

the sulphur atom³ in the transition state for re-arrangement (II).

Although benzylic type ethers do not readily undergo rearrangement-elimination in DMF, it has been found that aralkyl ethers (ArOR) do β -eliminate to form olefines if a more active dipolar solvent is used⁴. In a 0.62 molar solution of potassium *tert*-butoxide/dimethylsulphoxide (DMSO) β -elimination of *n*-butyl phenyl ether and *n*-propyl phenyl ether occurred at 55°. For these experiments, 2,2-dimethylbutane was added as an internal standard and the olefinic products were identified by gas chromatography (21 ft. Dow Corning silicone oil on 'Chromasorb'). In the case of the *n*-propyl compound, a 24.5 per cent yield of propylene was observed after 169 h of reaction. The *n*-butyl derivative gave a 17.6 per cent yield of an equilibrium mixture of but-1-ene and *cis*- and *trans*-but-2-ene in the same reaction time. However, the leaving group in these ethers is a resonance stabilized phenoxide ion. Attempts to activate dialkyl ethers under these conditions was, as anticipated, not successful.

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Preservation of Thin-layer Chromatograms

THE preservation of chromatograms is one aspect of thin-layer chromatography which cannot be translated directly from the paper chromatographic technique. It is obviously impracticable to store the glass plates themselves and the answer to the problem lies in removing the completed thin-layer chromatogram from its glass support. Techniques have been described which rely on spraying the thin layer with suitable polymer dispersion, which hardens on drying to coat the layer with a clear film. The coating, plus the layer, is then removed mechanically from the glass plate. The method which is described here is an alternative means of preserving thin layer chromatograms which obviates the need for any spraying of the completed chromatogram and which is, in consequence, particularly useful for thin layers of a brittle nature.

In this method the chromatogram is removed from the glass plate by means of a self-adhesive plastic film. Films composed of cellulose acetate and PVC have been used in this connexion. ('Transpasene' and 'Transpaseal', manufactured by Dispro, Ltd., were used in this investigation.) The PVC film was more flexible and less porous than the cellulose acetate film and because of this was more useful for the preservation of chromatograms, which have a tendency to fade.

The completed thin layer chromatogram is placed face upwards on a flat surface. A sheet of plastic film is cut to a size which allows about 1-in. overlap around the edges of the glass plate. The backing paper is removed from the film and one edge of the film is turned under the glass plate and fixed in position. The film is then carefully smoothed over the thin layer working from the fixed edge. When in position over the layer, pressure is applied uniformly to the surface of the film using the edge of a ruler or a glass rod until there is a uniform dispersion of the particles of the adsorbent over the adhesive surface.

In this way the surface layer of the adsorbent particles, including the coloured zones, are transferred to the plastic film, which can then be peeled from the plate.

As a final step the plastic film is backed by a sheet of white card or paper of the same size as the original glass plate.

It has been found that using this procedure even amino-acid chromatograms, which fade readily, can be preserved in the absence of light for periods of several weeks provided the transfer of the thin layer to the plastic film is carried out immediately after completion of the chromatogram.

I thank the Directors of W. and R. Balston, Ltd., for permission to publish this work.

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BIOPHYSICS

Preparation of Sieves with Microscopic Pore Diameters

SIEVES with pore diameters in the micron range are finding increasingly frequent applications. While a number of commercially available articles fulfil criteria of pore size uniformity, these pathways are generally tortuous, with great individual variation in the exact hole pattern. A simple technique for preparation of such filters has therefore been developed.

A bundle of nylon monofilaments (kindly supplied by E. I. du Pont de Nemours and Co., Inc.) were oriented parallel to the long axis of a gelatin capsule mould, and embedded in butyl methacrylate polymerized at 60° C for 24 h. These blocks were then cut perpendicular to the fibres with a sliding microtome, and the sections floated in 37 per cent hydrochloric acid, dissolving the nylon.

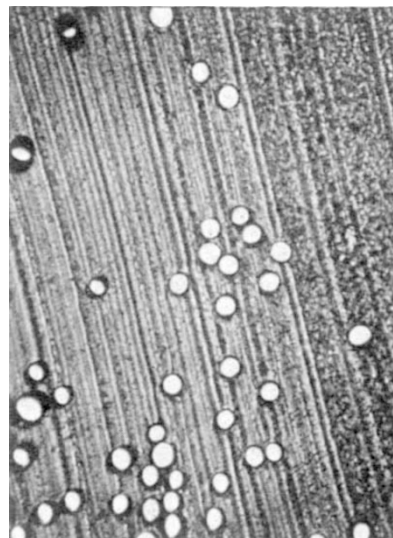


Fig. 1

The illustrated sieve in Fig. 1 has pores approximately 22 μ diameter in a 40 μ thick matrix. Orifice diameter/depth ratios as high as 1/10 are attainable with prolonged acid treatment, while the other specifications depend on choice of fibre, the mould used, and the microtomy technique. Serial sections are virtually identical, while the parallel orientation of the holes can be improved by keeping the fibres under tension during the embedding procedure.

Other fibre-embedding media combinations are now being investigated.

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