Detection of Spermaceti in a Hand Cream

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ABSTRACT

The presence of spermaceti, an embargoed item, was confirmed in a sample of hand cream detained by federal agents. The identification was based on chromatographic characteristics of the intact wax esters and of their component fatty acids and alcohols. These acids and alcohols were combined in a manner so closely resembling spermaceti that they undoubtedly came from this source.

INTRODUCTION

In the early 1970's, sperm whales were placed on the list of endangered species and products from these whales were excluded from the United States by embargo. Since then, the Northern Regional Research Center has been called upon to determine whether or not sperm whale products were present in several different kinds of commercial items. The present example concerns the analysis of a hand cream detained upon importation by agents of the National Marine Fisheries Service of the U.S. Department of Commerce.

EXPERIMENTAL PROCEDURES

Approximately 1 g of the hand cream was placed on a 50 x 1.5 cm column containing 20 g of Hi-Flosil (Applied Science Labs). The elution solvents were: benzene (270 ml), 5% Et_2O in benzene (180 ml), and 20% Et_2O in benzene (150 ml). Fractions (30 ml each) were collected and the progress of the chromatography was monitored by thin layer chromatography (TLC). Fraction 2 from this chromatography was reapplied to the column and eluted with 200 ml of benzene:hexane (1:1). This time, 10-ml fractions were collected.

Precoated plates (0.25 mm, Silica Gel 60 F-254, EM Laboratories) were used for TLC with benzene or benzene: hexane (1:1) as the developing solvents. Preparative layer chromatography (PLC) was carried out on 2-mm layers with the mixed solvent system. Bands were made visible under ultraviolet (UV) light by spraying the plate with a 2% solution of dichlorofluorescein in EtOH. They were scraped from the plate and recovered from the adsorbent with warm Et_2O.

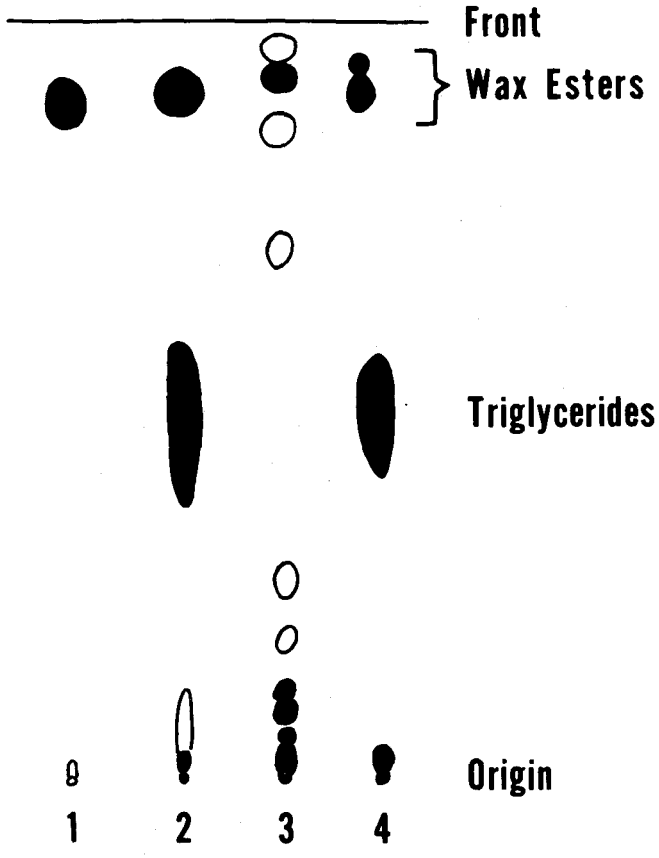

FIG. 1. TLC on silica gel, development in benzene. Lanes: 1 = spermaceti, 2 = sperm whale oil, 3 = beeswax, 4 = hand cream. Shaded regions indicate darker spots.

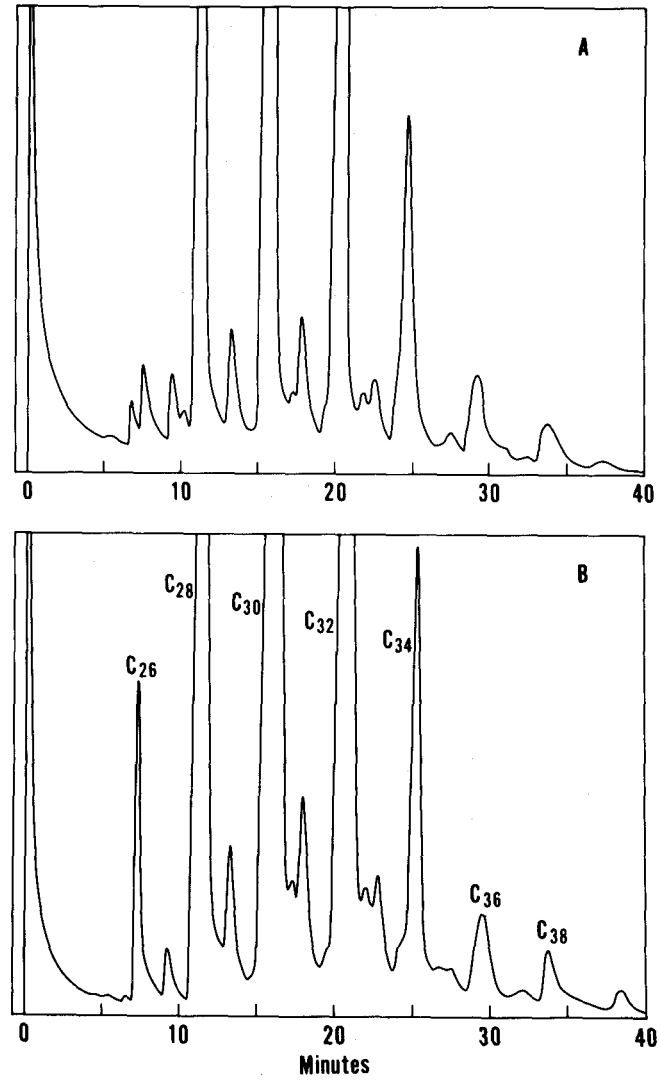

FIG. 2. GC of lower-eluting wax esters from the hand cream (A) and spermaceti (B). Column: 100 x 0.30 cm stainless steel packed with 3% OV-1 on Gas Chrom Q. Temperature programmed from 200 C to 300 C at 2 C/min.

TABLE I

Isomeric Composition of Wax Esters by Chain Length

Chain length	Acid-alcohol combination	Relative abundance (%)	
		Hand cream	Literature (3)
28	12-16	83	85
	14-14	17	15
30	12-18	9	8
	14-16	79	82
	16-14	12	9
32	14-18	15	19
	16-16	82	79
	18-14	3	2

For high-pressure liquid chromatography (HPLC), a Waters ALC201 chromatograph was equipped with a μ-Bondapak C_{18} column (30 x 0.78 cm, Waters Assoc.) and a differential refractometer. The solvent was acetone-acetonitrile (2:1). GC analytical methods have previously been described (1) Hydrolysis/methanolysis of wax esters and triglycerides was done in NaOH/MeOH followed by BF_3/MeOH (1). Alcohols were converted to trimethylsilyl ethers with bis(trimethylsilyl)triflouroacetamide.

RESULTS AND DISCUSSION

Inconsistencies in the labeling information (spermaceti was listed as an ingredient on some labels and not on others) caused the agents to request confirmation that the hand cream contained spermaceti. Beeswax was also among the ingredients, along with some odor-causing constituents. A preliminary examination of the hand cream by TLC (Fig. 1) showed spots corresponding to spermaceti and sperm oil wax esters, as well as to sperm oil triglycerides and beeswax components. Gas chromatography (GC) of the hand cream and standards confirmed the tentative identifications made by TLC and showed peaks in the waxester and triglyceride regions of the curves.

The first column chromatography separated the wax esters and triglycerides from the more polar components. A second pass of fraction 2 separated the wax esters from the triglycerides and yielded fractions composed primarily of the lower eluting wax esters. These were the components corresponding to spermaceti by both TLC and GC. In order to rule out the presence of beeswax esters in these fractions, a beeswax sample was separated by PLC. The wax esters from beeswax were identical to those of the higher eluting wax esters from the hand cream, and no components corresponding to the lower eluting wax esters were found in beeswax. Beeswax wax esters migrate slightly further on TLC than spermaceti, ostensibly due to their longer chain length (2).

The lower eluting wax esters from the hand cream were virtually indistinguishable from spermaceti by GC (Fig. 2), differing only in the relative proportions of a few minor components (e.g., the C_{26} wax esters). Further separation by chain length (HPLC) yielded fractions composed primarily of the C_{28}, C_{30}, and C_{32} wax esters. Hydrolysis/methanolysis followed by silylation and GC was used to determine the combinations of alcohols and fatty acids in each fraction. This technique was earlier used to determine the isomeric combinations in the wax esters of jojoba oil (1). The combinations found for each chain length (Table I) closely resembled those published for spermaceti (3).

The above information on TLC migration characteristics, GC retention data and the relative abundance of the isomers is strong evidence that spermaceti was used in the hand cream formulation. The likelihood that wax esters were obtained in these chain lengths and combinations from synthesis or from another source is infinitesimal. Precise quantitation of the spermaceti in the hand cream was not sought (the mere presence constitutes a violation), but it appeared to represent about 5% of the mixture. GC analysis of methyl esters derived from the triglycerides, the most abundant species in the sample, showed them to be virtually indentical in composition to those of peanut oil.

ACKNOWLEDGMENTS

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