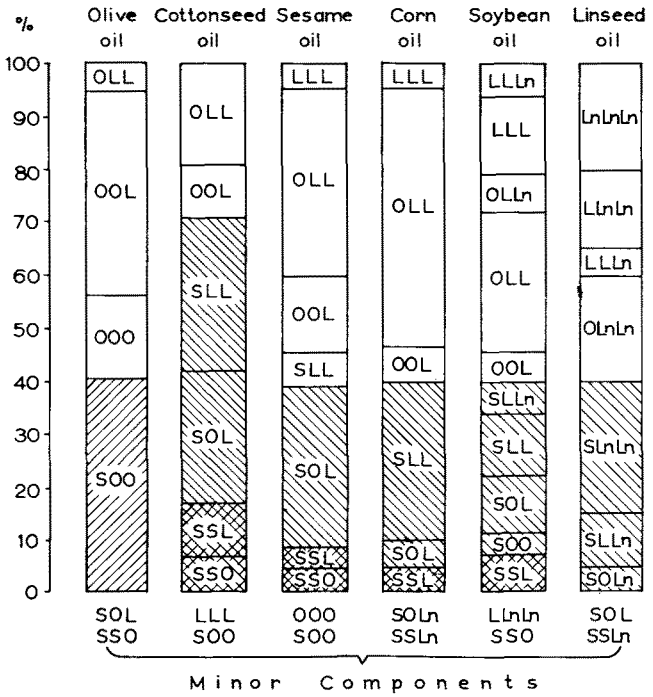

FIG. 1. Range of triglyceride types of some typical samples of plant oils, presented downwards in order of decreasing solubility in organic solvents. The percentage of each type is schematically given by the relative height of its strip. Single-shaded: SUU; Cross-shaded: SSU.

amounts of the various fatty acids.

While conventional low temperature crystallization can be used to detect adulteration, it is a tedious procedure. We overcame the disadvantages of the procedure by simplifying the technique. All crystallizations were carried out at 0C to 1C for at least 8–10 hours and, instead of modifying the temperature, various acetone-methanol mixtures of increasing polarity were used. Adulteration of olive oil is the most important case. Therefore, we devised the method described below permitting the certain identification of seed oils in olive oil even in cases of adulteration below 5%. Similar methods can obviously be worked out for other cases.

The oil (2 ± 0.1 g) is placed in a cylinder containing 25 ml of methanol-acetone (1:4) and kept in the refrigerator overnight. The supernatant solution is decanted. The insoluble fraction is dissolved in 12 ml of methanol-acetone (1:7) and kept at 0C for at least 8 hours. The supernatant solution is removed as completely as possible by decantion. The crystallized material (Fraction I) is dissolved in 8 ml of methanol-acetone (1:9), an aliquot is removed for gas-liquid chromatographic (GLC) analysis, and the rest is kept at 0C for 8–10 hours. Then, the supernatant solution is removed by decantion, while the crystallized material (Fraction II) is analyzed for fatty acids.

The fatty material crystallizing from olive and cottonseed oils is plotted against the composition of solvents in Figure 2. GLC analysis of fatty acids of the fractions thus isolated indicated that, although incomplete fractionation was achieved by this procedure, each branch of the curves thus formed corresponds mainly to one type of triglyceride. For example, as the methnol concentration is increased from 0 to 7%, the solubility of SSU of cottonseed oil decreases to a much greater extent than the solubility of SUU, thus resulting in a change of the slope of the curve at the point corresponding to methanol 7% because under

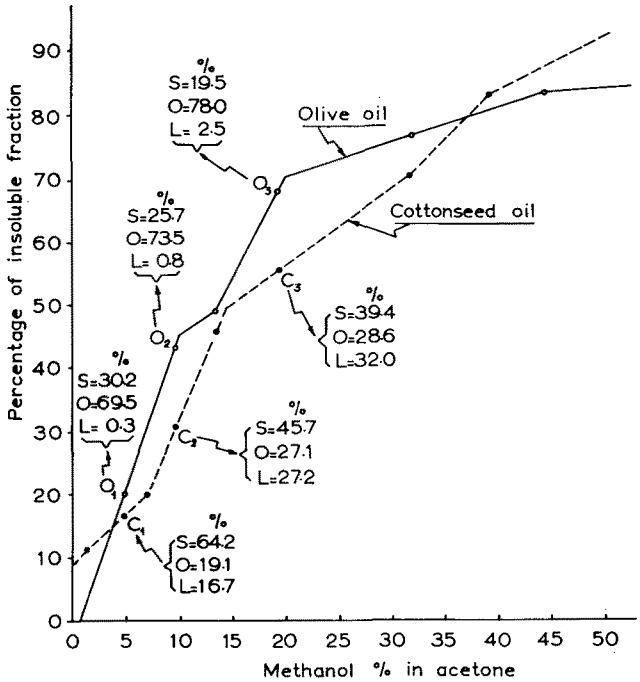

FIG. 2. Solubility data of olive oil and cottonseed oil in 9-fold amounts of several methanol-acetone mixtures at 0C. Fatty acid compositions of some characteristic fractions are included.

these conditions (9 volumes of solvent, 0C) almost all SSU crystallizes out. This is indicated by the fatty acid pattern of fraction C_1 of cottonseed oil (Fig. 2) from which a content of at least 92.5% of SSU can be assumed for the fraction.

In Table II some results of such analyses are given. Fraction I of pure olive oil contains mainly SOO accompanied by small amounts of OOO, OOL, etc. Consequently, its oleic acid content is always above 70% (usually 72–74%), while the linoleic acid content of this fraction never exceeds 5%. If olive oil is adulterated by an admixture of seed oils, the presence of SSL, SOL, SLL (SSO) lowers the oleic acid content of this fraction below 67%, while its linoleic acid content is increased above 10% for an adulteration of 5% only (Table II). On the other hand, the linoleic acid content of Fraction II was found below 2% for pure olive oils containing small or large amounts of linoleic acid, while in adulterated olive oils the linoleic acid content of the respective fraction is always higher than 5%. The examination of Fraction II is especially helpful for the detection of adulterations lower than 5%.

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• *Addendum*

"Determination of the Weight of Bulk Oil Shipments," JAOCS, February, 1965. On page 156, second column and second paragraph "1. Shore Tank Calibrations," delete the last two sentences, beginning "Further, this error" and ending with "the last compensation will be."