

Automation of sample dilution and injection for the determination of anions by ion chromatography

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Abstract

An automated system is described for sample dilution and subsequent injection into an ion chromatograph for the determination of anions in process solution. The system is controlled by FlowTEK, a program developed at Mintek. Anions such as chloride, nitrate, and sulphate have been successfully diluted with a dialysis unit and determined by ion chromatography using this program. The concentration ranges of anions that can be diluted and determined are 10 to 2 000 mg/l for chloride, and 40 to 10 000 mg/l for both nitrate and sulphate.

Introduction

Automation, as defined by the International Union for Pure and Applied Chemistry (IUPAC), refers to "the use of combinations of mechanical and instrumental devices to replace, refine, extend or supplement human effort and facilities in the performance of a given process, in which at least one major operation is controlled without human intervention, by a feedback mechanism" (Stockwell, 1990). Although there are a number of factors that stimulate the need for automation in a chemical laboratory, a major motivation is one of cost-effectiveness. In clinical laboratories, automated analysers have been used with considerable success. Industrial laboratories are devoting much attention to automation at present. Automated methods of analysis, using flow-injection analysis (FIA) techniques for the determination of ions, have become established, and automated analysers, either commercial or constructed in-house, have become features in many analytical laboratories. However, generally only individual ions can be determined at one time.

Since its inception in 1975, ion chromatography (IC) (Small et al., 1975) has become a powerful tool for the simultaneous analysis of many different ions in one chromatographic run. Although the dilution of sample solutions containing macroamounts of anions such as chloride, nitrate, and sulphate is a time-consuming task, automatic dilution in IC has received little attention. The problem associated with this type of analysis is the need to automatically dilute macroamounts of anions to within the range required for the IC determination. Dialysis (Barnes, 1994; Hansen and Ruzicka, 1976; Martins et al., 1985; and Van Staden, 1986) has been used for the removal of interferents based on the selective permeation of solutes through a membrane i.e. as a separation method. The technique is also ideally suited to the dilution of either anions or cations (Hansen and Ruzicka, 1976; Martins et al., 1985; Van Staden, 1986). In principle, dialysis is a process that separates different solutes by means of their unequal transport rates through membranes. The driving force for dialysis is the concentration

gradient across the membrane. The use of suitable membranes in continuous flow systems has made it possible to selectively and reproducibly transfer the analyte from a relatively unknown sample stream into a carrier or collector stream of known composition. The reproducibility of sample transfer, provided all other parameters remain constant, can be utilised for the dilution of the samples (Martins et al., 1985; Van Staden, 1986).

FlowTEK (Marshall and Van Staden, 1992) software for analyser control developed at Mintek, is a program that is able to provide a flow-based analyser with microprocessor control and data acquisition capabilities. The program has numerous user-defined set-up options, and is relatively easy to use to improve data quality in automated systems.

In this investigation, a method for the automatic IC determination of chloride, nitrate, and sulphate, using the FlowTEK program, was developed. When dilution of macroamounts of anions was required, the use of a dialysis unit for sample dilution was investigated because of the ease of use, and the reproducibility and speed of solute transport. In addition, the feasibility of placing the dialysis unit ahead of the IC system was assessed in an attempt to avoid peak-broadening and consequently longer elution times. The removal of interferents prior to automatic injection was also considered.

Experimental

Apparatus

A schematic of the automated system is shown in Fig. 1. A Dionex Model 2000i ion chromatograph equipped with an air-actuated 8-port injection valve with 2 sample loops (7 and 50 µl), a low-capacity ion exchanger (AS4 separator column), and a Dionex micromembrane suppressor was used for the chromatographic determinations of the anions. A sample changer, equipped with 50 sample containers of 5 ml capacity, was connected to a Gilson Minipuls 2HP8 pump with Teflon tubing of 0.5 mm inner dia. When dilution was required, a dialysis unit containing a Technikon Autoanalyser Type C membrane was placed between the sample changer and the injection port of the IC system. The unit was constructed from 2 perspex blocks as described elsewhere (Barnes and Jones, 1989). An Epson PC, with the FlowTEK program installed and a colour monitor, was used to monitor the sequence of events.

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