The use of gas-liquid chromatography-mass spectrometry in biochemical pharmacology

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Although the use of paper, thin-layer, ion exchange and conventional column adsorption chromatography has greatly facilitated the separation and identification of substances of biological interest, the accurate identification of closely related substances cannot be relied upon solely by $R_{\rm F}$ values even with the use of co-chromatography.

Mass spectrometry has been used for many years to identify compounds but requires isolation of individual substances. The advent of gas-liquid chromatography has allowed rapid separation of many closely related substances. The combined gas chromatograph—mass spectrometer therefore provides a sophisticated technique for identification of individual components in mixtures of biological substances.

We have coupled an F & M 5750 series gas chromatograph to an EAI Quad 300 mass spectrometer with fast ultraviolet recorder.

The fast scanning speed (total spectrum <1 s) of this particular mass spectrometer has been utilized to scan repetitively during the time taken for components to emerge from the gas chromatograph. The scanning is controlled by an optical switch on the GLC recorder which automatically activates the recorder of the mass spectrometer. This ensures an optimum mass spectral response for a given gas chromatographic peak and also facilitates resolution of inadequately resolved components from the chromatograph.

The combined instruments will be demonstrated in relation to the analysis of amino-acids and basic drugs.

Analysis of radioactively labelled compounds of pharmacological interest by gas-liquid radiochromatography

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A Perkin-Elmer/Berthold RGC 170 gas flow proportional counter has been modified to facilitate direct coupling to an F & M 5750 series gas chromatograph using the shortest possible heated transfer line. The effluent from the chromatographic column is passed through a two-way stream splitter, one part going to the flame ionization mass detector. The other part passes through the heated transfer line to the combustion furnace of the RGC 170 radioactivity detector. Here the sample may be converted either by oxidative combustion or hydrogenative cracking into a mixture of permanent gases for counting at room temperature in the proportional counter.

Such a continuous-flow system allows all emergent peaks containing enough radioactivity to be monitored whether or not the mass of the peak is sufficient to be detected by the flame ionization detector.

The ability to convert the chromatographic effluent to permanent gases either by hydrogenation or by oxidation means that ¹⁴C compounds, for example, can be converted to ¹⁴CO₂ or ¹⁴CH₄ and ³H-compounds to C³H₄ and N³H₃.