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# A new approach to dealing with high-to-low concentration ratios of sodium and ammonium ions in ion chromatography

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**Abstract**

In ion chromatography, samples of very dissimilar concentration ratios of ammonium-to-sodium are difficult to quantify since these two cations have similar selectivities for stationary phases containing commonly used sulfonic acid or carboxylic acid cation-exchange functional groups. A new column with carboxylic acid and phosphonate functional groups as well as a crown ether group has been developed to address this limitation. Selectivity of the common inorganic cations is different from conventional columns in that the separation between sodium and ammonium has been greatly increased, and potassium ion elutes after magnesium and calcium. Applications involving very dissimilar concentration ratios of cations can now be done isocratically, with a single column. Other applications of this new column will also be discussed. © 1998 Elsevier Science B.V.

*Keywords:* Stationary phases; Mobile phase composition; Sodium; Ammonium; Inorganic cations

## 1. Introduction

In environmental samples usually low levels of ammonium need to be determined in the presence of high concentration levels of sodium, and in the power industry the converse is true. These applications often pose a challenge to ion chromatography, where sodium and ammonium often elute next to each other. Typically, cation-exchange columns with sulfonic acid functional groups do not have high selectivity for hydronium ions, and in order to elute the divalent cations in a reasonable time, a divalent eluent component such as 1,2-diaminopropionic acid needs to be present [1]. With this strong eluent system, monovalent cations, such as sodium and ammonium, elute in close proximity to each other, as is shown in Fig. 1.

Cation-exchange columns with carboxylic acid functional groups [2–4] also have similar selectivities for sodium and ammonium ions, as shown in Fig. 2.

Ethanolamines are only partially resolved from the common Group I and Group II cations in stationary phases containing carboxylic acid functional groups. Fig. 3 shows an eluent step separation on such a column. The initial concentration of acid in the eluent is very low to enhance the resolution of the monovalent cations. Once these have eluted, the acid concentration in the eluent is increased to elute the divalent cations in a reasonable time. There is a baseline change when the higher acid concentration is used. A cation trap column needs to be placed before the injection valve so that impurities in the

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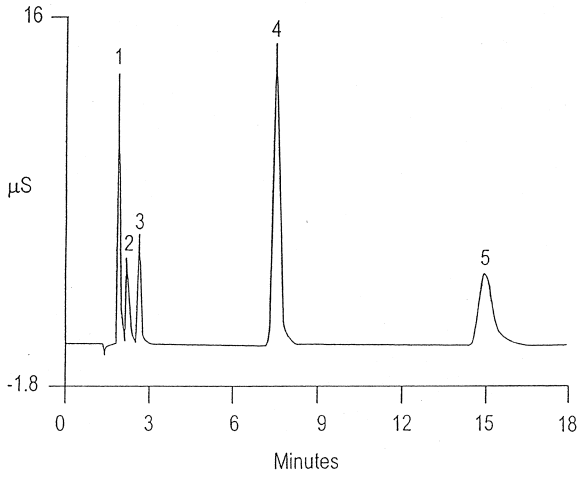


Fig. 1. Isocratic separation of common cations on a sulfonated polymeric stationary phase. Column: IonPac CS10 (Dionex). Eluent: 40 m*M* HCl+4 m*M* 1,2-diaminopropionic acid monohydrochloride. Flow-rate: 1.0 ml/min. Injection volume: 10 μl. Peaks: 1=sodium (5 mg/l); 2=ammonium (5 mg/l); 3=potassium (5 mg/l); 4=magnesium (10 mg/l); 5=calcium (10 mg/l).

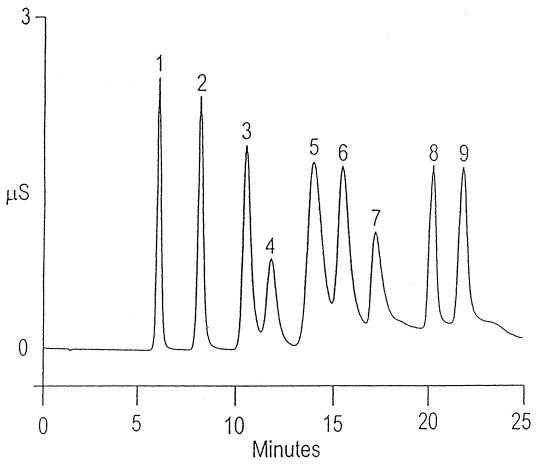


Fig. 3. Eluent step change separation of ethanolamines from the six common cations on a carboxylic acid column. Column: IonPac CS12. Eluent: 5 m*M* methanesulfonic acid, step change at 12 min to 21 m*M* methanesulfonic acid. Flow-rate: 1.0 ml/min. Injection volume: 25 μl. Peaks: 1=lithium (0.5 mg/l); 2=sodium (2 mg/l); 3=ammonium (2.5 mg/l); 4=ethanolammonium (4 mg/l); 5=diethanolammonium (25 mg/l); 6=potassium (5 mg/l); 7=triethanolammmonium (200 mg/l); 8=magnesium (2.5 mg/l); 9=calcium (5 mg/l).

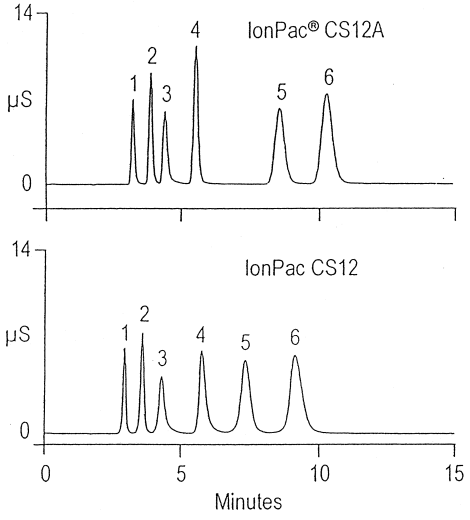


Fig. 2. Isocratic separation of the six common cations on two carboxylic acid polymeric stationary phases. Eluent: 11 m*M* sulfuric acid. Flow-rate: 1.0 ml/min. Injection volume: 25 μl. Peaks: 1=lithium (0.5 mg/l); 2=sodium (2 mg/l); 3=ammonium (2.5 mg/l); 4=potassium (5 mg/l); 5=magnesium (2.5 mg/l); 6=calcium (5 mg/l).

weak eluent are not concentrated in the column, potentially creating a high blank.

Column switching between a sulfonic acid and a carboxylic acid column [5] greatly increases the resolution between sodium and ammonium ions, and allows determination of sodium-to-ammonium concentration ratios in the order of up to 20 000-to-1. This is done isocratically, and chromatographic results are shown in Fig. 4. Column switching, however, has its drawbacks. For example, column switching is a more expensive alternative as two columns are needed instead of just one. Column switching also requires a switching valve. In addition, column switching is found by many to be complicated and not very rugged. Troubleshooting a problem in this chromatographic system is more difficult, as more components are involved. These problems were the driving force for the development of a new stationary phase. In order for this new column to provide a simpler solution, it had to have very different selectivity from the traditional sulfonic acid and carboxylic acid cation-exchange groups.

Crown ethers are macrocyclic ligands [6] with

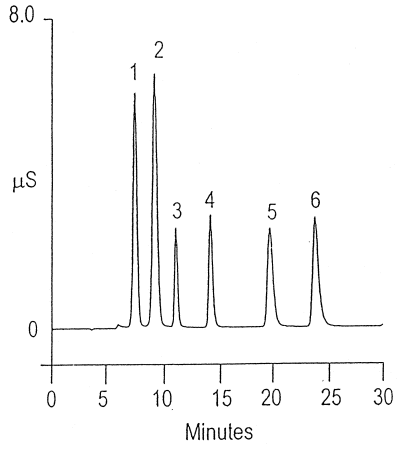


Fig. 4. Separation of the six common cations via column switching. Columns: IonPac CS12A and CS10. Eluent: 24 m*M* methanesulfonic acid. Column switching time: 6 mintes. Flow-rate: 1.0 ml/min. Injection volume: 25 μl. Peaks: 1=magnesium (2.5 mg/l); 2=calcium (5 mg/l); 3=lithium (0.5 mg/l); 4=sodium (2 mg/l); 5=ammonium (2.5 mg/l); 6=potassium (5 mg/l).

hydrophilic interior cavities and hydrophobic exteriors; they form stable complexes by coordinating metal cations within their cavities. Pedersen [7] was the first to observe complex formation of the ammonium cation with 18-crown-6. Selectivity of these cyclic polyethers for cations depends on the relationship between the crown ether cavity and the metal ion diameters [8]. They can be used in the mobile phase, applied through physical adsorption or as a coating on a solid support, or covalently bonded to silica or polymeric packings [9].

## 2. Experimental

### *2.1. Apparatus*

All experiments were carried out with a DX 500 ion chromatographic system (Dionex, Sunnyvale, CA, USA) consisting of a quaternary gradient pump (GP 40) with automated membrane eluent degassing, a chromatographic oven (LC 30), and suppressed conductivity detection (CD 20 conductivity detector). Eluent flow-rate, unless otherwise stated, was 1.2 ml/min for the 4 mm I.D. column format, and 0.3 ml/min for the 2 mm column format. All instrument control, data collection and data processing was performed with the PeakNet Chromatography Workstation (Dionex). The output of the conductivity detector is automatically normalized so that a readout of 1 μS is equivalent to 1 μS/cm. When neither acetonitrile nor hydroxylamine sulfate was used in the eluent, the cation self-regenerating suppressor (CSRS-2) was used in the recycle mode; when either of the two was used as an eluent component, the suppressor was used in the external water mode.

### *2.2. Stationary phases*

The IonPac CS15 stationary phase consists of polymeric macroporous substrate beads with carboxylic acid groups, phosphonate groups, and 18-crown-6 ether groups permanently attached (Dionex). The IonPac CS15 is the new column mainly used throughout this work. It will be compared to the IonPac CS12A column, which is similar to the CS15 resin except that it has no crown ether functional groups incorporated in its structure. The raw resin for either column has an average particle size and pore size of 8.5 mm and 150 Å respectively, and the average surface area of the substrate beads is 450 $m^2/g$. Dimensions of the columns are 250×4 mm for standard bore application, and 250×2 mm for narrow bore. Unless otherwise stated, the 4 mm column format was used in this work.

### *2.3. Chemicals*

Deionized water (18 MΩ cm resistivity at room temperature) from a water purification system (Continental Type I, Laboratory Reagent Grade Water System) was used for the preparation of the eluents and standards. Methanesulfonic acid (Fluka) and sulfuric acid (Aldrich) were of analytical reagent grade. Acetonitrile (UV grade for high-performance liquid chromatography) was from Burdick and Jackson. Hydroxylamine sulfate, 99% purity, was from Aldrich. Standards were prepared from analytical reagent grade chemicals.

## 3. Results and discussion

### *3.1. Comparison of the IonPac CS12A and the CS15*

The CS15 is similar to its predecessor, the CS12A column, but it also has an 18-crown-6 ether incorporated in its structure. Fig. 5 shows the selectivity change obtained when the crown ether is attached to the stationary phase. Note that potassium, a monovalent, elutes last in the CS15. Note also the enhanced resolution between sodium and ammonium, peaks 2 and 3. Potassium and ammonium are retained longer in the stationary phase as they form a more stable complex with the crown ether.

### *3.2. Effect of temperature on the IonPac CS15 with aqueous-only and solvent-containing eluent*

Fig. 6 shows the effect of temperature on the separation of lithium, sodium, ammonium, potassium, magnesium and calcium in the CS15 column. The two chromatograms were obtained under identical eluent conditions (acetonitrile and sulfuric acid), just the column temperature was varied. At 40°C, potassium (peak 6) improves in peak shape and efficiency, and elutes earlier. The divalent cations, on the other hand, elute later when the column temperature is raised. It is speculated that sulfate ions from the eluent form a complex with the divalent cations, reducing the effective positive charge of these [4]. This complex is not as stable at the higher temperature, so that divalent cations are then retained longer by the stationary phase.

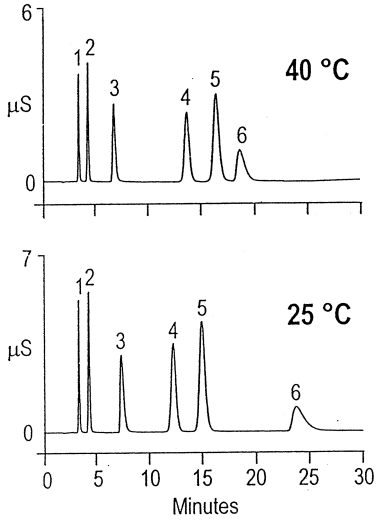


Fig. 6. Temperature effect on the CS15 with a solvent-containing eluent. Column: IonPac CG15 and CS15. Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 1.2 ml/min. Injection volume: 25 µl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=ammonium (10 mg/l); 4=magnesium (5 mg/l); 5=calcium (10 mg/l); 6=potassium (10 mg/l).

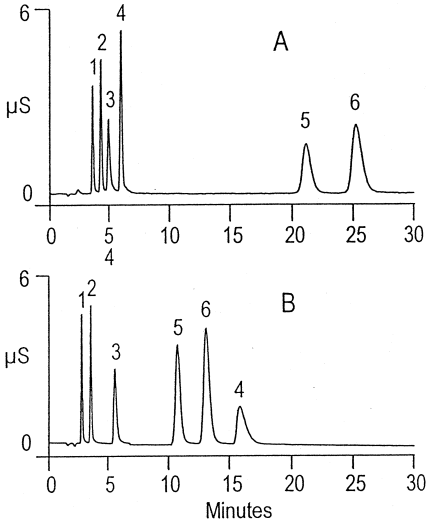


Fig. 5. Comparison of IonPac CS12A and CS15. Chromatogram A: IonPac CS12A (no crown ether). Chromatogram B: IonPac CS15 (with crown ether). Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 1.2 ml/min. Column temperature: 40°C. Injection volume: 25 µl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=ammonium (10 mg/l); 4=potassium (10 mg/l); 5=magnesium (5 mg/l); 6=calcium (10 mg/l).

Fig. 7 shows an eluent containing only sulfuric acid and water, no solvent, and the effect of temperature on the chromatography. Once again, there is an improvement in the peak symmetry of potassium (and of ammonium to a lower extent).

### *3.3. High-to-low concentration ratios of the common monovalent cations*

The 18-crown-6 ether in the stationary phase is responsible for the high resolution between sodium and ammonium ions, allowing quantitation of low levels of one in the presence of high levels of the other. Fig. 8 shows a 4000-to-1 concentration ratio of

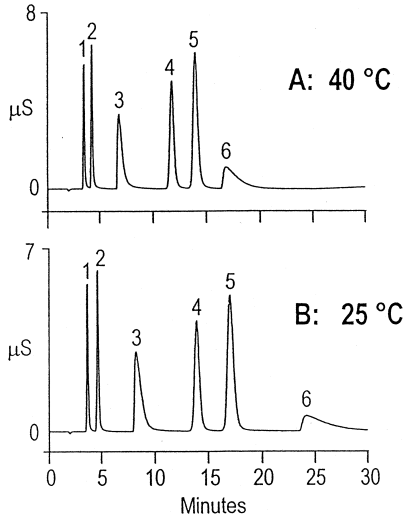


Fig. 7. Temperature effect on the CS15 with an aqueous eluent. Column: IonPac CG15 and CS15. Eluent A: 7.5 m*M* sulfuric acid. Eluent B: 6.5 m*M* sulfuric acid. Flow-rate: 1.2 ml/min. Injection volume: 25 µl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=ammonium (10 mg/l); 4=magnesium (5 mg/l); 5=calcium (10 mg/l); 6=potassium (10 mg/l).

sodium to ammonium ion with the CS15 2 mm column and an isocratic eluent at 40°C. Note the better signal-to-noise ratio obtained when a ten times larger sample injection loop was used. Also, note that using this much larger sample volume [10] does not cause appreciable decrease in peak efficiency, and that the large amount of sodium does not cause overloading effects that compromise the elution of ammonium.

In Fig. 9 we see the reverse situation of that in Fig. 8: a 10 000-to-1 ratio of ammonium to sodium ions. In this case, a 2 mm CS15 column was used under standard isocratic eluent conditions with a 1000 µl sample loop. Chromatogram A shows a 10 mg/l standard of ammonium; the small peak 1 is probably sodium impurities in the standard and in the water used. In chromatogram B, the same standard was spiked with 1 µg/l of sodium. Peak 3, not quantitated, corresponds to calcium impurities. Note that by doing a direct injection on the CS15 column with a large (1 ml) sample loop, it is possible to quantitate isocratically and with one column, 1 ppb of sodium in the presence of 10 ppm of ammonium.

The CS15 is ideal for samples with high concentrations of potassium, as the high selectivity which potassium has for the 18-crown-6 ether in the stationary phase allows it to elute last. Fig. 10 shows a sample containing a 10 000-to-1 ratio of potassium to ammonium, such as can be encountered in KCl

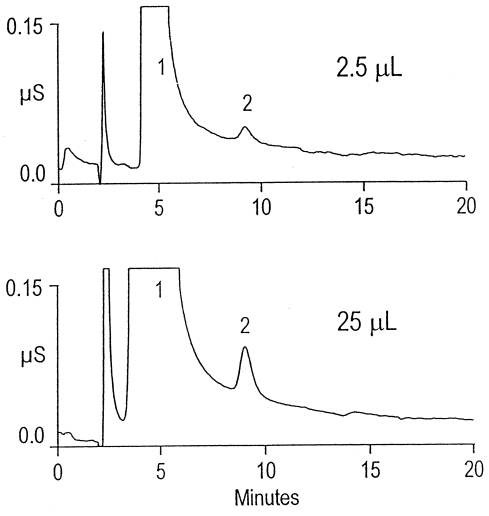


Fig. 8. High sodium-to-low ammonium and sample volume. Column: IonPac CG15 and CS15, 2 mm. Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 0.3 ml/min. Column temperature: 40°C. Peaks: 1=sodium (100 mg/l); 2=ammonium (0.025 mg/l).

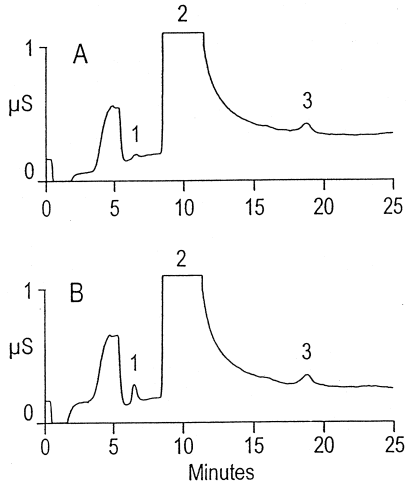


Fig. 9. High ammonium-to-low sodium. Column: IonPac CG15 and CS15, 2 mm. Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 0.3 ml/min. Column temperature: 40°C. Injection volume: 1000 µl. Peaks: 1=sodium; 2=ammonium (10 mg/l); 3=calcium. Sample in chromatogram B was spiked with 0.001 mg/l sodium.

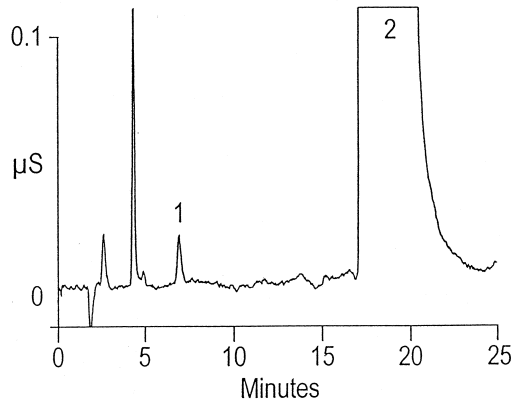


Fig. 10. High potassium-to-low ammonium. Column: IonPac CG15 and CS15. Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 1.2 ml/min. Column temperature: 40°C. Injection volume: 25 µl. Peaks: 1=ammonium (0.01 mg/l); 2=potassium (100 mg/l).

extracts of a soil sample. Fig. 11 shows a real soil sample extracted with 1 *M* ammonium acetate. Notice that in spite of the large amounts of ammonium and calcium in the sample, the other cations can still be quantitated.

### *3.4. Isocratic separation of power plant additives*

In stationary phases with carboxylic acid functionalities (such as the CS12A), ethanolamine, a popular additive used in the power industry to inhibit corrosion, and ammonium, elute close to each other. Eluent gradients or step changes are necessary to improve the resolution of these two, and even then they are not completely separated. Ethanolammonium has a p$K_a$ of about 9.52 [11], and is positively charged in the acidic eluents commonly used with carboxylic acid columns; similarly to ammonium ion, it is retained by cation-exchange in these columns. In the case of the CS15, the selectivity of the 18-crown-6 ether for ammonium is greater than for ethanolammonium. Ammonium ion is therefore retained longer in the stationary phase, and the two peaks are well resolved from each other under isocratic eluent conditions (see Fig. 12). In Fig. 13, a sample matrix of 200 ppm ethanolamine was found to contain 10 ppb of sodium, or a 20 000-to-1 concentration ratio of ethanolamine to sodium ion. The shoulder on the ethanolammonium peak is probably ammonium ion impurities. In Fig. 14 we can see the isocratic elution and baseline resolution of morpholine, another common additive used in the power industry to inhibit corrosion.

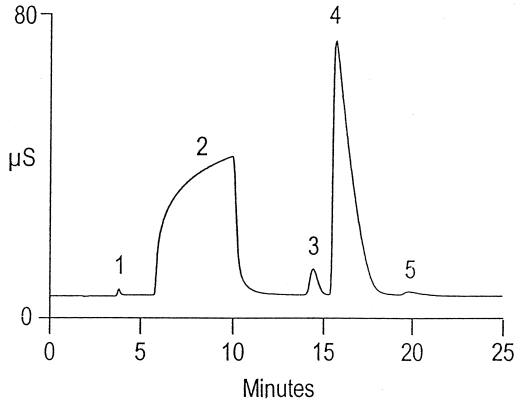


Fig. 11. Ammonium acetate extraction of a soil sample. Column: IonPac CG15 and CS15. Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 1.2 ml/min. Injection volume: 25 µl. Column temperature: 40°C. Peaks: 1=sodium; 2=ammonium; 3=magnesium; 4=calcium; 5=potassium.

### *3.5. Effect of hydroxylamine as eluent*

Hydroxylamine is a derivative of ammonia where

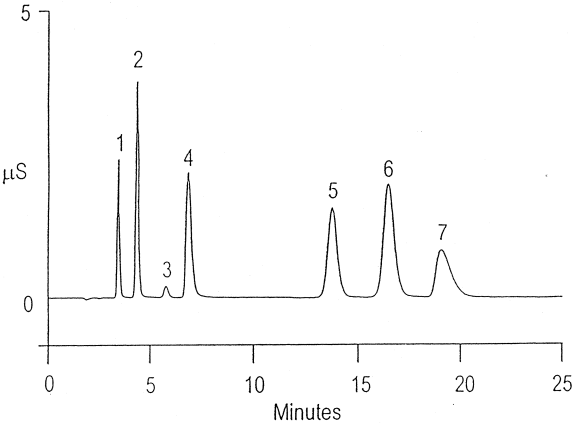


Fig. 12. Isocratic separation of ethanolammonium from the six common cations. Column: IonPac CG15 and CS15. Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 1.2 ml/min. Column temperature: 40°C. Injection volume: 25 µl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=ethanolammonium (3 mg/l); 4=ammonium (5 mg/l); 5=magnesium (5 mg/l); 6=calcium (10 mg/l); 7=potassium (10 mg/l).

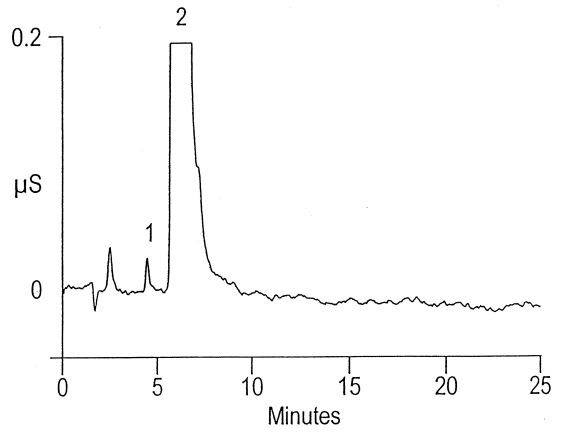


Fig. 13. High ethanolamine-to-low sodium. Column: IonPac CG15 and CS15. Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 1.2 ml/min. Column temperature: 40°C. Injection volume: 25 µl. Peaks: 1=sodium (0.01 mg/l); 2=ethanolammonium (200 mg/l).

a hydrogen atom has been replaced by a hydroxyl group. This electron-withdrawing group makes it a weaker base than ammonia, with a p$K_a$ of approximately 6 while that of ammonia is about 9.2 [12]. The eluent, 7.5 m*M* sulfuric acid, corresponds to a pH of about 1.8, and at this pH hydroxylamine is positively charged. It therefore helps elute cations retained by cation-exchange interaction with the carboxylic acid groups of the stationary phase. Furthermore, the 18-crown-6 ether used in the stationary phase has similar selectivity for hydroxylamine and ammonia. Thus, hydroxylamine is a strong eluting ion for the analytes which are strongly complexed by the 18-crown-6 ether groups of the stationary phase, such as potassium and ammonium.

The result is seen in Fig. 15, where chromatogram A was obtained with an eluent containing 7.5 m*M* sulfuric acid, and chromatogram B was obtained by adding to it 15 m*M* hydroxylamine. In both cases, the systems were run at room temperature. Notice the improvement in peak symmetry and efficiency for ammonium and potassium when hydroxylamine is added to the eluent (chromatogram B). Total run time was reduced to almost $\frac{1}{3}$, and is comparable under these conditions to that of the CS12A. A limitation of using hydroxylamine as an eluent component is that it increases the noise by about a factor of 5. Also, the suppressor cannot be used in the eluent recycle mode, instead it must be used in the external water mode.

In the previous chromatograms, sulfuric acid was

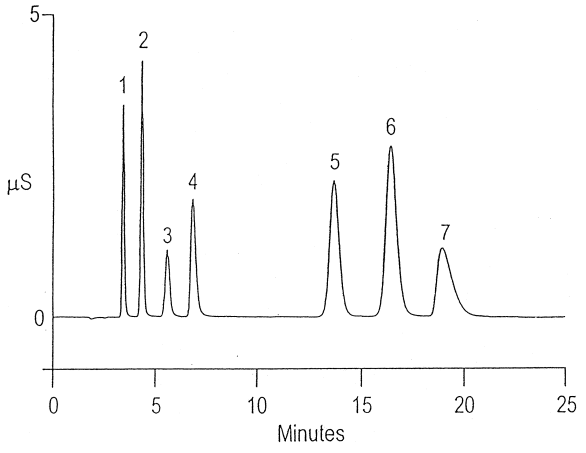


Fig. 14. Isocratic separation of morpholine from the six common cations. Column: IonPac CG15 and CS15. Eluent: 5 m*M* sulfuric acid+9% acetonitrile. Flow-rate: 1.2 ml/min. Column temperature: 40°C. Injection volume: 25 µl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=morpholine (50 mg/l); 4=ammonium (5 mg/l); 5=magnesium (5 mg/l); 6=calcium (10 mg/l); 7=potassium (10 mg/l).

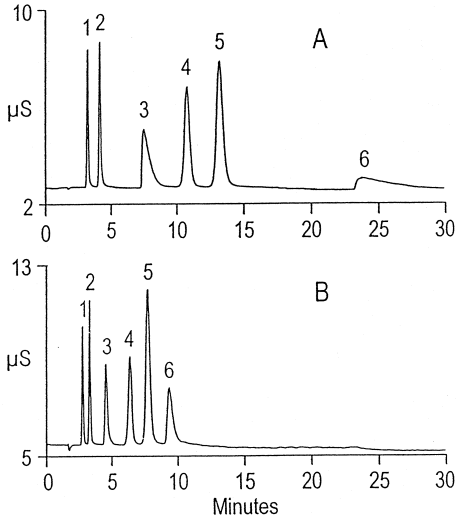


Fig. 15. Effect of hydroxylamine as eluent in the CS15. Column: IonPac CG15 and CS15. Eluent A: 7.5 m*M* sulfuric acid. Eluent B: 7.5 m*M* sulfuric acid+7.5 m*M* hydroxylamine. Flow-rate: 1.2 ml/min. Injection volume: 25 µl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=ammonium (10 mg/l); 4=magnesium (5 mg/l); 5=calcium (10 mg/l); 6=potassium (10 mg/l).

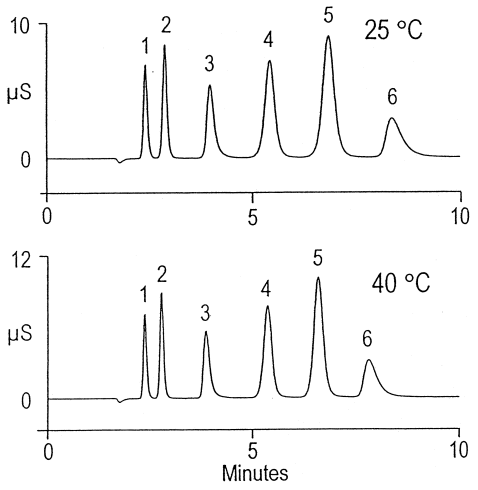


Fig. 16. Effect of temperature when the eluent contains hydroxylamine. Column: IonPac CG15 and CS15. Eluent: 15 m*M* methanesulfonic acid+7.5 m*M* hydroxylamine. Flow-rate: 1.2 ml/min. Injection volume: 25 µl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=ammonium (10 mg/l); 4=magnesium (5 mg/l); 5=calcium (10 mg/l); 6=potassium (10 mg/l).

used as eluent. In Fig. 16, methanesulfonic acid was used instead. For the same acid concentration, methanesulfonic acid results in slightly faster elution of the analytes, but the main difference between the two acids is how divalent cations behave at elevated temperature with one acid versus the other [4]. In the case of sulfuric acid, as discussed above in Section 3.2, elevated temperature causes divalent cations to actually elute later. On the other hand, in the case of methanesulfonic acid, elevated temperature causes faster elution of the divalent cations. For practical purposes, in general, either acid can be used.

Efficiency and peak symmetry for ammonium and (especially) potassium dramatically improved when the CS15 column was run at elevated temperature with sulfuric acid as eluent (see Fig. 7). When acetonitrile was added to the eluent (Fig. 6), the effect of temperature was lessened, but still noticeable. In Fig. 16 we can see that the separation, the total run time, and the peak efficiencies of the analytes already are more than adequate at room temperature when hydroxylamine is used. Raising the temperature only decreases slightly the total run time and slightly improves peak efficiencies. This indicates that hydroxylamine is actually a more effective eluting ion than acid, water or acetonitrile for the interaction of ammonium and potassium with the crown ether in the stationary phase.

### *3.6. Fast elution of the common six cations with the CS15*

In Fig. 17, the eluent in chromatogram A (sulfuric acid and acetonitrile) was optimized to give the fastest elution of the six common cations with baseline resolution. Total analysis time with the CS15 guard and separator columns was about 13 min.

Elution of these cations can be almost 50% faster when hydroxylamine is present in the eluent, as is shown in chromatogram B of Fig. 17. Furthermore, resolution among ammonium, magnesium and calcium is improved.

Fig. 18 shows the fastest run of the common six cations that can be obtained with a CS12A guard and separator columns. Compared to chromatogram B in Fig. 17, total run time can be almost as fast as with a CS15 guard and separator columns, but resolution among the sodium and ammonium peaks is better on the ‘‘fast run’’ of the CS15.

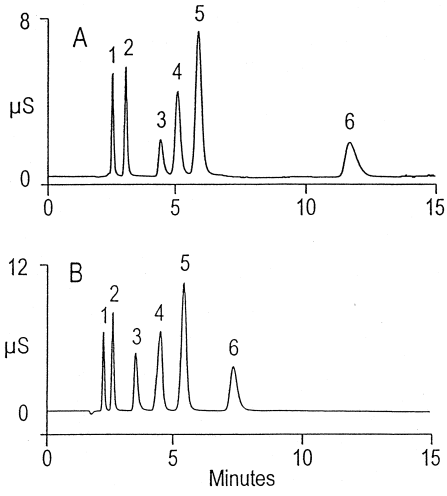


Fig. 17. Fast analysis with the CS15. Column: IonPac CG15 and CS15. Eluent A: 9 m*M* sulfuric acid+13% acetonitrile. Eluent B: 15 m*M* methanesulfonic acid+7.5 m*M* hydroxylamine+5% acetonitrile. Column temperature: 40°C. Flow-rate: 1.2 ml/min. Injection volume: 25 µl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=ammonium (10 mg/l); 4=magnesium (5 mg/l); 5=calcium (10 mg/l); 6=potassium (10 mg/l).

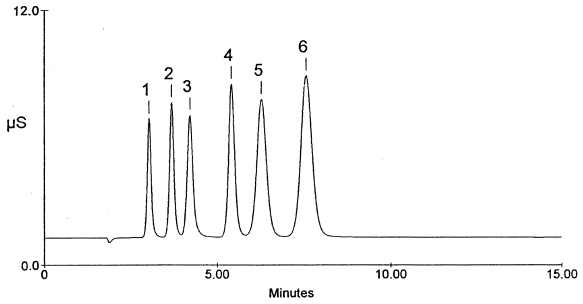


Fig. 18. Fast analysis with the CS12A. Column: IonPac CG12A and CS12A. Eluent: 15 m*M* sulfuric acid. Flow-rate: 1.0 ml/min. Injection volume: 25 μl. Peaks: 1=lithium (1 mg/l); 2=sodium (4 mg/l); 3=ammonium (10 mg/l); 4=potassium (10 mg/l); 5=magnesium (5 mg/l); 6=calcium (10 mg/l).

## 4. Conclusions

Incorporating an 18-crown-6 ether group in the structure of polymeric carboxylic acid stationary phases dramatically changes the selectivity properties for this new column as compared to conventional cation-exchange materials with either carboxylic acid or sulfonic acid ion-exchange groups. This column was specifically developed to enable sodium/ammonium quantitation at very diverse concentration ratios of these two. Because of the high selectivity of this column for potassium, it elutes after the other five common cations (lithium, sodium, ammonium, magnesium and calcium), and this makes it useful for sample matrices with high potassium. Ethanolammonium and ammonium are well resolved in this new column with an isocratic eluent.

With the use of hydroxylamine in the eluent, the CS15 can also be used as a high sample throughput column. With a similar short analysis time, the CS15 actually provides better resolution among the six common cations than the CS12A, but it should be noted that the suppressor has to be used in the external water mode and that the noise is about five times higher. For the isocratic separation of the Group I and Group II cations, the CS12A is the column of choice.

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