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Note

Counter-flow source for isotachophoresis

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A counter flow of the leading electrolyte is used in isotachophoresis for improving the separation capacity by increasing the separation path[1–5] and for micropreparative flushing of particular separated fractions[6]. Also, focusing of zones or improvement of the detector response can be achieved by the application of a counter flow[7–9]. Likewise, a counter flow is useful in the continuous sampling technique[10], permitting analyses of low-concentration components by isotachophoresis. The flowrates used are low (usually 10^{-8}–10^{-7} l/sec) and it is necessary to regulate them precisely. Several types of mechanical pumps have been tried[2] and alternatively counter flows have been produced by a hydrostatic pressure[1,11] created by a controlled difference in the levels of electrolytes in the respective compartments of the isotachopherograph[2,12]. More recently, a pump was described in which the pressure of an electrolytically generated gas was transferred to the leading electrolyte by means of an elastic membrane[13].

In our laboratory, a counter-flow source containing no mechanical moving parts was constructed[14]. The operation of the pump is based on the utilization of electroosmosis, and its size, power and the possibility of accurate regulation make it suitable for use in isotachophoresis.

DESCRIPTION OF THE APPARATUS

The electroosmotic pump is illustrated schematically in Fig. 1. The electrodes (1 and 2) in the electrode compartments (3 and 4) are separated by using two membranes (5 and 6) made from different materials. The electrode compartments and the space between the membranes (7) are filled with a working liquid (= electroosmotically active solution). The pump produces a hydrodynamic flow as the result of the electro-

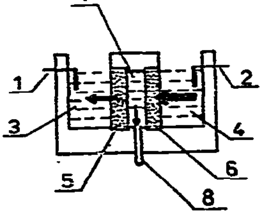

Fig. 1. Schematic diagram of the electroosmotic pump.

osmotic flow. If there is an electrokinetic potential between a membrane permeable to the working liquid and the working liquid then, after application of an external electric field, the working liquid starts to flow through the membrane. The resulting volume flow-rate out of the outlet tube (8) is given by the difference in the volume flow-rates through the individual membranes. Therefore, membranes made from electrokinetically different materials should be used.

In our laboratory, membranes with effective diameters of 6 mm, made from S 4 glass frit (Kavalier Sázava, Sázava, Czechoslovakia) and cellulose acetate (Sartorius-Membranenfilter, Göttingen, G.F.R.) are currently used. Various solutions, *e.g.*, 0.1 m*M* hydrochloric acid and 0.1 m*M* sodium hydroxide solution, serve as the working liquid.

In Fig. 2, the output of the electroosmotic pump, *i.e.*, the volume flow-rate, *versus* the voltage applied is plotted. If the efficiency of the pump decreases after prolonged contact (approximately 2 days) of the working liquid with the membranes, it can be recovered by washing and drying the membranes. This effect is probably caused by the exchange reactions of ions on the surface of the glass frit and, consequently, changes in the zeta potential.

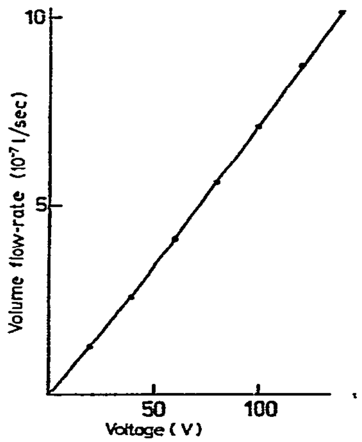

Fig. 2. Dependence of volume flow-rate on voltage for 0.1 *M* acetic acid.

Connection to isotachophoretic column

The pump is joined to an isotachophoretic column by means of a protective capillary, which prevents the working liquid from mixing with the contents of the column. The protective capillary is placed between two valves, as shown in Fig. 3. One valve connects the protective capillary with either the pump or a syringe and the other valve connects the protective capillary with the leading-electrolyte reservoir or with the isotachophoretic column. After an analysis has been completed, the protective capillary is rinsed and filled with the leading electrolyte (valve positions according to Fig. 3). By linking the column via the protective capillary to the isotachophoretic column, the counter-flow source is ready. The direction and rate of flow can easily be changed by changing the voltage or electric current. The pump is small and it can be built into or connected with existing isotachophoretic columns. The performance and pressure ranges in which the pump can operate depend on the material and cross-section of the membranes and on the nature of the working liquid used. The electric current and performance may fluctuate if the current density and thereby also heat generation increase in the membrane pores to such an extent that gas bubbles are produced. This phenomenon can be eliminated if the working liquid and its concentration and/or the membrane diameter are chosen properly.

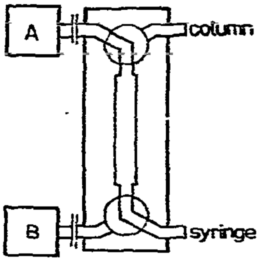

Fig. 3. Schematic diagram of the connection to the column.

CONCLUSION

The electroosmotic pump is used in our laboratory not only as a counter flow device but also for sampling by the continuous sampling technique[10]. Its advantage consists in the simple and accurate control of the flow, including changes in the direction of flow. The pump has no elastic parts, which could cause disturbing effects.

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