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NOTE

Separation of Oleic Acid from Fatty Acid Impurities

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Abstract

A method of oleic acid purification is described. The method consists of the following five steps: 1) cooling of the sample to 4°C for a partial separation of palmitic acid by crystallization, 2) distillation at reduced pressure (0.8 mmHg) for removal of lauric and myristic acids, 3) crystallization of stearic and palmitic acids from acetone at −25°C, 4) separation of oleic acid from palmitoleic and linoleic acids by oleic acid crystallization from aqueous methanol solutions at −10°C, 5) reduced pressure (0.5 mmHg) distillation of the resulting oleic acid sample for removal of water and methanol. By utilizing the procedure described above, a sample containing only 82% oleic acid was refined to a product containing 98.7-98.9% oleic acid.

INTRODUCTION

Oleic acid, a common constituent of vegetable oils, is widely used in many industrial fields including mineral processing. The properties of oleic acid have been extensively studied in various laboratories due to the existence of a number of different chemical species in aqueous solutions (bulk solutions, micellar solution, liquid crystals, soaps, acid soaps, etc.). Any investigation of the oleic acid properties, without leaving any margin for artifacts which may be caused by impurities, requires using high purity oleic acid as a reagent. Oleic acid is readily available from various sources (*1*). However, high purity oleic acid is usually expensive.

Furthermore, in some cases the purity of the available oleic acid is not as high as claimed. For these and other reasons, one has to purify oleic acid which is usually contaminated with various long-chain fatty acids.

A very satisfactory procedure for purifying oleic acid was proposed by Brown and co-workers (*2*, *3*). The method involves separating oleic acid from saturated acids and linoleic acid by a series of crystalliations from acetone at low temperatures. Details of the Brown method are given elsewhere (*2–4*). Low-temperature crystallization of free acids from various organic solvents has been applied by others (*5–7*). Oleic acid can also be purified by means of molecular sieves (*8*, *9*) and zeolites (*10*, *11*).

There are other methods for purifying oleic acid. These indirect procedures require forming either lead salts (*12*, *13*), sodium salts (*14*), methyl esters (*15*, *16*), mercuric acetate (*17*), urea complexes (*18–21*), hydroxylated compounds (*22*), or oxidative polymerization products (*23*) before selective crystallization is carried out.

In 1984–87 during an investigation of the chemistry of oleic acid in aqueous solution (*24–26*) and in aqueous emulsions (*27*), yet another procedure for purifying oleic acid was worked out. This procedure is described below.

EXPERIMENTAL

The sample of oleic acid used for purification was purchased from International Enzymes Ltd. As may be seen from a chromatogram of the material (Line a in Fig. 1), it contained, in addition to oleic acid, significant amounts of saturated acids (myristic, palmitic, stearic, lauric) and unsaturated acids (palmitoleic, linoleic).

The purity of various fractions produced during separation of oleic acid from the other acids was monitored by means of a gas–liquid Perkin-Elmer chromatograph equipped with a 1000 × 3 mm i.d. column. The packing of the column was 2% diethyl glycol succinate ester on Chromosorb G. The flow rate of nitrogen was 48 cm^3/min, column temperature 190°C, detection FID. The oleic acid content of a sample was calculated from the chromatogram of the sample by means of the equation: oleic acid % = (area of oleic acid peak/area of all peaks) × 100%.

PURIFICATION PROCEDURE

A 1.0-dm^3 sample of oleic acid was kept at 4°C for 3 days and filtered by means of a Schott G3 funnel. This operation was repeated several

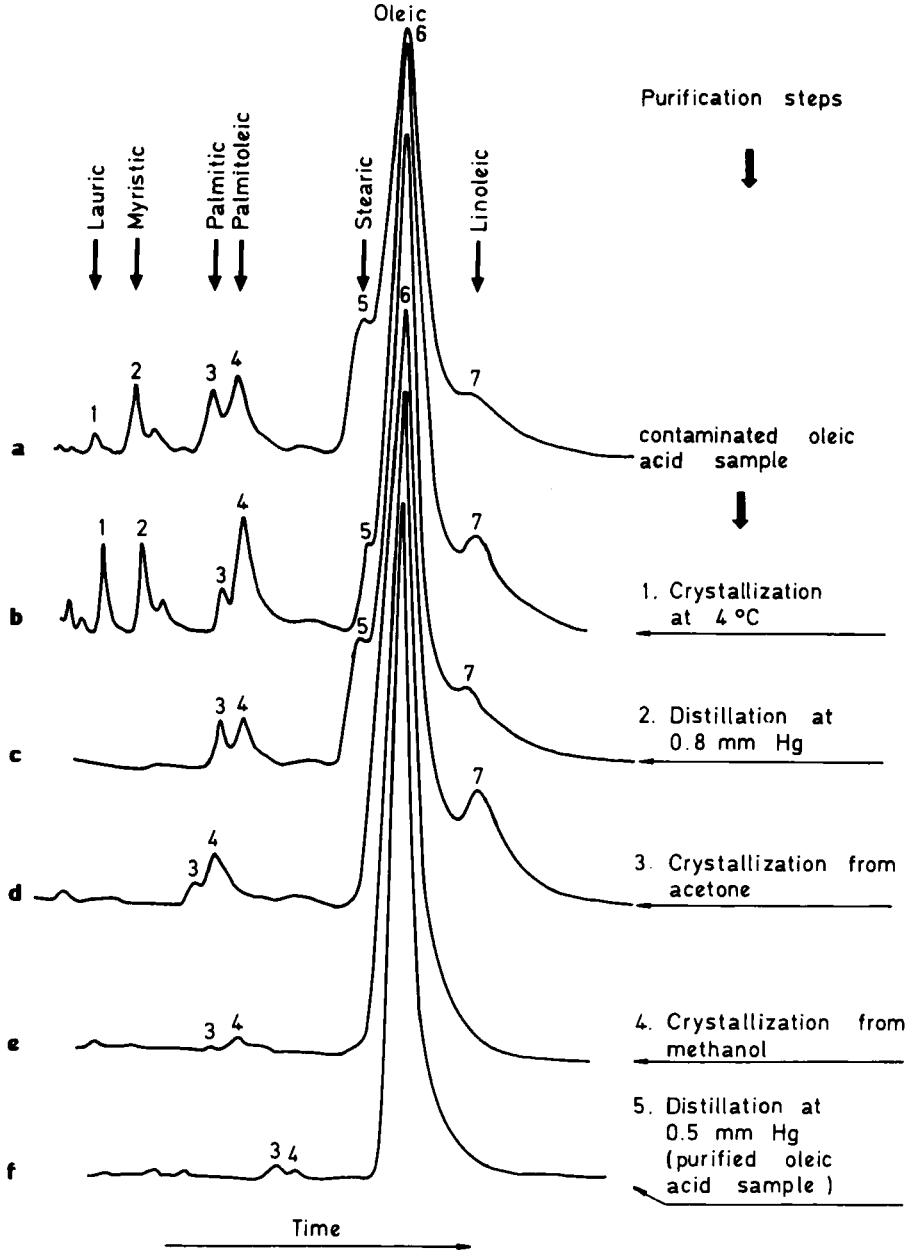

FIG. 1. Chromatograms illustrating five different purification steps of oleic acid containing other long-chain fatty acids.

times until no further solids appeared in the sample. The resulting crystal fraction contained saturated fatty acids, mainly palmitic acid. The filtrate was subjected to fractional distillation at a reduced pressure (0.8 mmHg) under a nitrogen atmosphere in a 75-cm long by 1.5 cm diameter Vigreux column equipped with a heating coating. The oleic acid fraction boiling at 182–184°C was collected and cooled. As seen from Chromatogram c in Fig. 1, this separation step accomplished virtually a complete removal of lauric and myristic acids from the sample. The resulting oleic acid was about 90% pure.

A 250-g sample of distilled oleic acid was made up to 1000 cm^3 with acetone and left overnight at −25°C in a Dewar cell containing a mixture of acetone and dry ice. The resulting crystals of stearic and palmitic acids were removed by filtration and rejected. Next, the acetone was removed from the filtrate by evaporation under reduced pressure. The solid product was dissolved in 650 cm^3 of 90% aqueous methyl alcohol solution and cooled to −10°C. The crystal crop was oleic acid in a needle-like form. The crystals were rinsed twice on a Schott funnel with 90% aqueous methanol cooled to −30°C. The materials remaining in the filtrate were palmitoleic and linoleic acids. To remove excess water and methanol from the purified oleic acid, a final distillation of oleic acid was carried out under reduced pressure (0.5 mmHg) and 170°C. The resulting oleic acid was colorless and odorless. Its refractive index (n_D^{20}) was equal to 1.4602 and its iodine number to 89.5. It was determined from Chromatogram e in Fig. 1 that the final refined oleic acid was 98.7–98.9% pure.

CONCLUSIONS

A procedure is presented for separating long-chain fatty acids from oleic acid which consists of a combination of fractional distillation and crystallization from acetone and from methanol. This procedure seems to be much simpler than other methods presented in the literature. The sequence of refining stages described above is probably not crucial for determining the final purity of oleic acid, and therefore the method may be modified to some extent. For instance, the first step (partial separation of palmitic acid by crystallization at 4°C) can be omitted because this acid can be removed later in the fourth step during crystallization from methanol. The method of separation is capable of producing oleic acid having a purity of 99%.

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