

Tunable separation of anions and cations by column switching in ion chromatography

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Abstract

A convenient ion chromatography method has been proposed for the routine and simple determination of anions (Cl^- , SO_4^{2-} and NO_3^-) and/or cations (Na^+ , NH_4^+ , K^+ , Mg^{2+} and Ca^{2+}) using a single pump, a single eluent and a single detector. The present system used cation-exchange and anion-exchange columns connected in series via two 6-port switching valves or a single 10-port valve. The connection order of the ion-exchange columns could be varied by switching the valve(s). The present system therefore allowed the separation of either cations or anions in a single chromatographic run. While one ion-exchange column is being operated, the other ion-exchange column is being conditioned, i.e., the columns are always ready for analysis at any time. When 2.4 mM 5-sulfosalicylic acid was used as the eluent, the three anions and the five cations could be separated on the anion-exchange column and cation-exchange column, respectively. In order to obtain the separations of the target ions, the injection valve was placed between the two columns. Complete separations of the above anions or cations were demonstrated within 10 min each. The detection limits at $S/N = 3$ were 19–50 ppb ($\mu\text{g/l}$) for cations and 10–14 ppb for anions. The relative standard deviations of the analyte ions were less than 1.1, 2.9 and 2.8% for retention time, peak area and peak height, respectively. This proposed technique was applied to the determination of common anions and cations in river water samples.

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1. Introduction

Ion chromatography (IC) has become a mature analytical method since its introduction by Small et al. [1] owing to its wide applicability to the determination of ionic species in various water samples. Regularly, anions and cations are independently separated by using suppressed IC methods and nowadays, a great number of methods have been intimately developed for the simultaneous determination of anions and cations in a single chromatographic run, leading to achievement of more convenient, more simple and less solvent consuming systems [2–25].

There are two approaches currently known for the simultaneous determination of anions and cations in a single chromatographic run. The first approach uses a single column for this purpose based on chelation of cations to form anions [3–5], use of zwitterionic ion-exchangers [6–9] or mixed-bed ion-exchange

columns [10], etc. However, these methods allow partially simultaneous separation of anions and cations. An alternative method involves mixed-mode separation such as ion-exclusion chromatography and cation-exchange chromatography [11–15]. This system provides a simple, rapid determination means for inorganic common ions. However, it is ineffective for a wide variety of anions contained in samples and an appropriate eluent should also be carefully selected.

The second approach uses two columns connected in series or parallel with or without column switching. For the approach without column switching [16–18], a suitable eluent should be chosen for the simultaneous separation of both anions and cations, but these peaks sometimes overlap and the method is not applicable widely. The determination of these common ions can be also achieved on short permanently coated silica monolithic anion-exchange and cation-exchange columns [19]. The both columns are connected in parallel arrangement. However, common monovalent cations such as Na^+ , NH_4^+ and K^+ could not be separated when these ions contained in real samples.

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Two separation columns (cation-exchange and anion-exchange columns) can be connected in series via a single 6-port switching valve [20,21] or two switching valves [22]. We also developed separation systems using two 6-port switching valves for the determination of anions and cations and applied the developed system to the determination of ions in tap, river and pond water samples [23,24]. In our developed system named peak parking technique, cation-exchange and anion-exchange columns were connected in series via two 6-port switching valves. However, the above methods required accurate column-switching timing in order to avoid peak overlaps.

Yokoyama et al. [25] have also described a separation method using switching valves for the separation of anions and cations using cation-exchange and anion-exchange columns. They accumulated temporarily anions in loop while the cation IC was performed. Subsequently, the accumulated fraction was introduced into the anion-exchange column and then determined.

As a competitive practical method, this paper presents a convenient ion chromatography method to determine either of anions or cations in a single system. The method comprises one pump, one injection valve, an anion-exchange column, a cation-exchange column, two 6-port switching valves or a single 10-port switching valve and a conductivity detector. This method is easy and simple to operate compared with our previous method [23,24]. In previously method, good resolution of cations and anions was achieved when the peak parking technique was performed. However, the column switching timing had to be focused to control accurately in order to trap analyte anion on the anion-exchange column, i.e., to avoid peak overlaps.

In the present method, while one ion-exchange column is being operated, the other ion-exchange column is being conditioned. In other words, both the columns are being conditioned with the eluent and are possible to use at any time. Furthermore, column switching is needed only when the required separation will be changed. On that purpose, the two ion-exchange columns are then connected in series via two 6-port switching valves or a single 10-port switching valve while the injection valve is placed between the two columns to allow separating the target ion. The optimized operating system was then applied to the determination of anions and cations in river water samples.

2. Experimental

2.1. Apparatus

The diagrams of the apparatus employed in this study are illustrated in Fig. 1a and b. The ion chromatograph consisted of a PU-2080i plus HPLC pump (Jasco, Tokyo, Japan), a Rheodyne 5095 injector equipped with a 20 μ l sample loop (Cotati, CA, USA), a CM-8020 conductivity detector (Tosoh, Tokyo, Japan), two Model 7000 6-port switching valves (Rheodyne) or a Model 7610-600 10-port switching valve (Rheodyne) and a Computer Aided Chromatography data processor (Nippon Filcon, Tokyo, Japan). The columns employed were a TSK_{gel} Super-IC-Cation

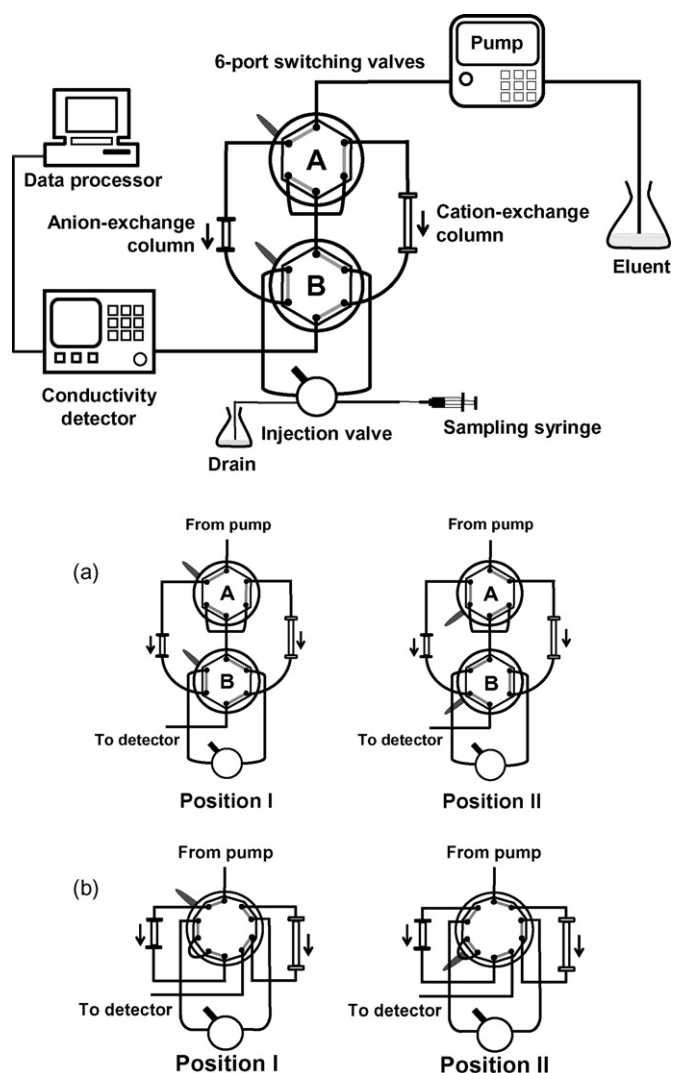


Fig. 1. Schematic diagrams of the instruments and the positions of the switching valve(s) for (a) two 6-port switching valves and (b) single 10-port switching valve. The injection valve was placed between anion-exchange and cation-exchange columns, which allowed the tuning of either cations separation or anions separation. Separation of cations (position I) and separation of anions (position II).

column (150 mm \times 4.6 mm i.d.) and a TSK_{gel} IC-Anion-PW_{XL} column (75 mm \times 4.6 mm i.d.). Both columns were obtained from Tosoh (Tokyo, Japan).

2.2. Reagents, standards and sampling

Analytical reagent grade chemicals were used and purchased from Nacalai Tesque (Kyoto, Japan), unless otherwise noted. Purified water was prepared in the laboratory by using a GS-590 water distillation system (Advantec, Tokyo, Japan). A single stock standard solution containing a mixture of all interest ions was prepared by dissolving 0.2 mM of each NaCl, CaCl₂, MgSO₄, NH₄NO₃ and KNO₃. All standard solutions were stored in polyethylene containers and kept under refrigeration at 4 °C. 5-Sulfosalicylic acid was obtained from Wako (Osaka, Japan) and used for the eluent.

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