methyl esters, such as that from rat fat, to be separated and collected in less than 25 min. The sample size that can be efficiently separated and collected depends, of course, upon the number of components and the concentration of each sample. Samples that contain solvents or components with high vapor pressures make injections without loss of sample difficult because of the back pressure they create. In spite of these losses, recoveries in excess of 90% have been obtained from approx 200 μl injections of solvent-free methyl laurate in recovery tests.

We have been unable to satisfactorily separate large samples according to degree of unsaturation using diethylene glycol succinate polyester (DEGS) or other polar phases. This difficulty is circumvented by preliminary separation according to carbon number followed by separation according to degree of unsaturation by silver nitrate thin-layer chromatography (TLC).

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REFERENCES

1. Meinertz, A., and V. P. Dole, J. Lipid Res. *3*, 140 (1962).
2. Hajara, A. K., and N. S. Radin, *Ibid. 3*, 131 (1962).
3. Schlenk, H., and D. M. Sand, Anal. Chem. *34*, 1676 (1962).

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Improved Techniques in Neutral Oil Determination

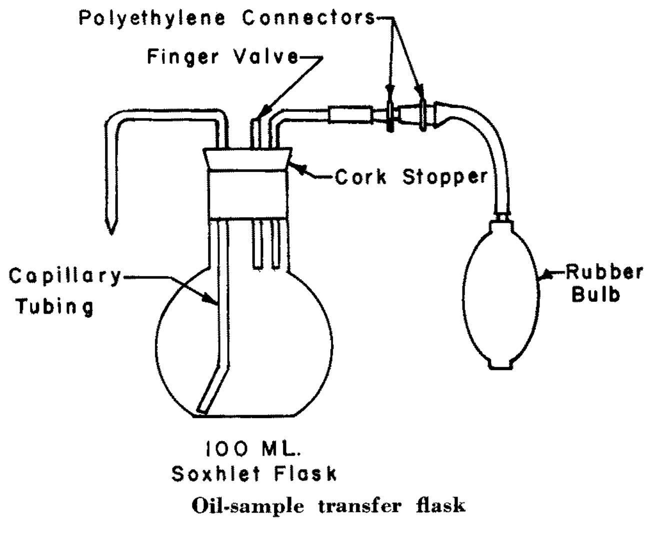
Because of the recent adoption of neutral oil as a basis for price settlement of crude soybean oil, this analysis is becoming routine in vegetable-oil laboratories.

The following techniques in preparing the alumina column and the transfer of oil sample into the chromatographic tube have been found, by this laboratory, to simplify and improve neutral oil determinations. These techniques have been used for some time and have been modified to accommodate the new glassware specified in AOCS Method Ca 9f-57.

A. Preparation of the Alumina Column

Activated alumina (20 g ± 1 g) is carefully poured through a small bore funnel into a 125 ml separatory funnel which has been half-filled with ether-methanol solvent. The alumina is allowed to settle to the bottom of the separatory funnel before dispensing into the solvent-filled chromatographic tube. The chromatographic tube is maintained at near-full solvent level by adjustment of the funnel and tube stopcocks. The top of the alumina column is made level by closing the tube stopcock and tapping the tube. A full head of solvent in the chromatographic tube aids this adjustment.

This technique simplifies transfer of the alumina and improves the formation of the alumina column. Heat of reaction between the alumina and the ether-methanol solvent is sometimes responsible for formation of ether bubbles which tend to rise and break up the alumina column. This heat is initially dissipated in the solvent contained in the separatory funnel.



Oil-sample transfer flask

Further heat dissipation and release of ether bubbles in the alumina slurry occurs as the slurry is allowed to fall through the solvent in the chromatographic tube.

B. Transfer of Sample

A weighed sample of crude oil is transferred quantitatively onto the alumina column by means of a pressure flask as shown in diagram.

The transfer device consists of a cork of suitable size, for a 100 ml Soxhlet flask, fitted with three pieces of glass tubing.

1. A delivery tube; capillary bore.
2. Pressure inlet tube; fitted with a short piece of rubber tubing and polyethylene connector.
3. Finger valve tube.

A polyethylene connector is attached to the tubing end of a rubber atomizer bulb (pressure type with two valves).

The crude oil sample is weighed into the flask and dissolved with 10 ml ether-methanol solvent. The transfer device is placed in the flask and the delivery tube is adjusted to reach the bottom and side of flask. The flask is then positioned above the alumina column so that the stream from the delivery tube will be directed well inside and on the side of the chromatographic tube. Forefinger is placed over the glass valve and the rubber bulb is gently squeezed and pressure maintained until the flask is empty. Delivery tube is rinsed inside column and the rubber bulb is disconnected.

The flask is rinsed by adding 10 ml of solvent and washing down the sides of the flask with solvent from a squeeze bottle. The flask is rinsed three times into the chromatographic tube and the remaining 100 ml of solvent is added using the 50 ml burette.

Results of the neutral oil analysis of a sample of crude soybean oil with and without the improved techniques were compared. Twelve determinations were made, six by the regular AOCS Method, and six using the outlined techniques.

The mean recovery of neutral oil was 0.21% higher using the improved techniques, with a standard deviation of ± 0.11% as compared to ± 0.14% by the AOCS Method.

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