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Compact, field-portable capillary ion chromatograph

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Abstract

We describe a fully computer controlled, field-portable capillary scale suppressed conductometric ion chromatograph that fits in a standard briefcase (28×43×15 cm) and weighs 10 kg. The system uses an electrodialytic sodium hydroxide generator on the high-pressure side of a syringe pump. This provides isocratic or gradient operation with excellent eluent purity. A 100 nl loop-volume, electrically actuated valve is used for sample injection and allows for facile automation. Analytical columns, 50 cm×180 μm I.D., were packed in the laboratory using commercial packing and fused-silica capillaries. A novel flow-through conductivity detection cell was developed. The system provides detection limits in the sub- to low ppb range, with mass limits of detection >100 times better than standard IC systems. Sample preconcentration is demonstrated with a concentrator column, 10 cm in length. © 1998 Elsevier Science B.V.

Keywords: Instrumentation; Ion chromatography; Eluent generation, electrodialytic; Environmental analysis; Inorganic anions; Organic acids

1. Introduction

The need for on-site characterization has become important in diverse areas, from pristine National Parks to hazardous waste disposal sites, and has led to a considerable interest in the development of self-contained field-portable instrumentation. Presently, two factors limit the use of field portable instruments for environmental analysis. First, most portable instruments do not compare favorably to laboratory-based instruments with respect to reliability and performance. Second, the availability of self-contained field-portable equipment is largely limited to chemical analyzers that measure a single physicochemical property such as pH, temperature, or UV–Vis absorbance, although more sophisticated instruments such as X-ray fluorescence analyzers, mass spectrometers, and Fourier transform IR systems have recently been developed [1]. Much hope is held for the prospects of on-chip separation and analysis systems; it is not clear when such devices would be practical such that they can be operated without the need for large bench top lasers. Bringing samples collected in the field back to the laboratory for analysis results in a time lag that can compromise sample integrity as well as delay any needed response prompted by the analytical result. This can be a critical issue where human health is concerned.

Analysis often requires the separation of multiple analyte species before detection and quantitation. For the environmental analyst, liquid chromatography (LC) and gas chromatography (GC) remain the primary techniques of choice. Although field portable GC systems have been commercially available for some time, field portable LC systems are virtually non-existent. This is at least partly due to the

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bulky hardware used in LC. UV–Vis spectrometers, most commonly used for detection in LC, are particularly large. If used in the field, LC systems typically have to be located in a mobile laboratory, making them at best only moderately portable.

The practice of capillary LC has undergone extensive development since its introduction twenty years ago [2]. There are many advantages of moving from conventional size (≥4 mm I.D.) columns to the capillary domain: higher efficiencies, better mass limits of detection (MLODs), low eluent consumption, and a very small sample requirement. LC hardware components other than the absorbance detector have been miniaturized along with the commercial development of capillary columns. Fully automatable injection valves are available down to 20 nl loop volumes; small, compact, and inexpensive syringe pumps with very low power requirements have been developed.

Ion chromatography (IC) is a well-established LC technique for the separation of small ions. Recently, we described a capillary ion chromatograph with suppressed conductometric detection for anion analysis for use in the laboratory [3]. Employing an on-line electrodialytic NaOH generator capable of withstanding high pressures for eluent production, the system provides significantly better peak efficiencies compared to similar conventional size columns operated under the same chromatographic conditions. Unlike the absorbance detectors used in capillary LC, conductivity detectors (including the electronics) can be miniaturized without sacrificing performance. This feature of conductance detection, in combination with the excellent performance of the laboratory scale capillary IC, led us to investigate the feasibility of a field portable capillary IC system.

In this paper, we present a fully computer controlled, field portable capillary IC system. Further, we describe a novel, robust, easy to fabricate, adjustable cell constant conductivity cell for capillary systems. The construction and performance of the field portable IC system is reported.

2. Experimental

2.1. System layout

The layout of the portable IC system is shown in Fig. 1. The briefcase and instrumental components, excluding the laptop computer, weighed 10 kg. The instrument case was constructed in the laboratory from aluminum sheets. Fully assembled, the closed case measured (28×43×15 cm). The top part is 4 cm deep; the bottom part is 11 cm deep, including a concealed 2.5 cm deep undercarriage (not shown in Fig. 1) that provided an easily accessible housing for electrical wiring and some of the electronics. Holes of appropriate size were drilled in the chassis to hold components in their proper places. As necessary, aluminum L-brackets were affixed to the chassis or small notches were made to serve as braces to secure components in place.

The basic instrument configuration is similar to that presented previously [3]; only a brief description is given here. The pump is a fully computer controlled 48 000 step, motor driven syringe type dispenser (Model 50300, Kloehn, Reno, NV, USA) equipped with a 500 μl syringe. A Kynar block was

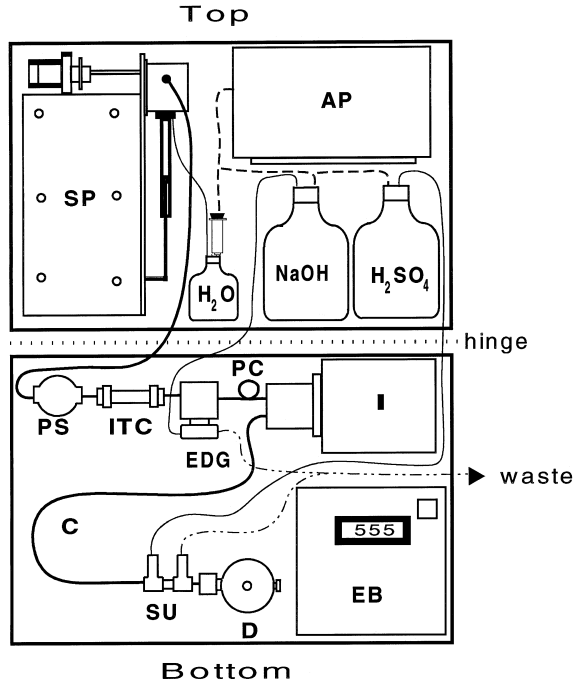

Fig. 1. Schematic layout of the portable IC system. Figure designations: SP, syringe pump; AP, air pressure pump; PS, pressure sensor; ITC, ion trap column; EDG, electrodialytic sodium hydroxide generator; PC, polystyrene capillary; I, motorized injector; C, capillary column; SU, chemical suppressor; D, detector; EB, electronics box.

machined in the laboratory with appropriately sized ports to accommodate the pump head, a low leak dual ball and seat inlet check valve (P/N 44541, Dionex, Sunnyvale, CA, USA), and a liquid output port. A small volume reservoir (~25 ml) of deionized water was affixed next to the syringe dispenser on the top portion of the briefcase. The water reservoir was connected to the inlet check valve using 0.5 mm I.D.×1.5 mm O.D. polyether ether ketone (PEEK) tubing to avoid intrusion of CO_2 . The water reservoir was pressurized by air pump AP (see below). A Tygon tubing sleeve was placed around the glass syringe as a protective measure should the syringe shatter due to high pressure. A pressure sensor (Model SP70-A3000, Senso-Metrics, Simi Valley, CA, USA) was connected to the liquid output port of the Kynar block using 0.25 mm I.D.×1.5 mm O.D. PEEK tubing. The system pressure was continuously monitored to insure proper system performance. Signal processing circuitry for the pressure sensor was built the laboratory and housed in an electronics box. A digital panel meter (Jewell Electrical Instruments, Manchester, NH, USA) was affixed to the top of the electronics box to provide the pressure readout.

An impurity trap column was connected at the exit of the pressure sensor using 75 μm I.D.×365 μm O.D. fused-silica capillary tubing (Polymicro Technologies, Phoenix, AZ, USA). The trap column was packed with a chelating metal resin (Chelex 100, sodium form, Bio-Rad, Richmond, CA, USA) in the entrance half and a mixed bed ion-exchange resin (Amberlite MB-1, Sigma, St. Louis, MO, USA) in the exit half. This served to remove any metal ions, silicate and other ions leached from glass and metal contact parts in the upstream components.

A previously described [3] microscale electrodialytic sodium hydroxide generator (EDG) was used for eluent production. A 350-ml capacity reservoir, typically containing ca. 25 m*M* sodium hydroxide, was used to supply the donor solution for the EDG. The reservoir was mildly pressurized (<1 p.s.i.; 1 p.s.i.=6894.76 Pa) with an aquarium style diaphragm type air pump (AP) to produce a small flow-rate of donor NaOH through the EDG. Beyond supplying an adequate amount of donor sodium ions, the flow-rate of the feed solution was not critical. Due to the distension of the membrane in the EDG, this donor flow was somewhat dependent on the pressure drop on the column. A 10 cm long polystyrene capillary, ~80 μm I.D.×250 μm O.D., was placed at the exit of the EDG to remove the H_2 gas in the eluent stream by permeation through this tube. The polystyrene capillary was able to perform gas removal at pressures >900 p.s.i. at NaOH concentrations >40 m*M*. The exit of the polystyrene capillary was connected to an electrically actuated 4-port injection valve equipped with a 100 nl internal sample loop (Cheminert model C4-1044-.1EH, Valco Instruments, Houston, TX, USA).

Initial experiments were carried out to conductometrically measure the output NaOH concentration for a given current flowing through the EDG. These results showed that the EDG operates virtually with a 100% current efficiency. Henceforth, arrangements for conductometric measurement of the eluent concentration were omitted and the NaOH concentration (in m*M*) by the EDG was computed from:

$$\text{NaOH (m}M) = 0.6218 \times \text{EDG Current } (\mu\text{A}) / \text{flow-rate } (\mu\text{L min}^{-1}) \quad (1)$$

Analytical columns, ~50 cm×180 μm I.D.×365 μm O.D. fused-silica capillaries (Polymicro Technologies), were packed in the laboratory with AS-11 packing material from Dionex, using a capillary version of the proprietary packing technique provided by Dionex. The frit at the exit of the column was made by packing short pieces of glass wool into a 0.3 mm I.D. (1.5 mm O.D., ~1 cm long) PTFE tubing and push fitting this into the end of the column followed by push fitting a 75 μm I.D.×365 μm O.D. fused-silica capillary on the other side of the PTFE tube to confine the glass wool bed. No entrance frit was used; this allowed the top of the column to be easily trimmed when packing material was compressed over a period of use and a void developed.

For experiments using sample preconcentration, the preconcentration column consisted of an ~10 cm length of 180 μm I.D.×365 μm O.D. fused-silica tube packed with AS-11 packing. The exit frit was constructed by first pushing a small piece of glass fiber filter (Whatman, type GF/A) ~1 cm from the end of the preconcentrator. A 50 μm I.D.×150 μm O.D. fused-silica capillary was then pushed inside the larger capillary against the glass fiber filter and epoxied into place. In our experiments, the pre-

concentration column was connected as a loop in the injector and the mode of connection was such that the flow direction remained the same in both load and inject modes. This obviated the necessity of an inlet frit. The exit frit was able to withstand pressures at least to 900 p.s.i., the maximum pressure used in this work. The electrically actuated sample injector was equipped with a six port valve (Cheminert model C3-2346EH, Valco), having internal dead volumes of 110 nl in each port, to accommodate the preconcentrator.

A previously described, hollow fiber suppressor [3] was used prior to detection. A ~50 μm I.D.×200 μm O.D. radiation grafted, sulfonated PTFE membrane tube [4] (courtesy of Dionex), having an active length of 1.9 cm, was used to construct the suppressor. A 350-ml reservoir of 2.5 m*M* H_2SO_4 was modestly pressurized by the same air pump AP used to pressurize the NaOH reservoir and the liquid outlet was connected to the suppressor jacket using Tygon tubing. The suppressor regenerant flow was controlled by an adjustable compression type clamp and was typically maintained at ≤ 0.25 ml min^{-1}. At an eluent flow-rate of 1.5 μl min^{-1}, the device was able to suppress 0.5–40 m*M* NaOH to a background of ~1 μS cm^{-1}.

2.2. Conductivity cell

A novel conductivity cell was connected at the suppressor exit. The conductivity cell is shown schematically in Fig. 2. A Kel-F block was machined with ports to accommodate a $\frac{1}{4}$-28 NF threaded fitting at the inlet and a No. 1-72 screw at the outlet. In addition, a hole was machined vertically through the center of the block to provide a waste outlet for

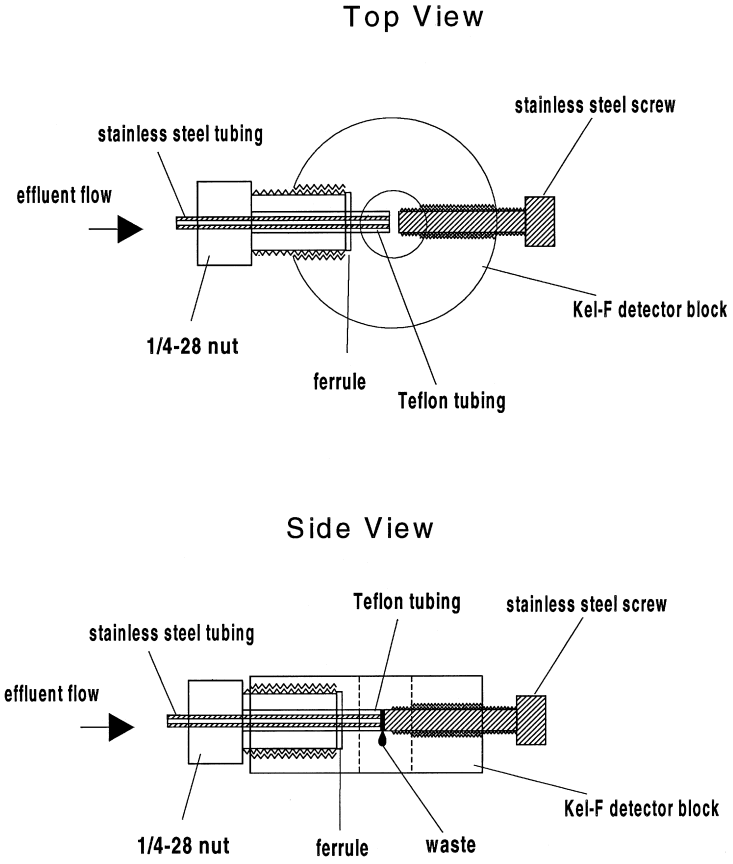

Fig. 2. Design of the conductivity detector cell. The cell is placed on sheets of absorbent paper, these suffice to take up the waste.

the column effluent as well as for visual inspection at any time. The entrance electrode was a 31 mm × 0.152 mm I.D. × 0.304 mm O.D. (P/N O-HTX-30, Small Parts, Miami Lakes, FL, USA), stainless steel (SS) tube. Both ends of the SS tubing were first sanded flat. A ferrule and $\frac{1}{4}$-28 NF threaded nut were affixed to one side of the stainless steel tube ~0.5 cm from the terminus. A small piece of 0.3 mm I.D., $\frac{1}{16}$ in. O.D. PTFE tubing was cut flat on both ends and pushed over the exposed SS tubing at this terminal end (1 in. = 2.54 cm). The protruding length of the SS tube was then forcibly pushed back such that it aligned evenly with the end of the PTFE tubing as shown in Fig. 2. The other electrode was a type 316 stainless steel screw. It has been our experience that the exact size of the screw (diameter and number of threads/unit length) is probably not critical, as screws in a wide size range have been successfully used with this detector design. The data presented in this paper were obtained with a No. 1-72 screw ($\frac{1}{2}$ in. long, P/N O-MX172-8, Small Parts). The terminal end of the SS screw was also sanded flat. The ends of the electrodes, both the SS tubing and SS screw forming the detection region of the cell, were hand polished using the nonabrasive side of 300-grit abrasive paper to a shiny finish to avoid grain boundary corrosion problems.

To complete detector construction, the entrance electrode assembly and the SS screw were threaded in their intended positions in the detector block. Using an ohmmeter that audibly indicates a short circuit, the SS screw was threaded in until it touched the entrance electrode. The screw was then turned in reverse just enough until there was no actual contact. In some experiments, the adjustment was done while a solution of known conductance was pumped through the detection cell connected to detection electronics for conductivity measurement. The screw was adjusted until a cell constant was obtained in the desired range. In use, the electrodes of the conductance cell were connected to a bipolar pulse conductance detection (BPCD) system [5], the relevant electronics were housed in box EB.

A portable personal computer-based data acquisition/instrument control system (Daqbook 100, Iotech, Cleveland, OH, USA) provided analog input/output to the BPCD system. Digital output from the Daqbook was used to control the status of the injection valve for system automation. The Daqbook was connected to the parallel port of an 80486 class laptop computer operating at 25 MHz (ThinkPad Model 360, IBM). An executable program written in C, provided a user interface for data acquisition and instrument control. The software enabled parameters to be set for operation of the bipolar pulse conductivity detector, and allowed the choice of the EDG current profile (isocratic or gradient) to be executed during a chromatographic run. The software numerically displayed the programmed current profile, the voltage required to obtain the current profile, and the suppressed conductance observed at the detector, in addition to a real time display of the chromatogram. Following a chromatographic run, the acquired data were analyzed using Microsoft Excel.

A 24 V d.c. power supply (Lambda Electronics, Melville, NY, USA) was used to power the pump, injection valve and pressure sensor. A d.c.–d.c. converter with 10 V output was built in the laboratory to provide the pressure sensor with the 10 V d.c. operating voltage it required. The bipolar pulse conductivity detector and data acquisition board were respectively equipped with 5 V d.c. and 15 V d.c. power supplies needed for their operation. Ports were machined in the undercarriage of the briefcase to provide plug-ins for the power cables and for an RS-232 cable that connects the pump to the laptop computer. Two additional cutouts were machined in the undercarriage to hold two thirty-seven pin male connectors (D-sub style). Corresponding female connectors were then used to interface the analog and digital input/output ports of the Daqbook to the BPCD electronics and the injection valve.

Analytical reagent grade chemicals were used throughout. Distilled deionized water was used to prepare solutions.

3. Results and discussion

3.1. Power requirements

From a practical view point, the power requirement of a field deployable instrument should be a minimum such that the system can run for a considerable amount of time on a battery pack of modest size. The power requirements for the present system

Table 1
Power requirements of the field portable capillary IC system

Component (operating voltage)	Operating current (mA)
Syringe pump (24 V d.c.)	400
Typical operation	400
Syringe refill (~10 s)	1500
Injection valve (24 V d.c.)	
Typical operation	60
Inject/load (10 ms)	2500
Data acquisition system (15 V d.c.)	510
Electronic circuitry	<10
Bipolar pulse detector (15 V d.c.)	
Pressure sensor (10 V d.c.)	

are summarized in Table 1. The power requirement for the laptop computer has not been considered in this listing. The primary power consumption is from the syringe pump and the data acquisition board. Under both idle and actual pumping conditions the power requirement of these components total ~17.5 W. During a syringe refill, a substantially larger amount of power is consumed, however, this occurs only for a very brief interval. Further, the volume of the syringe allowed continuous operation for several hours (typically >5) before a syringe refill was needed. The actual act of pumping does not significantly increase the power consumption; neglecting frictional losses, less than 0.2 mW is required to pump water at a flow-rate of 1.5 μl min^{-1} against a back-pressure of 800 p.s.i.. As shown in Table 1, the power consumption of the entire chromatographic system during typical operation is well below 20 W. Based on the power storage capacity of currently available Li-ion batteries, it should be easily possible to operate the portable IC system in a field setting for several hours without a major weight burden.

3.2. Conductance detector cell

The success of any chromatographic detector depends on its ability to provide sensitive detection without degrading the performance of the separation technique. Four different cell types, all of the same general design depicted in Fig. 2, were evaluated with respect to peak asymmetry (in this case a measure of the washout profile in the cell) and peak height reproducibility, as a function of the cell constant, using a flow injection configuration in which NaCl was injected into a water carrier stream. The detector cells differed only in the polymer tubing used to jacket the entrance electrode. The polymers used to jacket the entrance electrode were PTFE, PEEK, and sulfonated PEEK tubing (PEEK tubing treated with concentrated sulfuric acid at room temp for 1 h) and a detector cell without any polymer tubing to jacket the entrance electrode was also evaluated. The data are summarized in Table 2. Peak asymmetry was evaluated at 10% of the peak height. The washout profile of the detector cells constructed using a polymer tubing jacket over the entrance electrode were considerably better than those obtained using no jacket. Note that these results come from a flow injection experiment where the peak is intrinsically tailing and not Gaussian. The

Table 2
Performance of conductivity detector cells

Covering material	Cell constant (cm^{-1})	Average peak assymetry	R.S.D. (%)	
			peak assymetry	peak height
PTFE	3.6	1.43	2.79	4.2
	4.2	1.42	3.98	2.0
	8.6	1.35	1.07	0.6
PEEK	3.9	1.34	2.37	3.0
	4.9	1.43	3.90	5.4
	5.6	1.42	4.29	6.3
Sulfonated PEEK	2.5	1.55	4.80	2.1
	3.6	1.50	7.37	3.1
	4.3	1.55	1.43	2.8
No covering	2.2	2.29	26.20	1.9
	2.7	1.62	18.10	10.0

detector cell without a polymer jacket performed the worst, with a high and variable degree of tailing. Drops that formed between the electrodes of the detection cell likely caused this behavior. The drops formed a mixing pool of temporally variable size and resulted in peaks that severely tailed at times, depending on the volume of the drop(s) formed between the electrodes. The polymer jacket tube facilitated the removal of the flowing liquid from the detection zone. The circular geometry of the entrance electrode/jacket removed the effluent efficiently in all directions away from the electrodes, hence better peak shapes resulted. Possibly due to its high hydrophobicity, the cell with the PTFE jacketed entrance electrode showed the most efficient and uniform effluent removal and yielded better reproducibility compared to the cells made with PEEK and sulfonated PEEK tube jackets. Within each type, there was a marginal increase in the S/N value with increasing cell constant (e.g., 30% increase in S/N in going from a cell constant of 3.6 to a cell constant of 8.6 for the cells with the PTFE jacketed entrance electrodes), except for the cells with the PEEK jacketed entrance electrodes. For the latter, an opposite trend (but of an even smaller magnitude) was observed. The absolute S/N value was essentially the same for the entrance electrodes with PTFE or sulfonated PEEK jackets but that for the PEEK jackets was about a factor of $2.5-3\times$ better at comparable cell constant values. Nevertheless, cells

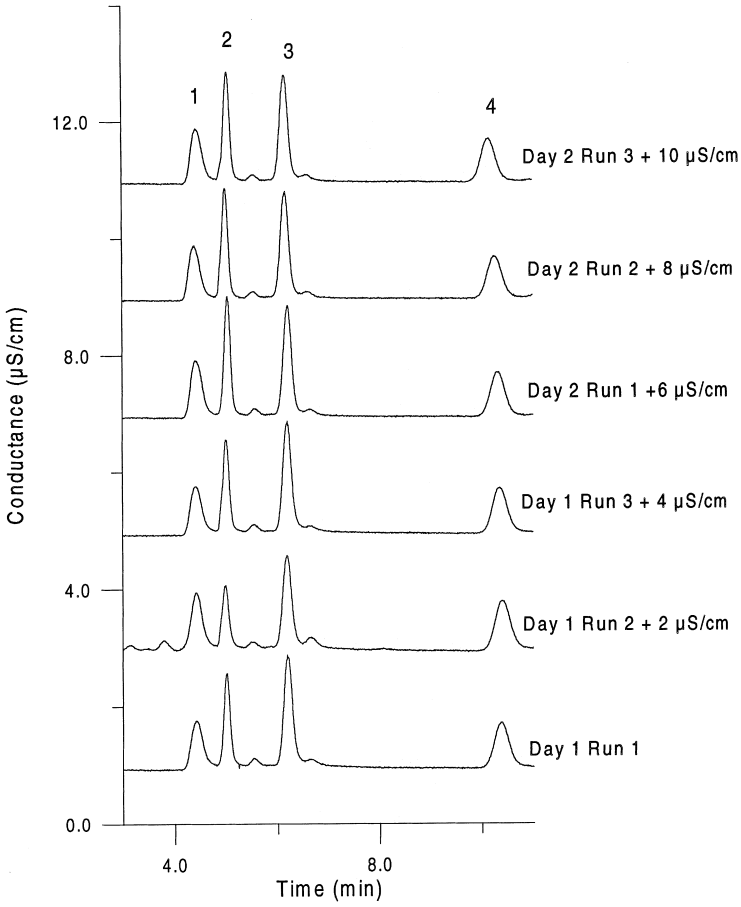

Fig. 3. Day-to-day system reproducibility; repeated 100 nl injections with ~20 mM NaOH eluent. Injected concentration was 20 µM for each ion. Peaks: 1, fluoride; 2, chloride; 3, sulfate; 4, phthalate.

with the PTFE jacketed electrodes were the easiest to construct and were henceforth used in all further work.

An experiment was conducted to determine how well the cell constants could be reproduced in such cells with adjustable electrode separation. In ten separate attempts, a mean cell constant of 3.6 was achieved with a 0.34% relative standard deviation (R.S.D.) and similarly, a cell constant of 4.9 was reproduced with a 0.36% R.S.D..

3.3. System performance

The day-to-day reproducibility of the portable IC system is shown in Fig. 3 for repeated injections of fluoride, chloride, sulfate, and phthalate. The chromatograms were obtained under isocratic conditions using an ~20 m*M* NaOH eluent at a flow-rate of 1.5 μl min^{-1}. The relative standard deviation of retention times ranged from 0.1% to 0.7% within one day and 0.3% to 0.8% day-to-day. Peak efficiencies for

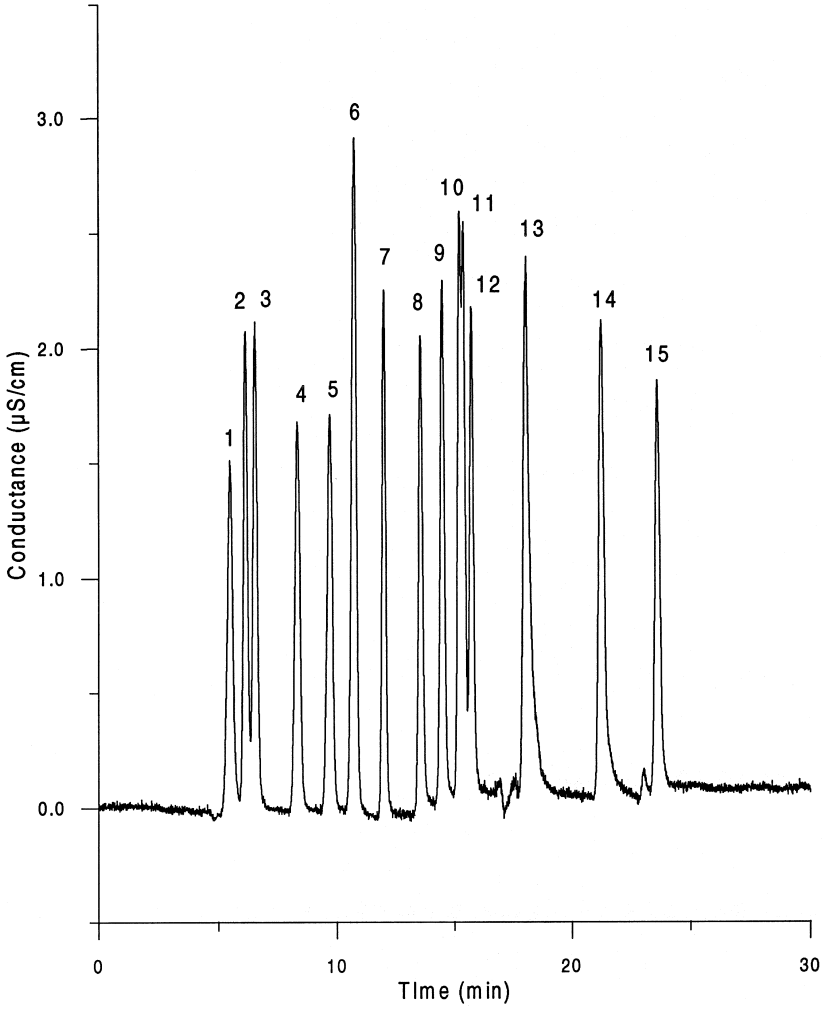

Fig. 4. Background subtracted gradient chromatogram. A linear gradient from 2 m*M* NaOH to 38 m*M* NaOH from 5 to 17 min was used. Peak identities: 1, acetate; 2, formate; 3, methanesulfonate; 4, monochloroacetate; 5, bromate; 6, chloride; 7, nitrite; 8, trifluoroacetate; 9, dichloroacetate; 10, bromide; 11, nitrate; 12, chlorate; 13, sulfate; 14, phthalate; 15, chromate. All ions were 50 μ*M* except dichloroacetate which was 60 μ*M*.

chloride, sulfate, and phthalate were 27 133; 21 018; and 15 422 plates m^{-1}, respectively. The efficiency of fluoride was not calculated due to its proximity to the void volume. Injected at this nominally sub ppm concentration, the chloride peak varies more from injection to injection than the other analytes, possibly due to contamination problems.

Response linearity was studied under the same chromatographic conditions as above. A sample solution containing chloride, sulfate, and phthalate over a concentration range of 10–200 μ*M* was used. Linear r^2 values for peak area response vs. injected concentration for chloride, sulfate, and phthalate were 0.9959, 0.9988, and 0.9974, respectively. Above a concentration of 200 μ*M*, peak broadening resulted from column overloading.

MLODs were also determined for each of the analytes in this three-constituent mixture under the same isocratic conditions. The intrinsic electronic noise of our bipolar pulse detector electronics as measured with a dummy resistor was 0.3–0.4 nS cm^{-1}. This increased to 2–3 nS cm^{-1} during chromatography, regardless of NaOH concentration or flow-rate. Based on the performance at an injected concentration level where baseline noise could be readily evaluated and determining the LOD on the basis of three times the peak-to-peak noise level, the MLOD in terms of mass in grams and moles and the concentration LOD in molar units for the three anions were as follows: chloride: 106 fg, 3 fmol, 30 n*M*; sulfate: 1.15 pg, 12 fmol, 120 n*M*; and phthalate 4.1 pg, 25 fmol and 250 n*M*. The concentration LODs are comparable to presently available commercial bench top IC systems but the MLODs are better by >2 orders of magnitude.

3.4. Gradient chromatography

Incorporation of the EDG on the high-pressure side of the pump allows gradient chromatography to be easily performed. The lag time, or time required for the produced NaOH to reach the head of the column from the EDG, was measured to be 1.5 min. Therefore, only a short time is needed for a specific programmed NaOH concentration to reach the head of the column. A gradient chromatogram of a sample containing 15 anions is shown in Fig. 4. A linear gradient of ~2 m*M* to 38 m*M* NaOH from 5 min to 17 min was used for the separation. This corresponded to a current requirement of 5–95 μA using a water flow-rate of 1.5 μl min^{-1}. The resulting separation was excellent. Peak efficiencies ranged from 10 648 plates m^{-1} for acetate to 240 152 plates m^{-1} for chromate, with an average of 80 000 plates m^{-1} being observed for the separation.

3.5. Sample preconcentration

Many samples require preconcentration before the analytes can be determined. We therefore investigated the use of a small precolumn in the place of the sample loop for preconcentrating samples before the valve was switched to the inject mode. In the present work, we chose a valve configuration that leads to unidirectional pumping during the load and inject modes since this obviated the need for an inlet frit for the precolumn.

Since surface area to volume ratios are much greater for capillary systems relative to macrobore systems, preconcentration techniques are more difficult to practice. The danger of contamination during sample loading, whether originating from sample pumping, wetted materials in the valve or the

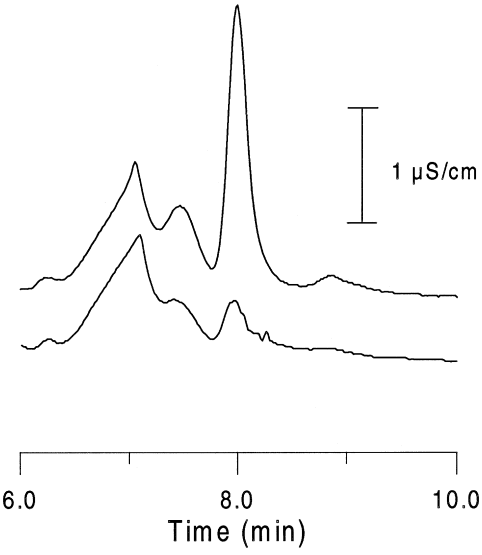

Fig. 5. Chromatograms with a 10 cm preconcentrator column: bottom, 18 μl water preconcentrated; top, 18 μl 100 n*M* sulfate solution preconcentrated. The sulfate peak appears at ~8 min. Eluent ~10 m*M* NaOH, flow-rate 1.7 μl min^{-1}.

precolumn material itself are substantially higher. While we have not as yet fully resolved these issues, preconcentration is clearly possible. Fig. 5 shows two chromatograms with sample preconcentration. In the bottom chromatogram, 18 μl of deionized water was preconcentrated at a rate of ~3 μl min^{-1} using a glass syringe. The top chromatogram was obtained under identical conditions except the water was spiked to contain 100 n*M* (ca. 10 ppb) sulfate. The first two peaks (at ca. 7 and 7.5 min) are due to carbon dioxide and an unknown impurity and the peak at ~8 min. is due to sulfate. The difference in the sulfate peak is obvious. The efficiency for the sulfate peak was observed to be 20 600 plates m^{-1} (isocratic elution with ~10 m*M* NaOH). In this type of experiment, the peak height reproducibility of precolumn loading of different analytes was found to range between 0.8 and 3.2%.

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