durch zwei Faktoren beeinflusst, einerseits durch das Wasser und andrerseits durch die Menge der einzelnen Substanzen. Letzteres manifestiert sich vor allem bei den wasserfreien Alkoholgemischen durch niedrigere Trennfaktoren. Der Grund dazu dürfte in einer Überlastung der Säulen zu suchen sein.

Eine Verschlechterung der Trennfähigkeit infolge des Wassergehaltes tritt erst bei sehr starken Verdünnungen, 0.1 $^0/_{00}$, andeutungsweise zutage. Störend wirkt sie sich bei noch niedrigeren Konzentrationen im Bereiche von 0.05 $^0/_{00}$ aus (vgl. Tabelle I).

Die Dauer einer Analyse liegt mit dem vorgeschlagenen Temperaturprogramm unter einer Stunde. *n*-Pentanol erscheint nach *ca.* 45 Min. Zudem ist die Trennsäule sofort wieder betriebsbereit, da das Wasser schon nach dem 2-Methylpropanol-2 eluiert wird. Allerdings wird der Wasser-peak durch den Flammenionisationsdetektor nur durch eine geringfügige Nullpunktverschiebung — und nur bei hoher Empfindlichkeit — angezeigt. Die vorgeschlagene Säule eignet sich somit für rasche, direkte Analysen wässeriger Alkohollösungen bis hinunter zu Konzentrationen von 0.1 $^0/_{00}$.

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A simple reproducible technique for sample introduction in analyses of volatile fatty acids by gas chromatography

The determination of the composition of volatile fatty acid mixtures by gas–liquid chromatography in association with automatic titration was first described by JAMES AND MARTIN[1]. Their technique has the advantage over more elaborate detection systems in that absolute values may be estimated easily and with greater precision. Usually relative ratios only of acids are obtained with other types of detectors. To judge from the literature, *cf.* for example SMITH[2], if actual concentrations are desired correction factors must be applied to the calculations.

A difficulty in the JAMES AND MARTIN technique is to obtain a satisfactory method for sample injection which gives a quantitative and smooth delivery of the ethereal solution. The technique now described is satisfactory for this purpose.

A pellet of sintered glass is immersed in the ethereal solution of volatile fatty acids prepared as described by MCINNES[3] and then placed inside the inlet end of the column well within the heated vapour jacket. The amount of acids introduced to the column depends on pellet size and time of immersion. With the equipment in the Fats Research Division D.S.I.R., New Zealand, satisfactory graphs were obtained using

ethereal solutions containing 50 to 150 μM/ml of a mixture of fatty acids comprising mainly acetic with lesser amounts of propionic and butyric, together with traces of iso- and *n*-valeric acids. The pellet (5 × 10 × 30 mm) was immersed for 2 min.

The column head and carrier gas inlet of the chromatograph was modified as shown in Fig. 1 to allow easy introduction of the sample.

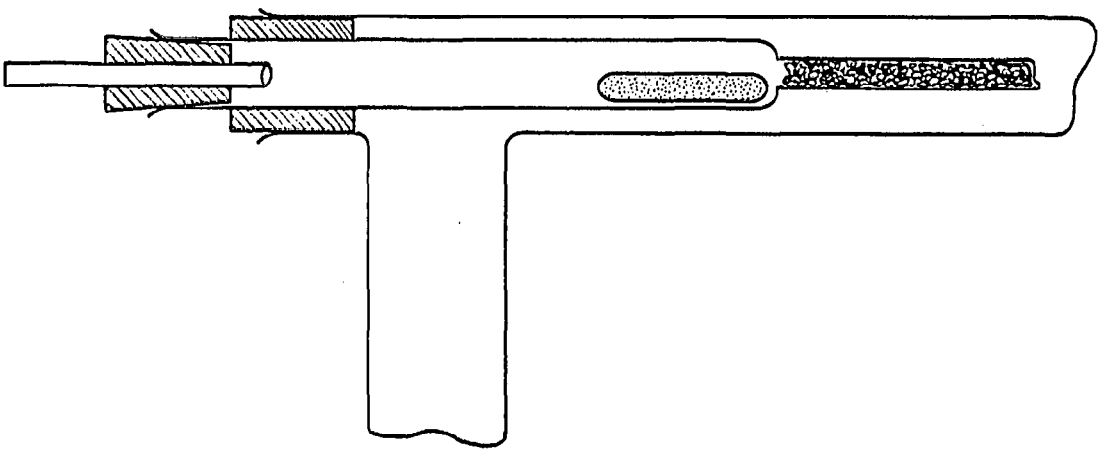

Fig. 1. Modified inlet of gas chromatograph column showing sintered glass pellet in position.

The carrier gas was preheated by passing it through a copper coil to improve the delivery of the sample to the column and prevent possible losses of volatile fatty acids by condensation. The coil was heated by wrapping it several times around the aluminium jacket of a heating mantle set at 220 V.

Comparison of the values of total volatile acids as estimated by titration of ethereal solutions with those calculated from GLC results showed a recovery of 99 ± 3 %.

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