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Determination of Hexadecanoic and Octadecanoic Acids in Stearin for Industrial Use by Reversed-Phase Ion Suppression High-Performance Liquid Chromatography

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**DETERMINATION OF HEXADECANOIC AND
OCTADECANOIC ACIDS IN STEARIN FOR
INDUSTRIAL USE BY REVERSED-PHASE ION
SUPPRESSION HIGH-PERFORMANCE
LIQUID CHROMATOGRAPHY**

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ABSTRACT

A rapid and simple high-performance liquid chromatographic (HPLC) method was developed for the simultaneous determination of hexadecanoic and octadecanoic acids in stearin. The samples are dissolved in ethanol without any additional pretreatment. HPLC was carried out on a MicroPak MCH-5 column (15cm × 4mm I. D.) using water (pH 2.5 with perchloric acid) - acetonitrile (10/90 V/V) as the mobile phase with UV detection at 210nm.

INTRODUCTION

Stearin for industrial use, made from animal and vegetable fats, consists chiefly of a mixture of octadecanoic and hexadecanoic acids. It is widely used in manufacture of surfactant, cosmetics, rubber and plastics, etc⁽¹⁾.

The quality grade of stearin was defined by the acid, saponification, iodine values and solidifying point⁽²⁾, which can only represent the total amount of hexadecanoic and octadecanoic acids, as well as that of impurities, but can not directly give the respective amount of hexadecanoic and octadecanoic acids. In recent years it was found that not only the total amount of hexadecanoic and octadecanoic acids, but also the ratio of octadecanoic to hexadecanoic acid has a conspicuous influence upon the technological process, products quality and economic profits.

Higher fatty acids are traditionally separated and determined using derivatization followed by either gas or liquid chromatography⁽²⁻³⁾. The derivatization step is usually time-consuming and requires several steps prior to injection. HPLC coupled with variable wavelength detection at 210nm provided a technique capable of direct determination of hexadecanoic and octadecanoic acids in stearin without chemical derivatization.

EXPERIMENTAL

Reagents and Chemicals

Acetonitrile was HPLC grade (Institute for Fine Chemical Engineering of Huaiyin Plastic Product Factory, Huaiyin, Jiangsu, PRC; WHO Collaborating Center for Research in Human Reproduction, Tianjing, PRC).

Hexadecanoic acid and Octadecanoic acid were GC grade (Shanghai No. 1 Reagent Factory, Shanghai, PRC).

Ethanol was analytical grade (Nanjing Chemical Reagent Factory, Nanjing, Jiangsu, PRC).

Water was distilled twice.

Stearin samples were provided by Nanjing Jinling Chemical Plant.

A stock solution of hexadecanoic acid (10mg/mL) and octadecanoic acid (10mg/mL) in ethanol was prepared.

Apparatus and Chromatographic Conditions

The instrument used for HPLC separations was a Varian VISTA 5060 equipped with a Varian UV-100 variable wavelength UV detector (Varian Instrument Division, Walnut Creek, CA, USA) set at 210nm with a sensitivity of 0.025 AUFS.

Chromatograms were recorded on a Yokogawa Hokushin Electric Type 3066 pen recorder (Sino-Japanese No. 4 Meter Factory, Chongqing, Sichuan, PRC) and a Shimadzu C-R1B integrator (Shimadzu, Tokyo, Japan).

The separation was performed on a MicroPak MCH-5 Column (15cm × 4mm I.D.; Varian Instrument Division). The mobile phase was water (pH 2.5 with perchloric acid) - acetonitrile (10/90 V/V) at a flow - rate of 1.5 mL/min. The water is adjusted to a pH of 2.5 with perchloric acid to suppress ionization of the acids and provide better retention. The column temperature was maintained at 50°C to increase the solubility of these higher molecular weight acids in the mobile phase.

Procedure

Weigh out 0.5 gram of sample accurately into a 100-mL volumetric flask and diluted to volume with ethanol. Take 1 mL of sample solution and filter through a syringe filter with 0.5 µm micropore. 10 µL aliquots of the filtered solution were injected to the HPLC column.

The standard substances were dissolved the same way as the samples.

Calibration Curve

Standards at concentrations of 2-10mg/mL of hexadecanoic and octadecanoic acids were prepared by serial dilutions of the stock solution with ethanol. A

calibration graph for hexadecanoic and octadecanoic acids were obtained by measurements of peak heights.

RESULTS AND DISCUSSION

Chromatogram and Calibration Graph

Typical HPLC chromatograms for the hexadecanoic and octadecanoic acids in standard and stearin sample are given in Figure 1. Hexadecanoic and octadecanoic acids were clearly separated.

The quantitation was based on a calibration by series of dilution from primary standard. A linear regression analysis of the relationship between peak height versus amounts of standards was carried out within the range 20-100 μ g in 10 μ l injection volume. The results obtained were; $y=2.598x-0.202$ for hexadecanoic acid and $y=1.605x-0.146$ for octadecanoic acid, with correlation coefficients of 0.9991 and 0.9990 respectively, where y equaled peak height (cm) and x equaled final standard concentration (mg/mL).

Analysis of Industrial Samples

Table 1 gives results obtained by the proposed method in comparison with the iodine values. The results showed that the amounts of hexadecanoic and octadecanoic acids in stearin is consistent with the quality grade and the iodine value. On one hand the amount of hexadecanoic acid in the grade 1 and 2 is higher than that in the grade 3, and the amount of octadecanoic acid is reversed, the amount of hexadecanoic acid in the grade 1 is smaller than that in the grade 2, and the amount of octadecanoic acid is reversed, on the other hand the total amount of hexadecanoic and octadecanoic acids in the grade 1 (iodine value=2) is a bit higher than that in the grade 2 (iodine value=4), much higher than that in the grade 3 (iodine value=8). The amount of impurities in stearin for industrial use, which are chiefly unsaturated higher fatty acids such as oleic, linoleic and linolenic acids, gives

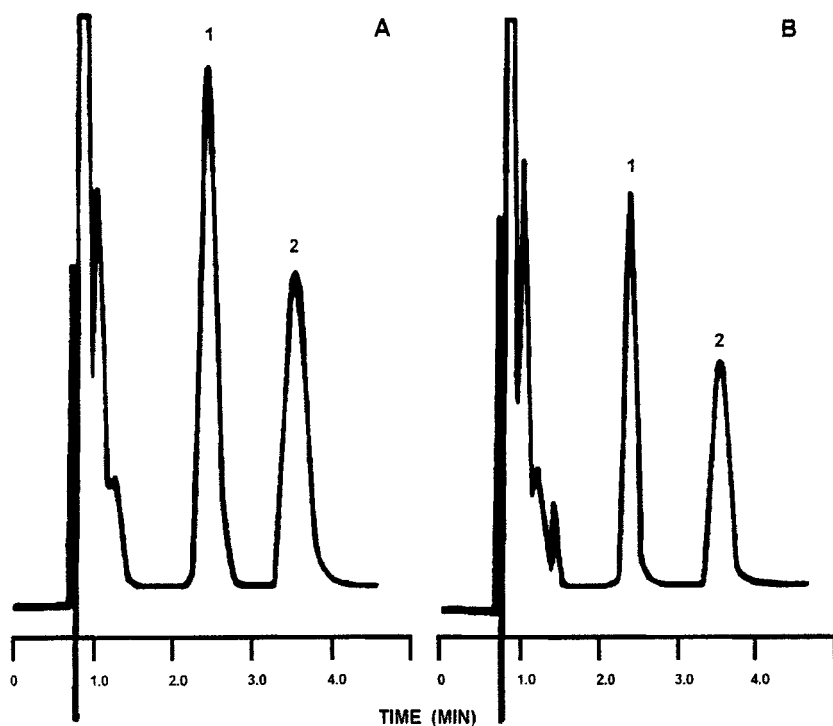


FIGURE 1. Chromatograms obtained from (A) standard, and (B) stearin (grade 3). Peaks identified are (1) hexadecanoic acid, (2) octadecanoic acid.

TABLE 1

Analysis of Hexadecanoic and Octadecanoic Acids in Stearin for Industrial Use

Quantity grade	Hexadecanoic acid* H	Octadecanoic acid* O	Total H+O	Rotio O/H	Iodine Value
1	53.70 ± 0.69	46.24 ± 0.70	99.94	0.86	2
2	58.35 ± 0.65	40.47 ± 0.75	98.82	0.69	4
3	31.83 ± 0.78	61.76 ± 0.55	93.59	1.94	8

* Each value in percent (m/m) represents the mean of the five samples ± standard deviation.

expression to the unsaturated value that was traditionally measured by adding iodine.

CONCLUSION

In this paper, an attempt was made to present a new method for determining the hexadecanoic and octadecanoic acids in various types of strearin. The simplicity and short time of the analysis make it a convenient alternative over earlier methods.

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