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[Received April 28, 1964—Accepted July 10, 1964]

Minor Component Fatty Acids of Common Vegetable Oils

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Abstract

A combination of gas-liquid chromatography (GLC) and oxidative cleavage on fractions isolated by mercury derivative chromatography has shown the presence of previously unreported minor component fatty acids in olive, soybean, cottonseed, corn, peanut, rapeseed and safflower oil. All of the oils examined contain small amt of saturated acids above arachidic, some as high as hexacosanoic acid. *Cis*-11-octadecenoic acid was found in amt ranging from 0.5–2.0%. *Cis*-11-eicosenoic acid is present in the 0.04–1.4% range (rapeseed oil excluded). The tetracosenoic acid present in rapeseed (0.4%) and safflower oil (0.1%) has been identified as the *cis*-15-tetracosenoic acid. No unusual polyenoic species were detected with the exception of those in rapeseed oil, which contains 0.6% of both 11,14-eicosadienoic and 13,16-docosadienoic acid.

Introduction

THE SUGGESTION THAT the unsaturated acids of common vegetable oils contain isomers or homologs of oleic and linoleic acid has appeared sporadically in the literature (1,4,6,14,19). These reports have attracted little attention, most probably because of the limited amt of supporting data presented.

A few well-documented reports of unusual fatty acids in common oils have appeared. The eicosenoic acid (1–2%) present in peanut oil has been identified as *cis*-11-eicosenoic acid (7,8). The presence of 15% *cis*-11-octadecenoic acid and 10% *cis*-9-hexadecenoic acid in milkweed seed oil has been shown (3). Although not a common oil, milkweed seed oil is a good example of an oil whose major unsaturated components had been thought to be oleic and linoleic acid. Also pertinent to the present work are the recent reports by GLC of tetracosenoic acid (1%) and eicosadienoic acid (0.5%) in rapeseed oil (13,15) and eicosenoic acid (trace) in safflower oil (9).

The ability of present-day separation and analysis techniques, when used in a co-ordinated analytical scheme, to detect small amt of geometrical and positional isomers prompted a new look at common edible oils whose fatty acid composition has been accepted for many years. Olive, soybean, cottonseed, corn, peanut, rapeseed and safflower oil were analyzed. The oils were randomly selected as representative of high quality edible products but their exact origin is unknown.

Experimental

General Analysis Scheme. The methyl esters of the oils (all refined and bleached except olive oil) were prepared by transesterification (10). Mercury derivatives of the methyl esters were prepared and subsequently separated into saturated, monoenoic and polyenoic fractions by column chromatography as previously described (11). The resulting fractions were analyzed by IR spectroscopy (2), GLC and oxidative cleavage. In many cases the monoenoic methyl esters were subjected to further column chromatography on silver nitrate-silica gel adsorbent, using a variation of de Vries' technique (5), to obtain enrichment of a minor component.

GLC Analysis of Fractions. Three types of columns were used to analyze the fractions isolated by column chromatography. Column A was a 24 x ¼ in. stainless steel column packed with 20% Dow-Corning 200 silicone fluid (12500 centistokes) on 60–80 mesh acid-washed Chromosorb W and was operated at temp of 230 or 280C with a helium flow rate of 95–100 ml/min at STP. Column B was a 72 x 3/16 in. aluminum column packed with 15% organosilicone-ethyleneglycol succinate on 100–200 mesh Gas-Chrom P (Applied Science Laboratories, Inc. EGSS-X) and was operated at a temp of 180–190C with a helium flow rate of 35–40 ml/min at STP. Column C was a 120 x ¼ in. stainless steel column packed with 15% stabilized diethyleneglycol succinate (Analabs, Inc. C6) on 60–70 mesh Anakrom ABS and was operated at 170C with a helium flow rate of 34–36 ml/min at STP. All chromatograms were obtained on an F&M Scientific Corp.

TABLE I

Analysis of Oils by Mercury Derivative Chromatography

Oil	Olive	Soybean	Cottonseed	Corn	Peanut	Rapeseed	Safflower
Weight %							
Saturated acids	14.1	15.9	26.2	13.8	19.4	6.8	10.3
Monoenoic acids	78.2	28.3	18.2	27.4	52.4	70.5	15.0
Polyenoic acids	7.7	55.8	55.6	58.8	28.2	22.7	74.7

TABLE II

Chain Length Distribution of Saturated Acids by GLC of Saturated Fraction

Oil	Olive	Soybean	Cottonseed	Corn	Peanut	Rapeseed	Safflower
Weight %							
C_{12}			0.1	<0.1	0.3	0.3	<0.1
C_{14}	<0.1	0.6	2.9	0.3	0.3	0.9	1.2
C_{15}		<0.1		<0.1	<0.1		0.1
C_{16}	75.9	70.3	86.9	79.5	54.8	53.5	63.4
C_{17}				0.4		0.7	
C_{18}	20.1	25.3	8.2	14.6	13.9	20.1	28.3
C_{19}	<0.1			<0.1		0.2	
C_{20}	2.7	1.9	0.9	2.8	6.5	12.4	3.5
C_{21}	<0.1		<0.1	0.2			
C_{22}	0.7	1.9	0.5	0.9	15.2	10.6	2.1
C_{23}	0.1		<0.1				
C_{24}	0.4		0.3	1.2	7.6	1.3	1.4
C_{26}			0 2		1.4		

Model 500 instrument using a thermal conductivity detector.

Columns A and B were used to analyze fractions containing high mol wt species, such as all the fractions from rapeseed and safflower oil and the saturated fractions from the remaining oils. Column B was preferred since it gave adequate resolution of the saturated and unsaturated methyl esters without requiring excessively long retention times. Column C was used as an alternative to Column B to analyze many of the unsaturated fractions where high mol wt ($>C_{20}$) species were not present.

Known mixtures of saturated and unsaturated methyl esters run under similar conditions, as well as plots of logarithm of retention time vs. chain length, were used to identify the various components. Area response was multipled by a factor (square root of the mol wt) and normalized to obtain wt percentages. The standard deviation of replicate analyses at an average value of 0.10% is 0.05% and at an average value of 5.0% is 0.25%.

Oxidative Cleavage. A modified periodate-permanganate oxidation method was used to cleave the unsaturated fractions. Details of the oxidation and the subsequent programmed temp GLC of the products have been reported elsewhere (12). Both mono- and dicarboxylic acid cleavage fragments (C_4 and up) are recovered and determined quantitatively (as their methyl esters) by this method.

Results and Discussion

Table I gives the levels of saturated, monoenoic and polyenoic acids in the original oils as determined by mercury derivative chromatography. The purity of the isolated methyl ester fractions as to the degree of unsaturation was 98–99% for the saturated fractions, 95–99% for the monoene fractions (see Table III) and 97–99% for the polyene fractions, as determined by GLC on polyester columns.

The chain-length distribution of the saturated acids is given in Table II. Palmitic and stearic acids were the major components, as expected. The fatty acids above arachidic are noteworthy. The odd-numbered species and some of the tetracosanoic and hexacosanoic acid components have not been reported previously. (Data presented by Iverson and Firestone at the October 1963 AOAC Meeting, Washington, indicated that odd-numbered saturated acids are present in olive oil.) Peanut oil has recently been shown (8) to contain hexacosanoic acid (0.4%) and trace amt (0.002–0.03%) of odd-numbered saturated acids from 13–27 carbon atoms. These latter acids were not detected in the present sample. The tetracosanoic acid content of rapeseed oil found here is low compared to previous reports. The presence of behenic acid in safflower oil confirms an earlier report (4) based on boiling point and saponification value of a distillation cut.

Table III summarizes GLC data obtained on the monoene fractions. In most instances the minor high mol wt components listed in Table III were concn 4–20 times by column chromatography on silver nitrate-silica gel. The first material eluted from this adsorbent was richer in methyl eicosenoate and higher monoenoic esters than the sample placed on the column. GLC of this first eluate confirmed the presence of the minor components seen in the gas chromatograms of the original monoenoic esters.

The fragments produced upon oxidative cleavage of the monoene fractions are listed in Table IV. A typical methyl oleate cleavage pattern is included for comparison. These GLC results were obtained by a single programmed temp run on a dilute methanol solution of the oxidation products (after esterification). A gas chromatogram typical of the cleavage patterns given by the monoene fractions has been published (12).

The isomer distribution of monoenoic acids given in Table V was obtained by a combination of the data of Tables III and IV. All the monoene fractions gave significant amt of heptanoic and undecanedioic acid upon oxidative cleavage. In calculating the level of 11-octadecenoic acid, corrections for 9-hexadecenoic and 11-eicosenoic acid were necessary since they give common fragments. All the species reported in Table V are *cis* isomers, since no *trans* bonds were detected by the quantitative IR method.

The composition of rapeseed oil monoenoic acids is

TABLE III

GLC of Monoene Fractions from Mercury Derivative Chromatography

Oil	Olive	Soybean	Cottonseed	Corn	Peanut	Rapeseed	Safflower
Weight %							
C_{16} Me ester	0.2	0.6	0.8	0.4	0.2	0.1	0.4
$C_{16}{}^{1=}$ Me ester	1.1	0.4	2.0	0.4	0.2	0.3	0.4
$C_{17}{}^{1=}$ Me ester	0.2	0.2	0.3	0.1	0.1		
C_{18} Me ester		0.3					
$C_{18}{}^{1=}$ Me ester	98.2	96.4	94.9	96.7	96.4	22.1	92.2
$C_{18}{}^{2=}$ Me ester		1.1	1.3	1.4	0.5		4.3
$C_{20}{}^{1=}$ Me ester	0.3[a]	0.8[a]	0.2	1.0	2.6[a]	14.0	1.8[a]
$C_{22}{}^{1=}$ Me ester						62.7	0.2[a]
$C_{24}{}^{1=}$ Me ester						0.6	0.7[a]
Unidentified		0.2	0.5			0.2	

[a] Identity confirmed by column chromatography of monoenoic esters on silver nitrate-silica gel with subsequent GLC of first material eluted.

TABLE IV

GLC of Fragments from Oxidative Cleavage of Monoene Fractions

Oil	Olive	Soybean	Cottonseed	Corn	Peanut	Rapeseed	Safflower	Me oleate
Weight %								
C_6 mc Me ester [a]	0.1	0.3	0.8	1.0	0.6	0.1	1.2	
C_7 mc Me ester	1.5	1.7	3.1	1.0	0.3	1.8	2.1	0.2
C_8 mc Me ester	0.4	0.3	0.5	0.3	0.3	0.4	0.3	0.2
C_9 mc Me ester	47.3	43.5	43.7	44.7	41.4	39.7	42.1	46.7
C_8 dc Me ester	0.2	0.5	0.7	0.2	0.5	0.1	0.3	0.2
C_9 dc Me ester [b]	48.6	49.8	47.9	49.9	50.6	12.0	48.5	51.8
C_{11} dc Me ester [c]	1.6	3.1	2.7	1.4	1.9	8.3	3.4	0.1
C_{12} dc Me ester						0 1		
C_{13} dc Me ester						34.2	0.1	
C_{15} dc Me ester						1.1	0.3	
Unidentified	0.3	0.8	0.6	1.5	4.4	2.2	1.7	0.8

[a] mc = Monocarboxylic acid, dc = dicarboxylic acid.
[b] Methyl palmitate co-elutes with this species under the conditions used for the analyses.
[c] Methyl stearate co-elutes with this species under the conditions used for the analyses.

not as well substantiated as that of the other oils. The tetracosenoic acid was assumed to be the 15-isomer and the remainder of the pentadecanedioic acid fragment produced by oxidative cleavage was calculated as 15-docosenoic acid. There appear to be two other isomers contributing a heptanoic acid cleavage fragment, 11-octadecenoic and 13-eicosenoic acid. The composition given in Table V is consistent with the GLC and oxidative cleavage data on the fraction, but further confirmation of the minor components would be desirable.

The monoene compositions of Table V confirm several previous literature reports and introduce some new components of vegetable oils. The widespread occurrence of eicosenoic acid as well as the presence of higher monoenoic acids in safflower oil do not appear to be common knowledge. This is one of the first reports of 11-octadecenoic acid in common edible oils. [Data presented by Bhatty and Craig at the October 1963 AOCS Meeting, Minneapolis (Paper No. 25), indicated that 11-octadecenoic acid is present in olive oil.] The present study supports the recent data of Tulloch and Craig (20) who found that undecanedioic acid was produced in minor amt upon oxidative cleavage of various (unfractionated) oils. In fact, the data suggest that the major monoenoic acids in vegetable oils always have an isomer associated with them having the double bond two carbons further away from the carboxyl group. Additional evidence in support of this suggestion is available. A commercial sample of azelaic acid, which is usually made by ozonolysis of oleic acid, was found to contain ca. 4% undecanedioic acid. Samples of highly purified methyl 9-hexadecenoate and methyl 11-eicosenoate contain 0.5–1.0% 11-hexadecenoate and 13-eicosenoate, respectively. It should also be mentioned that several lots of oleic and elaidic acid and their esters, some derived from olive oil, have not shown the 11-octadecenoic acid isomer. It is not known in these cases whether the purifications removed the isomer or whether the original oils were free of the isomer.

With the exception of rapeseed oil only linoleic and linolenic acid were found in the polyenoic fractions. Soybean and rapeseed oil contained large amt of linolenic acid as expected. All the other oils gave indications of small amt of linolenic acid. However, since conjugated dienes are frequently co-eluted with methyl linolenate in GLC on polyester columns, the presence of linolenic acid cannot be proven by GLC alone.

The polyene fraction from rapeseed oil contained 2.8% of both an eicosadienoic and a docosadienoic acid. These were identified as the 11,14-isomer and the 13,16-isomer, respectively, from the cleavage fragments obtained from oxidation of the polyenoic fraction. The eicosadienoic acid is not mentioned in common texts but has recently been detected by GLC (13,15).

All the polyenoic fractions showed IR absorption in the isolated *trans* bond region equivalent to 1–2% methyl elaidate. Part of this apparent *trans* acids content is believed due to a difference in background absorption given by *cis* dienes in comparison to the *cis* monoene (methyl oleate) used for calibration. Since highly purified methyl linoleate frequently gives this same apparent *trans* content, there is no reason to interpret the data as indicating the presence of *trans* dienes in common vegetable oils.

The cottonseed oil analyzed in the present work gave a positive Halphen test, indicative of cyclopropenoid fatty acids. The level of these acids (malvalic and sterculic) in freshly extracted, unrefined cottonseed oil has been reported to be in the 1–2% range (17). The acids are heat sensitive (16,18) and are decomposed by boiling acetic acid (18). Considering the refluxing methanol used to prepare methyl esters, the acetic acid liberated during formation of the mercury derivatives and the acidic methanol used to decompose the mercury derivatives after chromatography, it is quite likely that malvalic and sterculic acids would be degraded in the present separation scheme. Methyl malvalate and sterculate were not detected in the gas chromatograms of the unsaturated fractions, nor were any of the diketo-acids produced

TABLE V

Isomer Distribution of Monoenoic Acids

Oil	Olive	Soybean	Cottonseed	Corn	Peanut	Rapeseed	Safflower
Weight % [a]							
C_{16} Δ^9 [b]	1.1	0.4	2.0	0.4	0.2	0.3	0.4
C_{17} Δ^9 [b]	0.2	0.2	0.3	0.1	0.1		
C_{18} Δ^9	95.9	94.3	92.8	96.9	96.2	20.7	92.6
C_{18} Δ^{11}	2.5	4.3	4.7	1.6	0.9	1.5 [c]	4.3
C_{20} Δ^{11} [d]	0.3	0.8	0.2	1.0	2.6	13.0	1.8
C_{20} Δ^{13}						1.0 [c]	
C_{22} Δ^{13}						61.5	0.2
C_{22} Δ^{15}						1.4 [c]	
C_{24} Δ^{15}						0.6	0.7

[a] By combination of GLC and oxidative cleavage on monoene fractions.
[b] Assumed to be Δ^9. In most cases the isomer was present at too low a level for adequate confirmation by oxidative cleavage.
[c] The presence of these isomers at the indicated levels is consistent with the GLC and cleavage data. See discussion.
[d] Assumed to be Δ^{11} in the case of cottonseed oil.

by periodate-permanganate oxidation of the cyclopropene ring (18) seen in the cleavage products. It is concluded that little or no malvalic and sterculic acid are present in the unsaturated fractions isolated from cottonseed oil by mercury derivative chromatography. Furthermore, because of the 10 ppm lower limit of the Halphen test (16), there is some doubt in this author's mind whether refined cottonseed oil, even before methyl ester preparation, etc., contains more than a few tenths per cent of these acids.

The monoenoic acids found in abundance in nature can be considered related to oleic acid in that two-carbon units have been consecutively added to the carboxyl end of the molecule. Rapeseed oil is a good example of this series, since it contains major amt of 9-octadecenoic (oleic), 11-eicosenoic and 13-docosenoic (erucic) acid and a small amt of 15-tetracosenoic acid. *Cis*-9-hexadecenoic (palmitoleic) acid, found in most vegetable and animal fats, is an obvious exception to this rule. The present finding of additional monoenoic acid isomers in vegetable oils indicates a new series, which can be considered related to palmitoleic by the same two-carbon addition rule (i.e., 9-hexadecenoic, 11-octadecenoic, 13-eicosenoic and 15-docosenoic acid). The fatty acid composition of milkweed seed oil lends supports to the existence of this new series, since in this oil a high palmitoleic acid content was accompanied by a high 11-octadecenoic acid level. Other materials rich in palmitoleic acid (such as animal fats and marine oils) would be expected to contain significant amt of the related higher mol wt isomers.

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[Received March 26, 1964—Accepted June 3, 1964]

Water-Resistant, Oil-Based, Intumescing Fire-Retardant Coatings. I. Developmental Formulations[1]

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Abstract

Water-resistant, intumescing fire-retardant paints have been formulated from different types of chemically modified oil-based vehicles, carbonific polyurethanes, spumific melamine phosphates, spumific organohalophosphorus compounds, additives, pigments, driers and solvent. The synthesis of their three major components—the vehicle itself, the spumific melamine phosphates and the carbonific polyurethanes—is described. As a result of evaluation in the standard fire-test cabinet and in the Underwriters' Laboratories' 25-ft tunnel furnace, the formulation whose vehicle has a higher content of tung oil and whose carbonific components are less thermoplastic appear to be most promising. However, these research results have again manifested the importance of the contribution of *each* component in achieving effective fire retardance.

Introduction

SINCE THERE HAS BEEN A significant increase in fire losses, the development and proper application of good fire-retardant coatings have been strongly recommended by coating technologists, fire marshals and industrial and government officials to reduce loss of life and property (1,3,4,13,21,22,26,30,31). In fact, the National Fire Protective Assoc. reported that 11,800 lives and $1,590,000,000 worth of property were lost through fires in the U.S. in 1962 (5).

However, there have been misconceptions about the purposes of fire-retardant coatings. Although they do not prevent fires, they do temporarily suppress the spreading of flames. By permitting fire fighters to arrive at the scene before the building is engulfed by flames, this temporary delay could help save many lives and buildings. Since there are millions of non-fire-retardant buildings throughout the country (1, 13,22,26,31), effective fire-retardant coatings would have unlimited military and industrial potential.

In addition to being fire-retardant, these coatings should have the serviceable, protective and esthetic properties of conventional coatings: can stability, spreadability, drying and bonding characteristics, color and tint retention, serviceability, and water and weather resistance (4,13,15,17,24,25,31). However, none of the fire-retardant coatings presently available possess all of these desired characteristics (2,19,24).

Fire-retardant coatings vary in performance; but, generally speaking, there are two types: intumescing and nonintumescing. The former are more common and appear to be more effective, since their foamy expansion insulates the substrate from the effects of heat and flames and reduces their spread (1,3,4,19,

[1] Presented in part at the 6th Annual Symposium on New Coatings and New Coatings Raw Materials, Fargo, N.D., 1964; and presented at the AOCS Meeting in Chicago, 1964.
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