The prevention of condensation of very long chain fatty acid esters in certain gas chromatographs*

In GLC analysis of samples of rapeseed fatty acid methyl esters on polyester columns in an Aerograph A-350-B dual column instrument, the erucic acid ester (22:1) peak appeared as in chromatograms (A) or (B) in Fig. 1. Although the peak area, as measured by an electromechanical integrator, could be used in quantitative analysis with reasonable accuracy, the analysis conditions were not regarded

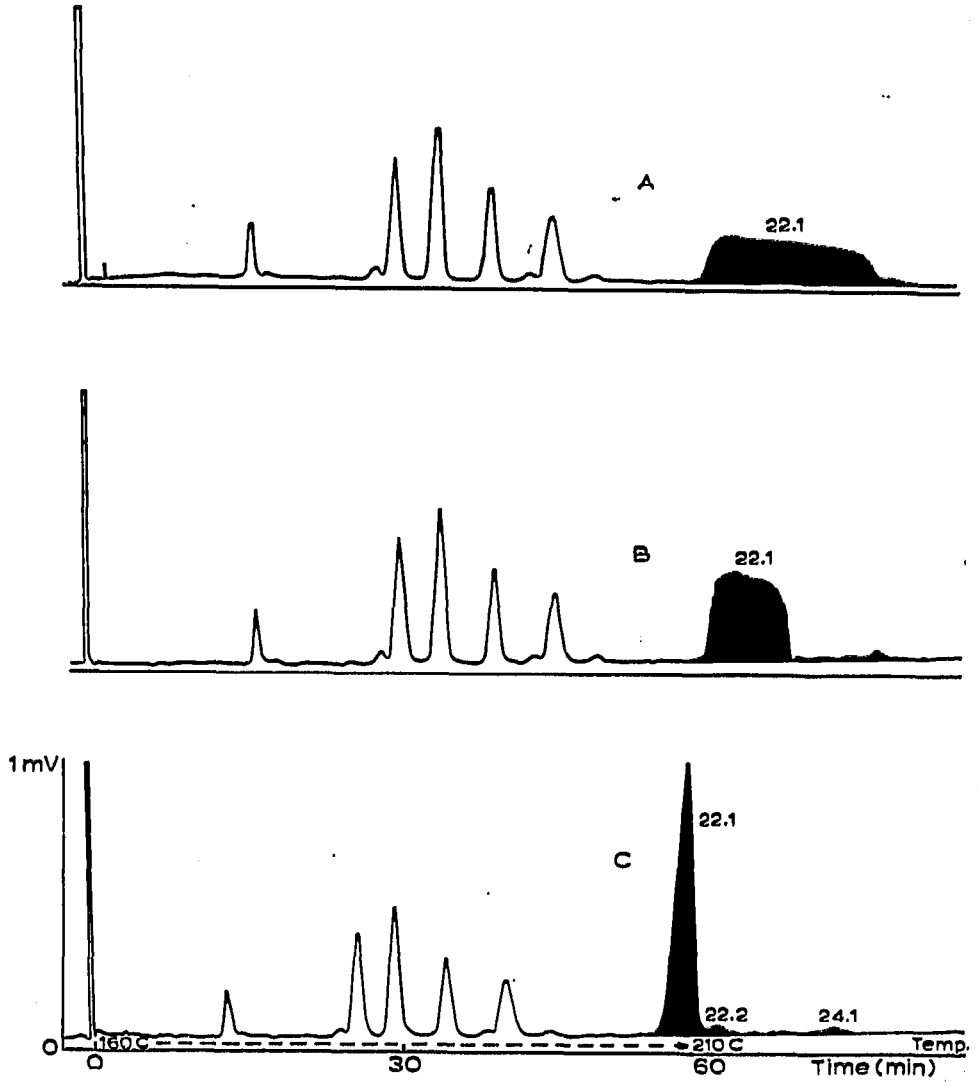

Fig. 1. Analysis of rapeseed oil fatty acid methyl esters on an Aerograph gas chromatograph model A 350-B. Analytical data: 8 ft. × ¼ in. copper columns packed with 60–80 mesh Celite containing 10% BDS. Sample size, 0.5 μl. Injector temperature, 275°; detector temperature, 240°; column temperature 160–210° at a nominal rate of 1°/min. Flow rate of helium, 100 ml/min. Attenuation × 1. The letters A, B and C are referred to within the text.

satisfactory, as the minor components, 22:2 and 24:1 were obscured. Analytical parameters such as sample size, flow rate of carrier gas and temperature programming

* Communication No. 282 from the Swedish Seed Association.

rate were changed in efforts to obtain a sharp erucic acid peak. However, such changes were ineffective in solving the problem. It was observed that if very small samples were used on columns with 10 % stationary phase, a sharp erucic acid peak was obtained. With this system minor components gave peaks too small to permit calculation, although the instrument was run on the highest sensitivity. Columns with 30% stationary phase gave, on the other hand, symmetrical erucic acid peaks for larger samples but the very long retention times made this solution of the problem less attractive. Consideration of overloading the column failed to account for the abnormal erucic acid peak as much larger concentrations of methyl oleate gave symmetrical peaks. The existence of so-called "cold spots" within the chromatograph, where high boiling components could condense temporarily was then considered[1].

A few layers of asbestos cord were wound round the two tubes between the columns and the detector, the connections between which are uninsulated for a distance of 2 cm between the column oven and the detector oven. This insulation had a striking effect as the subsequent chromatograms appeared as (C) in Fig. 1. Obviously the erucic acid methyl ester had condensed in the non-insulated connecting tube, when the concentration reached a critical level. It is believed that this is the reason why columns with 30 % stationary phase yielded symmetrical peaks for larger absolute amounts.

This modification worked properly until an exchange of damaged connecting tubes had to be made, whereupon chromatograms of types A and B reappeared. The phenomenon occurred if a temperature program starting at 160° was used. Isothermal runs at 210° gave symmetrical erucic acid peaks but did not resolve all components. The new connecting tubes were of stainless steel, whereas those used previously were brass, which has a higher thermal conductivity. Heating tape (Electrothermal Engineering Ltd., London), length 65 cm, was therefore wound round the tubes and connected to a 10 V a.c. supply, thereby providing about 10 W. The erucic acid peak again assumed the appearance demonstrated in chromatogram (C) in Fig. 1.

Even if this phenomenon does not appear in chromatographs equipped with more sensitive detectors, requiring smaller samples, it was thought that the solution of the problem would be of interest to all who have similar equipment in use with high boiling compounds.

The author wishes to thank Dr C. G. YOUNGS, Saskatoon, Canada, for valuable discussions; Svenska Extraktionsföreningen, Karlshamn, and Malmfonden—Swedish Foundation for Scientific Research and Industrial Development, for financial support.

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Received January 19th, 1967