

It can be concluded that crude cottonseed oils, since they contain phosphatides similar to the soybean phosphatides, give fictitiously high Halphen responses. It can also be concluded that a cottonseed oil of high Halphen-acid-moiety concentration can be obtained by hot hexane re-extraction of cold-extracted cottonseed meats. It may be possible to obtain a hot-extracted oil of even higher concentration by removing a larger proportion of the oil by cold extraction. This would be of special importance in the preparation of starting materials for concentrating or isolating Halphen acid from cottonseed oils.

The authors are indebted to Vidabelle O. Cirino

and Voyce P. Whitley for free fatty acid and phosphatide analyses and to Julius W. Dieckert for supplying the cephalin.

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[Received April 28, 1961]

• Letter to the Editor

WE NOTED the letter from Fryer, Ormand, and Crump, describing the behavior of triglyceride oils when subjected to gas chromatography (1). We confirm this but find further that conditions can be found such that mixtures of relatively pure tripalmitin and tristearin give sharp separations with moderate retention times.

Figure 1 shows results on a synthetic mixture made from relatively pure triglycerides. These results were obtained with a column containing 3/4% SE-30 on 80-100 mesh GAS-CHROM P, at 300°C. A 2-ft. coiled column of 1/4-in. stainless steel was used, with

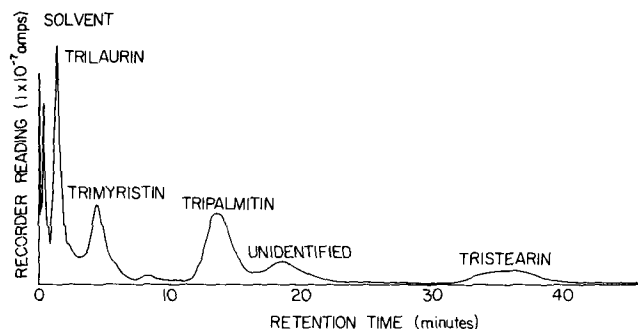


FIGURE 1

an argon rate of 300 ml. per minute and the entire flow passing through an argon ionization detector. The retention time of tristearin was 36 min. The broad peak to the right of the tripalmitin on Figure 1 was also present in chromatograms of the tripalmitin starting material before it was placed in the synthetic mixture.

Figure 2 is a composite chromatogram, showing results obtained with individual triglycerides with 16.7% SE-30 on 60-80 mesh GAS-CHROM P at 284°C in an 18-in. column. All the data give straight lines when carbon number is plotted *versus* log retention-time. Apiezon wax and relatively heat-stable polyester phases, such as ethylene glycol isophthalate and neopentyl succinate, gave unsatisfactory results.

Because of the nonpolar nature of SE-30 a separa-

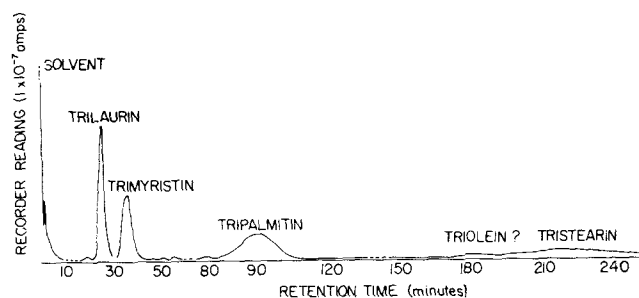


FIGURE 2

tion of triolein from tristearin was not expected, but there was some indication that triolein has an appreciably shorter retention time than tristearin on SE-30. A polar silicone such as QF-1 (2) may well be preferable for this purpose, but temperature programming or further reduction of the silicone percentage may be necessary further to reduce retention times and minimize decomposition or other reactions of the triolein.

No collection of effluents or studies of decomposition or other losses have been made, but it is judged from peak sizes that 90% of the 0.4 lambda samples of the higher triglycerides emerged from the column and showed on the recorder chart. Quantitative interpretation of the chromatograms would be premature until decomposition and calibration studies have been made. At present the procedure is chiefly useful for indication of impurities in triglycerides from various sources and for examination of relatively simple mixtures. The examination of natural oils presents difficulties probably because of the multiplicity of mixed glycerides that are present.

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[Received April 27, 1961]