



Portable, fully autonomous, ion chromatography system for on-site analyses



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ARTICLE INFO

Article history:

Received 20 March 2014

Received in revised form 7 May 2014

Accepted 18 May 2014

Available online 27 May 2014

Keywords:

Ion chromatography
Environmental analysis
Mobile laboratory
Portable instrumentation
Eluent reflux

ABSTRACT

The basic operating principles of a portable, fully autonomous, ion chromatography system are described. The system affords the user the ability to collect and analyze samples continuously for 27 days, or about 1930 injections before needing any user intervention. Within the 13 kg system, is a fully computer controlled autosampling, chromatography and data acquisition system. An eluent reflux device (ERD), which integrates eluent suppression and generation in a single multi-chambered device, is used to minimize eluent consumption. During operation, about 1 μL of water per minute is lost to waste while operating standard-bore chromatography at 0.5 mL min^{-1} due to eluent refluxing. Over the course of 27 days, about 100 mL of rinse water is consumed, effectively eliminating waste production. Data showing the reproducibility (below 1% relative standard deviation over 14 days) of the device is also presented. Chromatographic analyses of common anions (Cl^- , NO_3^- , SO_4^{2-} , PO_4^{3-}), is accomplished in under 15 min using a low backpressure guard column with ~ 25 mM KOH isocratic elution. For detection, a small capacitively-coupled contactless conductivity detector (C4D) is employed, able to report analytes in the sub to low micromolar range. Preconcentration of the injected samples gives a 50-fold decrease in detection limits, primarily utilized for in-situ detection of phosphate ($\text{LOQ } 10 \mu\text{g L}^{-1}$). Field analyses are shown for multiple on-site analyses of stream water indifferent weather conditions.

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1. Introduction

Developments in analytical instrumentation have seen the focus of measurement and analyses shift from the laboratory toward the field. The development of new, more versatile electronics and highly efficient components has been the driving force behind this trend [1,2]. Automated instruments in the field facilitate the ability to provide more reliable results with higher temporal resolution and reduced operating costs [3,4]. Most field instruments are designed to have sensitivities analogous to bench top instruments; yet, they are often smaller, more robust and easier to operate [3–5].

In environmental analyses, the demand for data collection over large areas and ranges of time, make autonomous instrumentation particularly favorable. The foremost problem with conventional analyses is the lag period between sample collection and analysis, which can often be problematic. Sampling on site and in real time is particularly beneficial in cases where product reliability or site conditions are the first priority [6]. In such cases, the advantages

of autonomous field instruments become quite evident. This has given rise to a variety of automated, field portable devices.

One method, which has shown potential for ion analyses, is ion chromatography (IC). Several portable ICs have been reported demonstrating sensitivity levels necessary for environmental monitoring [7–10]. While many laboratory applications of IC have been described, relatively few field applications exist. Difficulties such as limited eluent capacity, high-energy requirements and cumbersome user involvement have significantly hindered the development of portable ICs. Since the first discussion of a portable IC [9], several approaches have been presented, which have discussed methods to increased IC portability [10–12].

When considering the portability of a field instrument, importance is placed on robustness as well as the extent of necessary user input. Of the previously described portable ICs, few discuss the inclusion of an autosampler [8]. Sample pretreatment prior to injection is both common and important in IC. In order to reduce weight, many of these systems have omitted autosampling equipment as well as data processing and storage electronics. Though eliminating these factors contributes to the compactness of the IC instrument, it also increases the need for user input. This can be undesirable in many monitoring regimes, especially those requiring continuous analyses.

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Developments in eluent reuse technologies, automation techniques and solar and battery technology have renewed the appeal of a portable IC system [2,13–15]. Together, these improvements have been successful in reducing user involvement and maintenance intervals while preserving device portability. Eluent reflux [16,17] has provided IC systems with a method to operate a standard-bore system while only producing water waste on a capillary system scale. Consequently, IC can be operated for extended periods while effectively eliminating the generation of chromatographic waste.

In addition to eluent reflux, automatic sampling and data collection has also reduced user involvement [2,18–20]. A computer controlled autosampler, injection system, and data recorder can support all functions, which previously required user assistance. This ability to sample continuously is especially important in environmental monitoring where variation between significant events can be unknown [3–6,21].

Increased instrument portability has also been aided by advancements in photovoltaic (PV) and battery technologies [22–26]. Together, these two principles have substantially reduced the need for access to grid power. These contributions in energy technology as well as eluent reuse have enabled the mobilization of a full IC system from the laboratory to the field. The physical, operational, and chromatographic properties of a completely autonomous field portable ion chromatography system are described in this paper.

2. Experimental

2.1. Physical instrument description

The total weight of the portable IC device used in this study weighed 27.5 kg including the battery and PV cell. The mounting surfaces were fabricated from aluminum sheets, which were mounted into a plastic case measuring 62 × 48 × 30 cm (Southwest Public Safety, San Antonio, TX). All of the components were mounted into place using screws and aluminum mounting brackets. For the energy source, a 12 V 40 A h lithium ion battery (Smart Battery LLC., Tampa, FL) weighing 7 kg was used. The battery was combined with an 80 W polycrystalline solar panel (SolarTech Power Inc., Ontario, CA) weighing 7.5 kg. Together, the battery and PV cell provided enough energy to continuously power the system. When grid supplied power was available, a 40 W switch mode power supply (XP Power LLC., Sunnyvale, CA) was used to operate the device. This reduced the total weight of the device to 13 kg. A schematic of the chromatography system, including the autosampling system is shown in Fig. 1.

2.2. Eluent reuse

An eluent reflux device (ERD) was employed in the chromatographic system to recycle the eluent. This device described previously [16], uses multiple membrane separated resin chambers to integrate eluent suppression and generation into a single device. The ERD was capable of continuously generating and suppressing isocratic eluents, while generating virtually no chromatographic waste. By using recovered eluent in the analytical stream, a small volume of eluent could be reused multiple times over. With an ERD, the only source of eluent loss occurred during loop loading where the loop volume of KOH was lost to waste.

While the isolation of the electrodes provides the distinct advantage of a gas-free eluent, a separate reservoir must be used to provide an ion source to the electrodes. A small diaphragm pump, shown in Fig. 1, (KNF Neuberger AB, Stockholm, Sweden) was used to circulate de-ionized (DI) water from the electrode rinse reservoir through the electrode chambers at a rate of 0.5 mL min⁻¹.

Reservoirs containing 100 mL of ~25 mM KOH and 100 mL of DI water were used. The ERD was operated at a current of 28.5 mA in order to fully suppress the KOH eluent. At this current, water was added to the system via electro dialysis at a rate of 1.4 mL day⁻¹. To counteract the effect of eluent dilution from electro dialysis, a 3-port valve (DRV in Fig. 1) was placed at the conductivity cell outlet to remove water from electro dialytic addition. Assuming an injection volume of 23 μL and 4 injections per hour continuously, the instrument could operate for 27 days before reservoir refilling was necessary.

2.3. Flow arrangement and separation system

A mighty-mini pump (Scientific Systems Inc., State College, PA) was used to pump ~25 mM KOH eluent at a rate of 0.5 mL min⁻¹. In addition to the pump, a PEEK LO-Pulse, pulse damper (Scientific Systems Inc., State College, PA) was plumbed after the pump to remove pump pulsation. Eluent flow was directed from the pulse damper into an electrolytic eluent polisher described in [16], to remove ambient carbonate and other anions from the eluent stream (EEP as seen in Fig. 1). The EEP was used to strip anionic contaminants from the eluent, and continuously regenerated by the influx of hydroxide ions from the cathode. The polished (electrolytically purified), eluent then entered a 6-port, high-pressure injection valve (VICI Valco Instruments Co. Inc., Houston, TX).

From the valve outlet, flow was directed into a 4 × 50 mm AG15 column (Thermo-Fisher Dionex, Sunnyvale, CA). A guard column, rather than a standard analytical column was used to lower the back-pressure against the pump while still providing adequate analyte separations. The eluent then entered the ERD device [16], suppressing the eluent before the conductivity cell. After the suppressed eluent was measured, the flow was directed to a low pressure 3-port solenoid isolation or dilution relief valve (DRV in Fig. 1) (Bio-Chem Fluidics, Boonton, NJ), followed by an ATC-HC anion trap column (Thermo-Fisher Dionex, Sunnyvale, CA). To maintain eluent purity, an anion trap column was placed after the 3-port valve, which removed analyte ions post-detection. The analyte-free water then re-entered the ERD reflux chamber where the eluent was regenerated. The regenerated eluent then returned to the reservoir bottle for reuse.

2.4. Autosampling system

Samples were collected using a NE-500 syringe pump (New Era Pump Systems, Inc., Farmingdale, NY). A schematic of the autosampling process is shown in Fig. 2. Negative pressure was applied to the syringe passing the sample through a 0.45 μm filter (Whatman Polydisc GW, Sigma-Aldrich Co., St. Louis, MO). Once enough filtered sample was collected, a 3-port valve (Bio-Chem Fluidics, Boonton, NJ) directed sample to the injection valve. Before loop loading occurred, the sample was first passed through a CTC-1, cation trap column (Thermo-Fisher Dionex, Sunnyvale, CA), and a HRP, organic trap column (Thermo-Fisher Dionex, Sunnyvale, CA) to remove any dissolved impurities. To reduce carryover, the injection valve remained in the normally closed position until the two trap columns were flushed with new sample. The injection valve then switched into the load position. A total of 1 mL of sample was loaded on an Ionswift MAC-200 monolith anion concentrator column (Thermo-Fisher Dionex, Sunnyvale, CA), with a 23 μL void volume. After loop loading, the 3-port valve switched back to normally open to collect the next sample.

2.5. Detection

The capacitively-coupled contactless conductivity detector (C4D) cell was constructed according to previous publications

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