tographically by the total number of milliequivalents of acid found chromatographically, such variations in per cent recovery have no effect on the mole per cents obtained for the various acids. If per cent recovery must be known more exactly, use of a column with higher resolution should allow an internal standard to be employed.

If the recorder is operated at maximum sensitivity and the evaporation is carried to 0.5 ml., samples containing in the neighborhood of 0.1 meq. of total acidity may be successfully analyzed by the procedure as outlined. Samples containing total acidity in the range of about 0.1 to 4 meq. thus may be analyzed by chromatography of 20 μl. of the final solution. The introduction of such a large volume provides sensitivity at the expense of resolution. For those samples containing 1 meq. or more of total acidity, resolution may be improved by reducing the volume introduced to 2 μl. If the sample contains less than 0.1 meq. of total acidity, it may be necessary to carry the evaporation to less than 0.5 ml. Instrumental conditions may be varied widely to meet the requirements of a particular analysis or investigator.

Conclusions

The procedure may be readily adapted to methyl esters by reacting the acids with diazomethane (*3*) following the final concentration step. However, the Silicone 550–stearic acid column used for separating the free acids has been studied in detail by James and Martin (*6*) and found capable of resolving a wide spectrum of acids. Thus, for many applications, chromatography of the free acids is not only adequate but desirable. The techniques for sample concentration and water removal can undoubtedly be extended to other types of mixtures analyzed by gas chromatography.

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