

show an uniform trend in the 180°–140°F. range for the effect of builders on the solubilization of the two nonionic agents. Nonionic concentration, builder-nonionic mole ratio, temperature, and builder type appear to exert specific influences on the solubilization of specific builder-nonionic combinations. Previous work at this laboratory had shown that builder additions to 0.0062M octyl phenyl decaethylene glycol ether decreased Orange OT solubilization at 122°F. for 4.6:1 to 11.4:1 mole ratios of Na₂SO₄-surfactant and for 2.4:1 to 6.1:1 mole ratios of Na₄P₂O₇-surfactant (7). Similar decreased solubilization by the related nonionic PGNPE was obtained in this investigation in 0.01–0.025 M PGNPE at 1:1 to 5:1 mole ratios of Na₂SO₄-surfactant at 180°F. At 160° and 140°F. there was substantially no effect on solubilization by added Na₂SO₄ at the same builder and PGNPE concentrations. At 180°, 160°, and 140°F. additions of Na₅P₃O₁₀ increased solubilization by 0.01–0.025M PGNPE at 1:1 to 5:1 mole ratios of Na₅P₃O₁₀-surfactant.

Similarly in previous work with 0.0033M PSML at this laboratory (7) it was found that the Orange OT solubilization at 122°F. was substantially un-

affected by additions of Na₂SO₄ and Na₄P₂O₇ at 8.7:1 to 21.6:1 mole ratios of Na₂SO₄-surfactant and at 4.6:1 to 11.7:1 mole ratios of Na₄P₂O₇-surfactant. In this investigation the solubilization of 0.00725–0.0145M PSML was increased by 0.025–0.05M additions of Na₂SO₄ at 180°F. while these additions had no substantial effect at 160° and 140°F. The same builder additions to 0.00363M PSML had no effect on solubilization at 180°, 160°, and 140°F.

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• Letter to the Editor

Triglyceride Elution by Gas Chromatography

WE WISH TO REPORT a new development in gas-liquid phase chromatography (GLPC) analysis of fats and oils, not previously reported in the literature. We have successfully eluted triglycerides, through tristearin, with quantitative interpretation through trimyristin, and qualitative through tristearin, including most common edible oils (Figure 1).

Our initial studies gave us separation of the simple triglycerides, with both quantitative and qualitative interpretation, tributyrin through tripalmitin in polyester-packed columns ["Craig" polyester—ethylene glycol-succinate MW~1000, 30% on Chromosorb "W" (30/60 mesh), 18-in. column, operating at 250°C., 50 ml./min. He flow]. Because of the degradation of the polyester we have not attempted to operate these columns higher than 300°C.

We have extended the elutions through tristearin, using a silicone liquid phase at temperatures up to 350°C. The silicone used was fractionated from Dow Corning high-vacuum grease dispersed in ethyl acetate. A typical column used in this phase is an 18-in. stainless steel tube, packed with 30% silicone on Chromosorb "W" (30/60 mesh), operating at 300 to 350°C. with 50 to 200 ml./min. He flow. Table I shows a typical duplicate analysis of a synthetic mixture. Figure 2 shows typical log-retention plots for the columns used.

Despite what appears to be moderate degradation we have repeatedly reproduced the "fingerprint" of

TABLE I
Quantitative Interpretation of GLPC Elution,
Tributyrin Through Trimyristin

Triglyceride	Standard Mixture		Curve I area %	Curve II area %
	Mole %	Weight %		
Butyric.....	14.9	8.11	8.15	12.2
Hexanoic.....	10.3	7.18	11.2	11.8
Octanoic.....	8.54	7.25	14.2	10.0
Decanoic.....	17.9	17.93	21.2	23.4
Lauric.....	22.3	25.71	29.7	26.0
Myristic.....	25.9	33.82	15.3	16.4

edible oils, and the curves given in Figure 1 appear as excellent qualitative elution curves.

The edible oils containing a preponderance of lower-molecular-weight fatty acids (butter and coconut) give elution curves without degradation problems. It is interesting to note that butter oil shows 17 peaks, all reasonably symmetrical, and coconut 15 peaks, indicating 17 and 15 different molecular-weight triglycerides for these oils, respectively.

The triglycerides used in this work were prepared by standard ZnCl₂ catalyzed esterification, followed by distillation and/or crystallization.

The instrument is based on the Aerograph (Wilkins Instrument Company) circuitry with conventional oven and cell geometry. The cell is Gow-Mac TEIII Model 9230, with four tungsten filaments operating at ≈ 200 ma, 12 volts DC.

Column temperatures for the work ranged from 229 through 350°C., cell temperatures were kept at or slightly above column temperatures, injection and

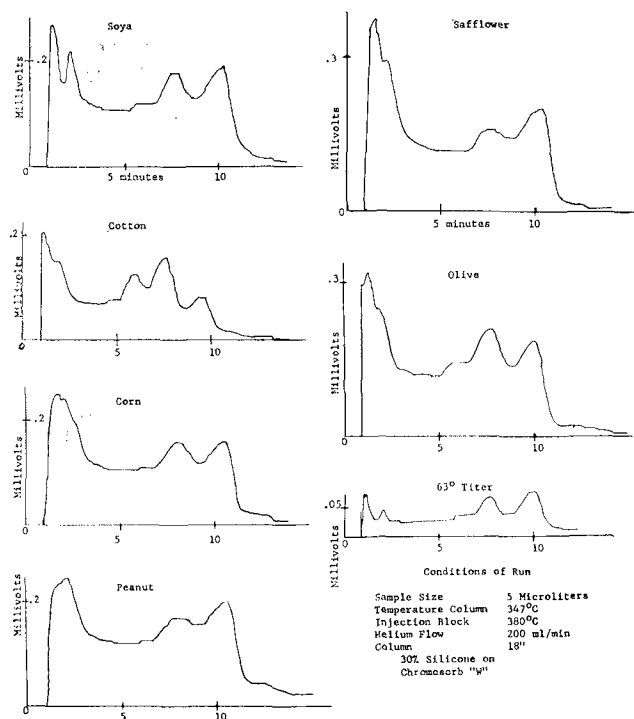


FIG. 1. Elution curves of some refined oils. GLPC runs.

exit heaters were held from 300 to 520°C. Helium flow rates were varied from 40 ml./min. to 200 ml./min. For any given run temperatures were held $\pm 2^\circ\text{C}$. and helium flow ± 1 ml./min.

The main problem in obtaining acceptable elution curves of the higher homologs is in vaporizing the oil without thermal decomposition. For triglycerides up to trimyristin this was obtained by increasing the injection block temperature. However the higher homologs do not respond as well, and increasing flow rate must be combined with the higher temperatures to obtain the desired result even though these changes have not yet given good curves for molecular weights above 800.

In making the quantitative estimates given in Table I, no corrections were made for the purity of

the individual components although individual compound elution showed minor contaminants in every case. The area of each peak was compared as a percentage of the sum of the areas of all the peaks in the data given in Table I.

$$\% n = \frac{A_n}{\sum A_1 \dots A_n} \quad A_n = \text{Area of Peak } n$$

F. H. FRYER, W. L. ORMAND, and G. B. CRUMP,
Research Laboratory, Margarine Manufacturing Division,
Standard Brands Inc., Indianapolis, Ind.

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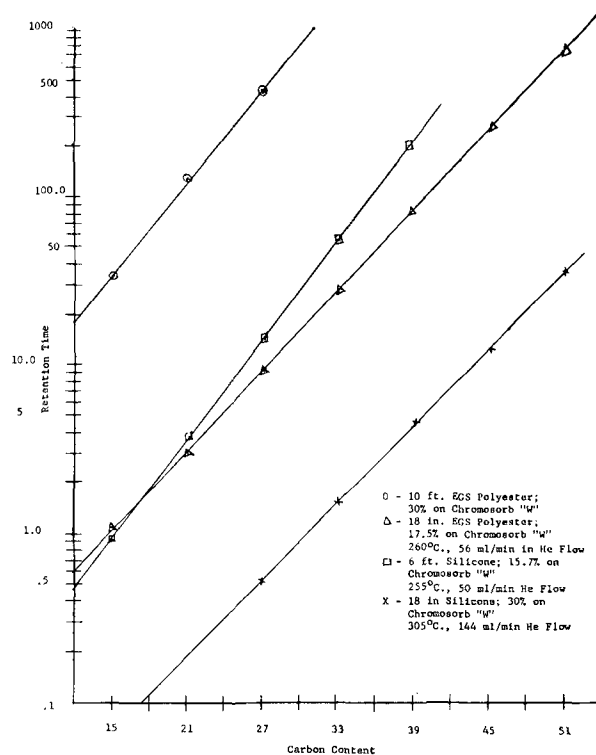


FIG. 2. Log retention-time vs. carbon content. Triglycerides GLPC.