CHROM. 3792

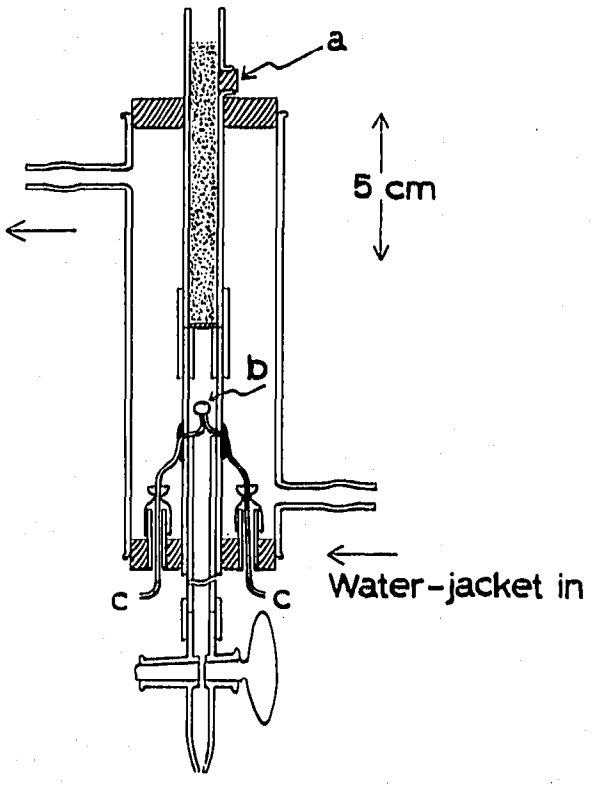
Thermal conductivity detection in liquid chromatography

Use of a micro adsorption detector in liquid chromatography has been reported by NAONO AND PRCHAL[1] and HUPE AND BAYER[2]. They detected the heats of adsorption and desorption in the chromatographic process by means of a micro thermistor placed in the center of the cell which was filled with a suitable adsorbent material. The chromatogram obtained did not show the ordinary Gaussian shape but rather its differential peak shape because the detector measures the gain and loss of heat. The liquid chromatograph constructed on the basis of measurement of adsorption-desorption heat was supplied by the Japan Electron Optics Laboratory (Nihon Denshi) and Varian Aerograph.

In this report we tried to introduce into liquid chromatography thermal conductivity detection by means of thermistors and to measure the temperature change due to the difference in thermal conductivities between eluent and eluted materials.

Experimental

Fig. 1 shows the column assembly. A thermistor, Ten KD-27, 4.2 mm in diameter and 2.2 mm thick, disk type, having an electrical resistance of 270 Ω at 25°, was used. It was coated with Araldite (Ciba Co.) to avoid electric contact with eluent,



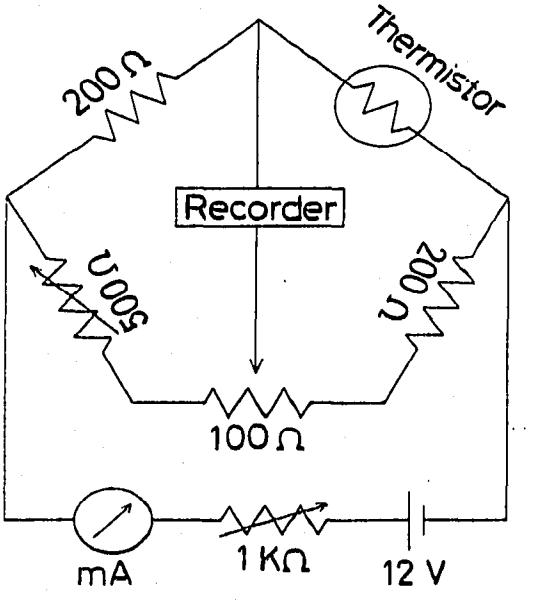


Fig. 1. Column assembly. (a) = Silicone-rubber stopper for sample injection; (b) = thermistor; (c) = lead wire.

Fig. 2. Bridge circuit.

and was placed in the center of a glass tube, 7.5 mm in diameter, connected directly to the column outlet. The sample was injected by means of a syringe through the silicone-rubber stopper inserted into the hole on the side of the column inlet. The column, made of glass tube, 7.5 mm in diameter, was packed with Sephadex G-25 Coarse (Pharmacia Co., Uppsala) and had an effective height of 8.3 cm. The temperature of column and detector was maintained at 10.7 $\pm$ 0.1° by a well-water circulation. The sample, Blue Dextran 2000 (Pharmacia Co.) and sodium chloride aqueous solution, was eluted with deionized water. The flow rate was kept constant by controlling the level of eluent in the reservoir with a stopcock.

In the bridge circuit (Fig. 2) a single active bridge arm was used in order to simplify the experimental work.

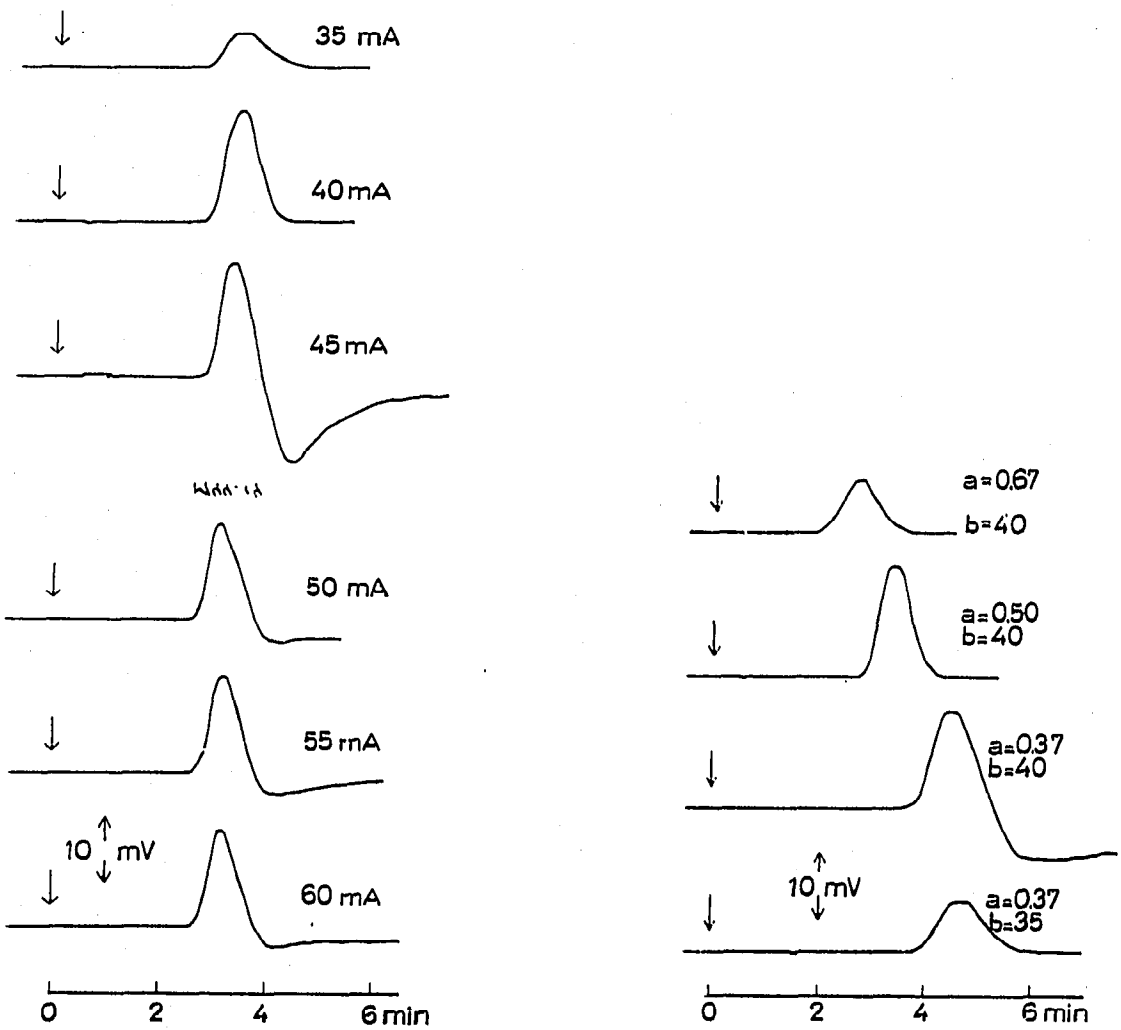


Fig. 3. Chromatograms of Blue Dextran 2000 eluted from the Sephadex column with water. The figures show the bridge current. When the bridge current exceeds 45 mA, a negative deflection appears in the chromatogram. The arrows indicate the injection point. The flow rate was 0.5 ml/min.

Fig. 4. Effect of flow rate on the chromatogram. (a) = Flow rate (ml · min^{-1}); (b) = bridge current (mA).

Results and discussion

The chromatograms recorded are given in Figs. 3–6. It may be safe to say that in principle the measurement of the electric resistance of the current-fed thermistor, cooled by eluate of various compositions, can be applied to liquid chromatography.

With increasing bridge current and decreasing flow rate, a slight negative deflection appears in the chromatogram, as shown in Figs. 3 and 4. The reason for this is not clear, however.

The chromatograms of Blue Dextran 2000 and sodium chloride are shown in Fig. 5 together with that of their mixture. The reproducibility of retention volume is good.

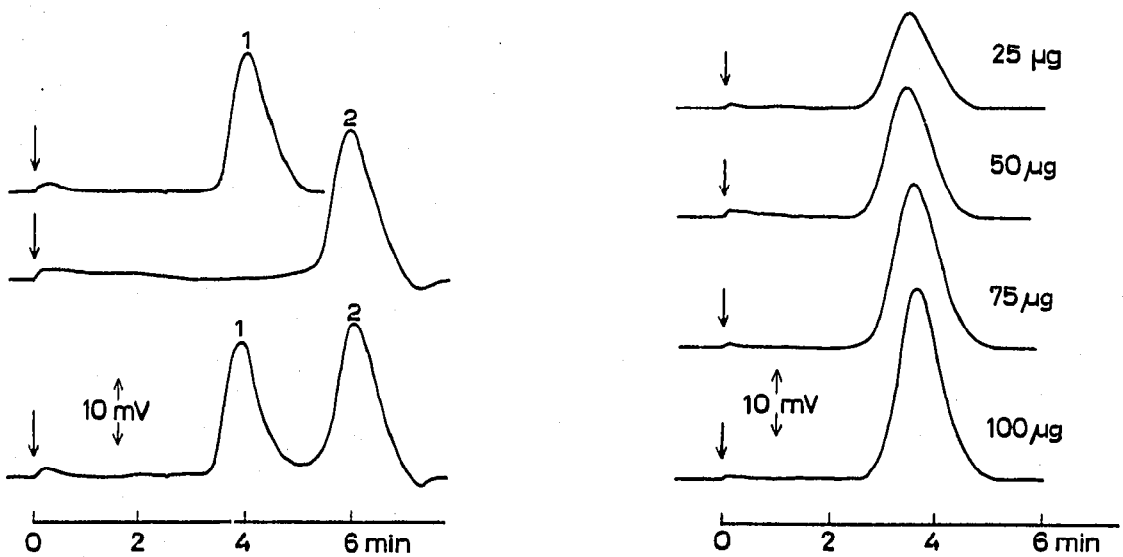


Fig. 5. Chromatograms of Blue Dextran 2000 (1) and NaCl (2), each 50 µg, and their mixture.

Fig. 6. Variation of the chromatogram with amount of Blue Dextran 2000 injected.

Variation of chromatogram with the amount of Blue Dextran 2000 injected is shown in Fig. 6. The peak area shows a linear relationship with the sample size, but the proportionality is not good.

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