CHROM. 3684

## Sintered glass as a permanent medium for thin-layer chromatography

Powdered glass has been used as an adsorbent and as a partition support in column chromatography, and by several workers including BRUD[1] as a medium for TLC, by coating a plate in the usual manner by means of a slurry.

Porous glass (Corning Glass Co.), in the form of a powder with a very high degree of microporosity, has been used by KRAMER *et al.*[2] in the same way. MACDONELL AND WILLIAMS[3] successfully separated coloured ink fractions on polished sheets of porous glass, using aqueous solvents and long elution periods. The separate zones were then capable of characterization by light absorption methods through the transparent glass. Activation was produced with hydrofluoric acid and with boiling methanol.

In the present preliminary work, fine powdered soda glass was fractionated by water flotation, retaining a particle size in the range 180 to 220 mesh. A water slurry was spread on glass, allowed to settle, and dried. The layer was sintered by heat, obtaining bonding between the particles and to the supporting clear glass sheet base. The resulting thin-layer plates are resistant to coating removal by accidental abrasion, and can be cleaned by chromic acid or any other normal glass cleaning method, without damage.

Activation of the surfaces of the layer was achieved by treatment with sulphuric acid and heating, and, less successfully, with hydrofluoric acid, phosphoric acid, and methanolic potassium hydroxide, respectively. The detailed procedure giving best activation to date was as follows:

(1) The layer, of 200 mesh soda-glass sintered to soda-glass sheet by heating at 850° for 5 min, resulting in a thickness of 50 to 100 $\mu$, was degreased and cleaned with detergent followed by 0.5% sodium edetate solution, then repeated alcohol and water washing.

(2) The surface was immersed in concentrated sulphuric acid at 100° for 30 min, then washed in distilled water until neutral to blue litmus paper touched to the surface, and finally dried at 150° for 2 h.

Plates prepared in this way are capable of separating the components of the Desaga dye mixture (butter yellow, Sudan red G and indophenol) by eluting with hexane for 2 to 3 min. Some separation was apparent using benzol, but the small size of the plates prepared at this stage did not allow for long elution distances.

Partial success was also achieved in the analysis of a mixture of alkaloids containing morphine, codeine, heroin, quinine and caffeine, on a sintered plate prepared as described, and eluted with benzol. Three distinct zones appeared after spraying with Dragendorff reagent, and a fourth, an overlap, probably quinine, was visible under U.V. light. The plate in this case was made from a microscope slide, and the time of elution was less than 3 min.

An advantage of the proposed layers is their permanence. The surfaces, after the preparation described, appear to retain their characteristics, and therefore produce highly repeatable results, for long periods and many experiments. This may help overcome the variations inherent in normal thin layers due to operational techniques when preparing the plates.

The Stahl dye mixture was also separated on a glass plate which had been

scribed, with a diamond-point, with twenty parallel and closely-spaced lines forming a strip 2.5 mm wide. The amount of material separated was minute (about half a microgram), and examination with a magnifier and coloured filters was necessary to observe the coloured zones. Activation of the glass was carried out in the same way as that of the sintered layers.

Sintered layer plates might also be used as support for a stationary phase in partition separations, with some advantages over analogous paper chromatography.

*Commonwealth Laboratory, Customs House,* IAN S. TAYLOR
*11 William Street, Melbourne 3000 (Australia)*

1 W. S. BRUD, *J. Chromatog.*, 18 (1965) 591.
2 J. K. G. KRAMER, E. O. SCHILLER, H. D. GESSER AND A. D. ROBINSON, *Anal. Chem.*, 36 (1964) 2379.
3 H. L. MACDONELL AND J. P. WILLIAMS, *Anal. Chem.*, 33 (1961) 1552.

Received July 5th, 1968



CHROM. 3686

## Quantitative determination of saturated triglycerides in fats

During the partial catalytic hydrogenation of edible oils, trisaturated glycerides are formed in varying amounts. The extent of formation of these high melting glycerides is not rules by chance, but governed by the hydrogenation conditions. In order to study the parameters which influence their formation there was needed a practically suitable and quantitatively reliable method for the determination of these glycerides. The method described below, based on a TLC–GLC procedure, has been shown to fulfil these requirements.

The classical method is based on a wet oxidation procedure with $KMnO_4$ originally described by HILDITCH AND LEA[1] and later modified by others[2,3] and recently reviewed by CHAKRABARTHY AND GAYEN[4]. The procedure is rather time consuming and the precision and accuracy may, no doubt, be questioned depending on the quantitatively unreliable reactions and manipulations involved. ESHELMAN AND HAMMOND[5] have critically studied the method and conclude that it does not give satisfactory results. Recently another method has been published[6], based upon mercury adduct formation, separation of the non-adduct-forming saturated glycerides and subsequent gravimetric determination.

The present procedure is based upon thin-layer separation of the saturated glycerides on silver nitrate–silica gel coated plates followed by gas chromatographic analysis of the saturated triglyceride fraction after its conversion to methyl esters. The quantification is accomplished with the aid of an internal standard consisting of a suitable saturated triglyceride, which is added to the sample *before* the thin-layer chromatographic procedure. The standard thus accompanies the sample throughout the analysis.