Chromatography on paper impregnated with inorganic ion-exchangers

The impregnation of filter paper with ion-exchange resins has been carried out by LEDERER, who achieved good separations of elements in this way and predicted other possible separations¹⁻³.

In this preliminary note we shall describe some experiments, of the same nature, using papers impregnated with zirconium phosphate and zirconium molybdate, whose ion-exchange properties were first demonstrated by Kraus *et al.*^{4,5}.

Preparation of the ion-exchange paper

Whatman No. 3 MM sheets, 28 cm long and 21 cm wide, were dipped into an aqueous solution of ZrOCl₂·8H₂O, containing 20 g Zr per liter. After impregnation they were quickly placed on pieces of Whatman No. 1 paper, in order to absorb the excess of liquid uniformly, and left to dry at room temperature. The papers were then dipped into a 3% H₃PO₄ solution or a 10% (NH₄)₆Mo₇O₂₄·4H₂O solution, transferred to a dish with distilled water, again placed on pieces of Whatman No. 1 paper and left to dry. Finally the edges of the sheets were cut off to eliminate errors due to a possible accumulation of the exchanger on those regions.

The treated paper has the appearance of untreated paper, but it is a little more hardened.

Separation of Ca, Sr and Ba

We tried to repeat on paper the work of KRAUS et al. concerning the separation of alkaline earths on zirconium molybdate columns.

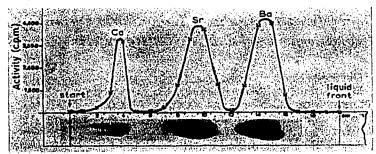


Fig. 1. Separation of a mixture of Ca, Sr and Ba on zirconium molybdate paper, with 0.9 N NH₄Cl for \sim 1h 40 min.

Strips of zirconium molybdate paper 25 cm long and 3 cm wide were spotted with a mixture of 45 Ca, 80 Sr, 140 Ba in 0.5 N HCl and developed for 20 cm with a NH₄Cl solution by the ascending method. The two last-mentioned radionuclides were in the carrier-free state.

To establish the most convenient concentration of the eluent, development was carried out with NH₄Cl solutions of various concentrations, ranging from 0.1 to 1.3 N. The best separation was obtained with 0.9 N NH₄Cl; this is shown in Fig. 1. It should be noted that the curve and the autoradiogram of this figure represent the radioactivity of the chromatogram measured 1 week after separation. If the measurements

were made shortly after elution another maximum was observed situated in the starting region before Ba, due to 149 La ($T_{2} = 40$ h), which was in radioactive equilibrium with 149 Ba.

Experiments were also performed with a mixture containing 5 μ g of each element, traced with the same radionuclides. The separation is similar though the R_F values are slightly higher.

Separation of Cs from Ca, Sr and Ba

We also made an attempt to repeat on paper the work of Kraus et al. concerning the separation of alkali metal from alkaline earths on zirconium phosphate columns.

Strips of zirconium phosphate paper were spotted with a mixture of carrier-free 137 Cs and of the radionuclides of Ca, Sr and Ba in 0.5 N HCl and developed for 15 cm with HCl solution by the ascending method. Development was carried out with HCl solutions of various concentrations.

It was observed that the separation is already possible with o.r N HCl and good

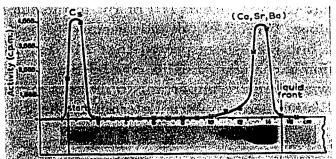


Fig. 2. Separation of a mixture of Cs, Ca, Sr and Ba on zirconium phosphate paper, with 0.5 N HCl for \sim 45 min.

results are obtained with a wide concentration range of the eluent. Fig. 2 shows the results obtained with 0.5 N HCl.

In another series of experiments a salt form (NH_4^+) of the exchanger was used. Strips of zirconium phosphate paper were dipped into a N NH_4OH solution, washed with distilled water and left to dry at room temperature. They were then spotted with the same mixture and developed with a 2N NH_4Cl solution.

A good separation was also obtained, but in this case the position of the elements is inverted, which agrees essentially with the results of Kraus et al.

The separation is equally good for quantities of the order of 5 μ g of each element.

Instituto de Alta Cultura-C.E.E.N., Centro de Quimica J. M. PEIXOTO CABRAL (I.S.T.), Lisbon (Portugal)

¹ M. LEDERER, Anal. Chim. Acta, 12 (1955) 142.

² M. LEDERER AND S. KERTES, Anal. Chim. Acta, 15 (1956) 226.

³ M. LEDERER, J. Chromatog., 1 (1958) 314; 2 (1959) 209. ⁴ K. A. KRAUS AND H. O. PHILLIPS, J. Am. Chem. Soc., 78 (1956) 694.

⁵ K. A. KRAUS, H. O. PHILLIPS, T. A. CARLSON AND J. S. JOHNSON, Proc. U. N. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 1958, P/1832.