

the anion exchange bed. This may readily be demonstrated by holding moist litmus paper in the exit gas stream or, more simply, by following a characteristic color change (yellow to amber) which this batch of resin shows on being saturated with HCl. Large amounts of HCl may be adsorbed by the ion exchanger and the frontal edge is sharp even with relatively rapid gas flow. With water-washed resins, the amount of HCl held was approximately 0.2 g per c.c. of bed corresponding to *ca.* 6 moles HCl per liter of bed.

If gas sweeping is continued until the resin column is saturated with HCl, there is danger of substantial loss of Ge(IV). For example, in one experiment gas sweeping was continued until the band of adsorbed HCl had reached the middle of the second column. Under these conditions *ca.* 20% of the ⁷⁷Ge activity was transferred to the second column and only 80% was retained by the first one.

One of the many advantages of the gas sweeping-anion exchange technique is the ease with which Ge(IV) may be recovered from the anion exchange columns. Desorption may readily be achieved by washing the columns with a few column volumes of water or, preferably, of dilute HCl solution. Since the first part of the effluent is rather concentrated in HCl, some care must be exercised to avoid loss of Ge(IV) by volatilization from the effluent.

The small scale technique described for separation and isolation of Ge(IV) should be adaptable to large scale applications provided precautions are taken to avoid complications resulting from the limited solubility of Ge(IV) in concentrated HCl solutions⁶ or in resin columns⁵.

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Plastic bag technique for paper chromatography

Conventional paper chromatography generally employs microgram quantities of the compounds to be separated per sheet of paper. Column chromatography is used for larger amounts. However, cellulose powder columns produce broader and not as well defined zones as separations on filter paper, which is more uniform in structure.

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In an attempt to separate larger amounts of material with the definition given by filter paper, MITCHELL AND HASKINS¹ developed the "chromatopile" method. The detection of the zones required the removal of many sheets from the pile. PORTER² modified this technique and clamped stacks of accurately cut paper strips to form a "chromatopack". The "Isolierpack" of FISCHER AND BEHRENS³ was a further modification. The sample was applied to each of the strips of paper before they were accurately lined up for elution, a time-consuming procedure. ZECHMEISTER⁴ used a glass column packed with filter paper discs. Unless a precision bore tube and precision-punched papers were used "channeling and gross irregularities in the flow of solutions became manifest". DANIELSON⁵ and HAGDAHL AND DANIELSON⁶ described paper columns for preparative work, using filter paper tightly wound around an inert cylindrical rod and pressed into an outer cylinder of polythene. Special machines would be required to obtain close packing, which is a prerequisite for sharp separations. BROWNELL, HAMILTON AND CASSELMAN⁷ introduced the heavy paper technique. The sample is applied to Whatman seed-test paper as a uniform streak but in quantitative work difficulties were experienced in applying a predetermined volume uniformly.

To overcome these drawbacks, the author attaches a plastic bag to the seed-test paper (Fig. 1) through which sample and eluent are applied by gravity as in column chromatography. Fractions may be collected from a drip point or left as zones on the paper. This technique combines the simplicity of sample addition as in column chromatography with the advantages of uniformity of the filter paper.

For ascending elution, after the sample has been applied and washed into the heavy paper, the strip is turned upside down as shown in Fig. 2. Another possibility

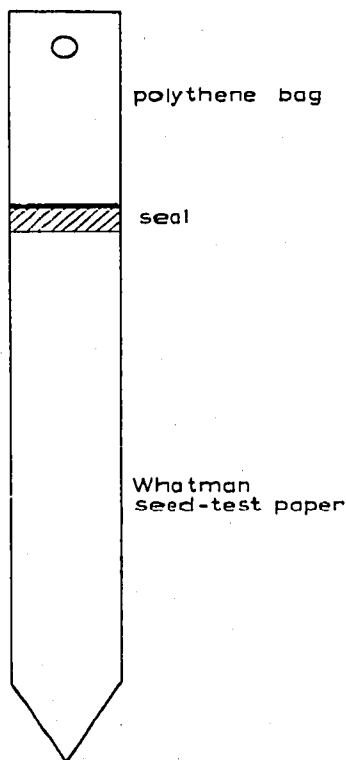


Fig. 1a

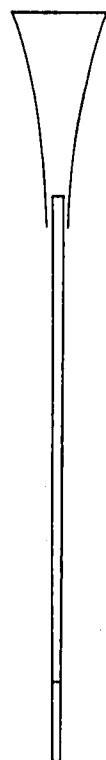


Fig. 1b

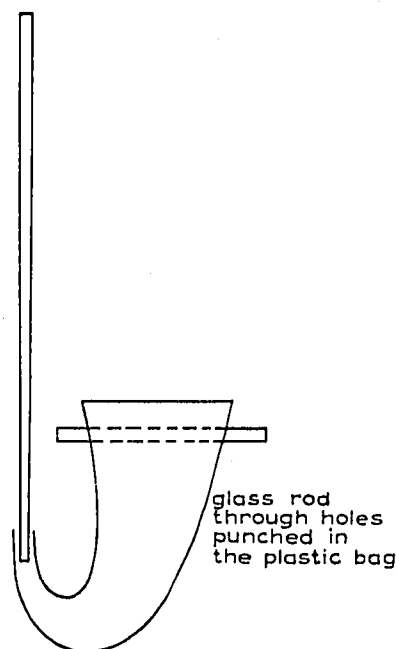


Fig. 2

would be, after the sample has been applied and washed in, to cut the plastic bag off and to proceed in the usual manner.

"Lay-flat" polythene tubing 2-3 inches wide and 0.005 inches thick proved most suitable for our purposes. However, other sizes may also be used.

Whatman seed-test paper is cut to suit the tubing available and to provide a tight fit when slipped about one quarter to half an inch into the tubing. The polythene is heat-sealed onto the paper, using a conventional strip sealing machine. A soldering iron run at reduced voltage produces an equally good seal, provided silicone grease is used to prevent the polythene from sticking to the copper tip.

Alternative but less satisfactory means of attaching the polythene tubing to the paper are clamping, sewing and sealing the stitches, or using an adhesive for polythene, which must not penetrate into the paper sufficiently to cause a blockage. Furthermore, the adhesive must not be soluble in the solvents subsequently used.

Fig. 3 shows the separation of a mixture of dyes by this technique. 5 ml of a water solution containing 5 mg each of Naphthalene Orange, Solway Blue, Azo Geranine, Fluorescein, Lissamine and Coomassie were simply pipetted into the polythene bag. Elution was done with water.

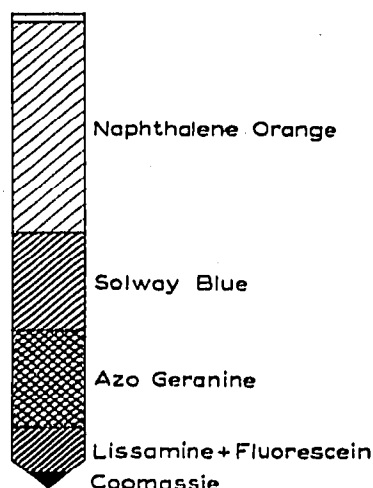


Fig. 3.

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