advantages. For example, it can be used to remove organic contaminants that may retain on the column through vigorous clean-up. Eluent concentration and pH also play important roles on the retention characteristics of anions on the P-DVB stationary phase.

### References

- [1] P.R. Haddad and P.E. Jackson, *Ion Chromatography Principles and Applications*, Elsevier, Amsterdam, 1990, Ch. 3.
- [2] J. Weiss, *Ion Chromatography*, Weinheim, New York, 2nd ed., 1995, Ch. 3.
- [3] R. Saari-Nordhaus and J.M. Anderson, *Am. Lab.*, January 1994.
- [4] D.T. Gjerde and J.S. Fritz, *Ion Chromatography*, Huthig, New York, 1987, Ch. 7.
- [5] P.R. Haddad, P.E. Jackson and A.L. Heckenberg, *J. Chromatogr.*, 346 (1985) 139.