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Radio gas chromatography by collection of radioactive compounds issuing from a gas chromatograph on a moving strip of paper

The best currently available methods for the detection and estimation of highly radioactive compounds separated by gas–liquid chromatography (GLC) consist in continuously counting the effluent[1]. However, if a compound is only weakly radioactive, it must be counted for a longer time than is possible in a continuous flow system and hitherto, this has involved trapping out discrete fractions with subsequent counting.

Several authors have collected the effluent from a gas chromatograph on a moving thin-layer plate[2-5] and a commercially available device is available. In our system, radioactive compounds are condensed onto a moving paper strip and subsequently counted, thus combining the sensitivity of a discontinuous counting system with the advantages of continuous operation as in a flow system.

Apparatus

A Pye series 104 chromatograph equipped with a splitter was used. The effluent from the splitter capillary tube was brought out of the chromatograph through a 9-cm length of bent 1.5-mm O.D. stainless-steel tubing. This tubing was heated through its entire length with 50 cm of 0.3 mm constantan wire wound in two layers, the layers being insulated from the tube and each other with 2-mm and 4-mm bore impregnated glass fibre sleeving (R.S. Components Ltd.). The windings were spaced more closely together near the outlet of the tube. The cold resistance of the winding was 19 Ω. Heater current was obtained from the 13-V tap of a transformer which was fed from a variable transformer.

The tube outlet almost touched the cold surface of a thermoelectric refrigerating device (type TL 0812P, MCP Electronics Ltd.) the hot side of which was screwed to a water-cooled brass block. In use, a strip of silica gel loaded paper (Whatman SG81) was pulled between the tube outlet and the cold surface by attaching it to the recorder chart: in this way, exact synchronism between the movement of the recorder chart and the paper strip was achieved.

Results

The method has been used to collect methyl esters of fatty acids emerging from a column of PEGA operating at 180°. The side arm was run at a temperature ranging from 300° inside the oven to 215° near the outlet; in the present apparatus, this was achieved by passing a current of 750 mA. Thermoelectric cooling conferred a slight advantage over the use of water cooling alone. A current of about 1 A was found to be optimum; more than this caused frost to appear on the cold surface.

Efficiency of recovery. The split ratio was determined by measuring the area of the peak, with or without the side–arm in position, when the same amount of methyl palmitate was applied to the column. It was found that 60% of the applied material went to the detector and 40% emerged through the side-arm. The split ratio remained constant over the range 1–10 μg of methyl palmitate.

Methyl-^{14}C palmitate was applied to the column and a length of SG81 paper

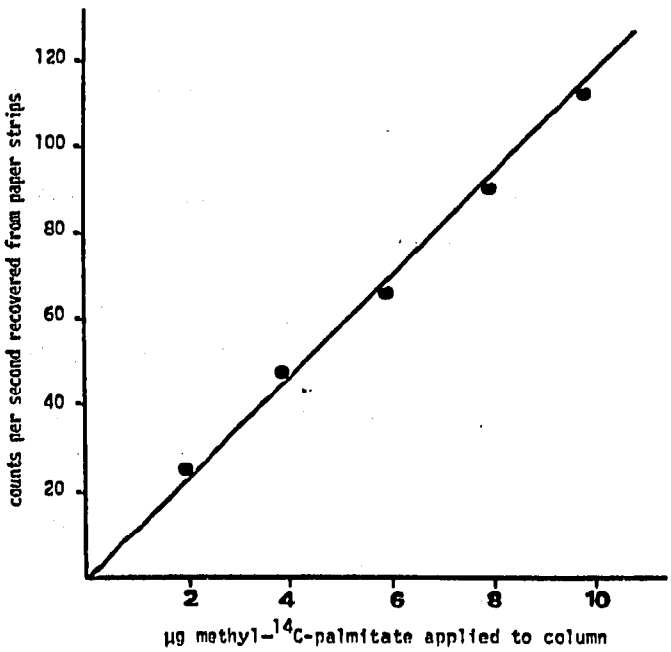

Fig. 1. Recovery of methyl-^{14}C palmitate from radio gas chromatograph.

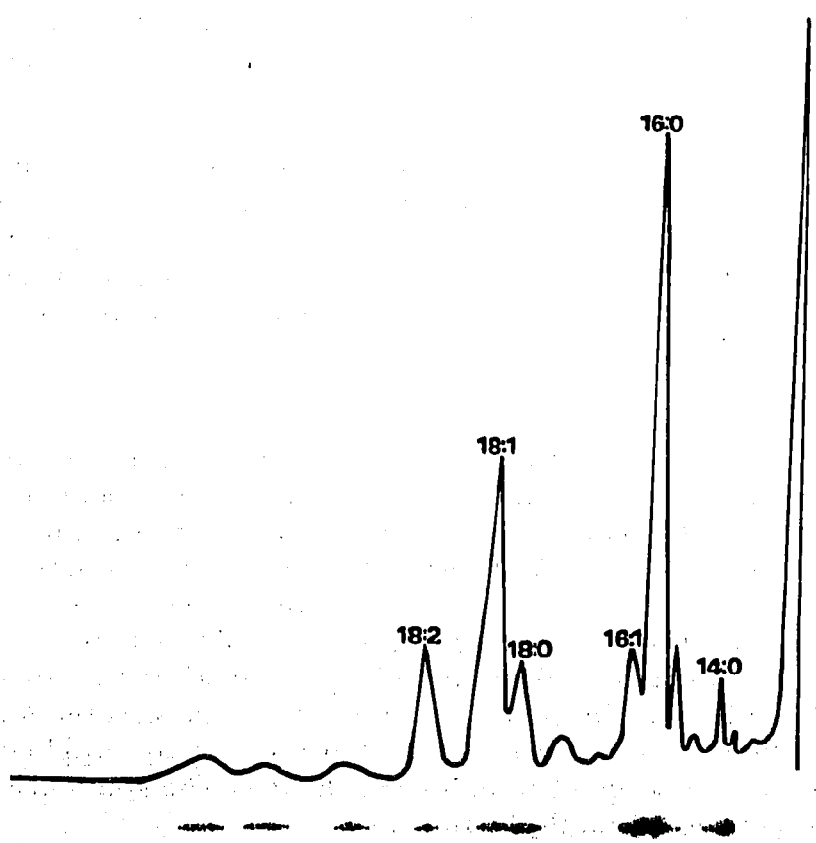

Fig. 2. Mass trace and autoradiograph of corresponding paper strip.

corresponding with the position of the peak was cut up and added to a liquid scintillation vial. Scintillation solution (5 ml) consisting of 0.4 % butyl-PBD in toluene was added and the activity counted. An identical amount of methyl-^{14}C palmitate was injected directly onto a piece of SG81 paper and its activity measured. Knowing the amount of radioactivity applied to the column, the amount recovered from the moving strip of paper and the split ratio, the recovery of material issuing from the side-arm was calculated to be 28 %. The recovery was linear over the range 1–10 μg methyl palmitate (Fig. 1).

Autoradiography. In exploratory work, it is convenient to make an autoradiograph of the strip of silica gel loaded paper which can then be aligned with the gas chromatograph trace. Fig. 2 shows the fatty acid composition and pattern of incorporation of [1-^{14}C]acetate into the fatty acids of a rat skin slice. It can be seen at once that there is little incorporation of label into the 18:2 acid, which is probably the essential fatty acid, linoleic acid.

The radio gas chromatograph has also been used to determine the distribution of radioactivity in the fatty acids of phosphatidyl choline which had been prepared from cultures of *Chlorella vulgaris* grown in the presence of [1-^{14}C]oleic acid. The distribution obtained was in agreement with the distribution of radioactivity in the fatty acid classes determined by thin-layer chromatography on silver nitrate plates[6].

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