

enough to allow distinct separation. Yet this system has the disadvantage that it causes tailing of the above-mentioned substances.

By substituting petroleum ether for decalin in the solvent system formamide-benzene-decalin, the method can be utilised in large scale work. The separation of artemetin and casticin on a column of cellulose powder was carried out to test the method.

Cellulose powder (200 g, Whatman standard grade) was impregnated with formamide (66 g) in acetone (150 ml). Then the acetone was evaporated at 50° under reduced pressure. The cellulose was pressed into a column 4 × 28 cm. The mixture of artemetin (185 mg) and casticin (198 mg), dissolved in a small amount of the mobile solvent, was dropped on the top of the column and the mobile solvent (benzene and 20% v/v petroleum ether 30–40°, saturated with formamide) allowed to pass through the column at the rate of 2 ml/1 min. Ten ml portions were collected and examined by paper chromatography. Fractions 1–7 contained artemetin and fractions 10–20 contained casticin. In fractions 8 and 9 neither flavone could be detected. Fractions 1–7, and 10–20, respectively, were combined, and the solvent evaporated. Of artemetin 190 mg and of casticin 200 mg were recovered. After one crystallisation from benzene-petroleum ether both compounds had the original melting points.

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Behaviour of the metals of the 1b group on zirconium phosphate columns and papers

Recent work has revived interest in inorganic ion exchangers and their application¹. Of these, zirconium phosphate was selected for our purpose because of its properties and high capacity. It was prepared according to the method described in the literature².

Columns were made from 6 mm diam. glass tubes constricted at one end and filled to a height of about 40 mm with zirconium phosphate (ZP). Its capacity and distribution coefficients were as previously recorded (capacity = 1.88 mequiv./g).

The zirconium phosphate papers were prepared as follows: Whatman No. 1 paper was impregnated with a solution of 0.2 M $ZrOCl_2 \cdot 8H_2O$ in 4 N HCl, the excess solution drained off and the paper dried at room temperature. These papers were then

dipped into a solution of 12% H_3PO_4 in 4 *N* HCl and washed with water until the pH was 4 and again dried at room temperature.

The behaviour of Ag(I) , Cu(II) and Au(III) was found to be as follows: Au(III) as AuCl_4^- is not retained on a column of ZP, contrary to what happens on an organic exchanger such as Dowex-50³. From a solution of Au(III) and Cu(II) (as CuCl_2) only Cu(II) is retained.

Ag(I) and Cu(II) as sulphates are retained on a column and can be readily separated by eluting first with 0.1 *N* HCl (2 ml suffice) to remove the Cu(II) and then with $\text{NH}_3\text{-NH}_4\text{Cl}$ (4 *N*) which removes the silver.

Au(III) has the same R_F value on ZP-impregnated paper as on ordinary Whatman No. 1 paper ($R_F = 0.65 - 0.70$) when developed with 0.01 *N* HCl or water.

When a mixture of Ag(I) and Cu(II) sulphates are developed with 0.1 *N* HCl , Ag(I) stays on the point of application and Cu(II) has an R_F of 0.7 - 0.8. Thus the behaviour is analogous to that on ZP columns.

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A rapid method for the preparation of packing material for gas chromatographic columns

The generally accepted practice in preparing the stationary phase for gas-liquid chromatography is to dissolve the liquid substrate in a suitable volatile solvent, add this to the support material and then remove the solvent by careful heating. This is often a tedious procedure and is subject to frequent "bumping" of the mixture.

A more satisfactory way of removing the solvent is to carry out the evaporation in a rotating-type vacuum evaporator. This operation takes only a matter of minutes and on completion the packing generally requires very little further conditioning.

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