Chemical Reactions Involved in the Deep-fat Frying of Foods. VII. Identification of Volatile Decomposition Products of Trilinolein[1]

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ABSTRACT

The acidic and nonacidic volatile decomposition products (VDP) produced by pure trilinolein maintained at 185 C with periodic injection of steam were collected, fractionated, and identified. One hundred thirty-three volatile compounds were positively or tentatively identified. The compounds identified included a number of new and interesting compounds, some with valuable organoleptic properties, such as the unsaturated lactones.

INTRODUCTION

The importance of the systematic identification of the volatile decomposition products (VDP) produced by fats and oils during deep-fat frying has been previously reported. A number of workers have reported on the volatile decomposition products of triglycerides, such as tricaprin, 2-oleyl dipalmitin, tricaproin, triolein, and tristearin (1-5). However, the VDP were produced by subjecting the triglycerides to relatively severe conditions, both in terms of temperature and time, with and without the presence of oxygen. Therefore, the VDP produced under these conditions may not be representative of the VDP produced during deep-fat frying conditions.

Previous work in this laboratory has resulted in the identification of the VDP of corn oil and hydrogenated cottonseed oil produced under conditions simulating deep-fat frying (6-9). The present paper reports the systematic identification of the VDP produced by trilinolein under simulated deep-fat frying conditions. Since linoleic acid is one of the more unstable fatty acids found in most oils used for deep-fat frying, studying it as a model system may serve to guide the elucidation of the chemical reactions involved in the deep-fat frying of foods.

EXPERIMENTAL PROCEDURES

Materials

The composition, synthesis and chemical properties of the trilinolein used in this investigation have been previously reported (10).

Simulated Deep-fat Frying and Isolation of VDP

The apparatus and procedure used for the generation and simultaneous collection of the VDP produced by the synthetic trilinolein under simulated deep-fat frying conditions has previously been reported (10). The apparatus used in the present investigation is a modification of the apparatus used by Krishnamurthy et al. (11). In this modified apparatus, steam was periodically injected into the heated trilinolein (185 C) in order to simulate deep-fat frying. At the end of each 7-hr. day of frying, the cold traps containing the VDP were washed with distilled water and reagent grade ethyl ether. The washings were stored in amber glass bottles at -10 C. The trilinolein, after treatment under simulated frying conditions for 74 hr., was freed from VDP by vacuum steam distillation at 150 C. and 0.01 Torr for 1 hr. The VDP collected from the vacuum distillation were combined with the VDP obtained during the simulated frying treatment.

The combined VDP were extracted with reagent grade ethyl ether, and the extract was separated into acidic and nonacidic fractions by extraction with 10% aqueous sodium carbonate solution. The ethyl ether solutions of acidic and nonacidic VDP, respectively, were then concentrated according to the method of Chang (12). Since both of the concentrated ethyl ether solutions were contaminated with some entrained trilinolein, they were molecularly distilled at 150 C and 1×10^{-3} Torr for 6 hr (11). The acidic VDP were converted into their methyl esters by the use of diazomethane (13).

Fractionation of Acidic VDP by Gas Chromatography

The methyl esters of the acidic VDP were separated into 21 fractions by gas chromatography with an Aerograph A-90P using a 10 ft. x 3/8 in. I.D. aluminum column packed with 15% DEGS on Chromosorb W, AW 70/80 mesh (Figure 1). The temperature was nonlinearly programmed from 60-190 C and held at 190 C for 18 min. The He flow rate was 80 ml/min. Each of the 21 fractions was accumulatively collected in one trap according to the method of Deck, et al. (14).

Each of the 21 preliminary fractions of the acidic VDP was rechormatographed with an Aerograph 1520 using a 20 ft. x 1/8 in. I.D. stainless steel column packed with 12% SE-30 on Chromosorb W 50/60 mesh. The He flow rate was 46 ml/min. The conditions used for the rechromatography of each preliminary fraction were chosen for maximum resolution. The subfractions obtained in the second and

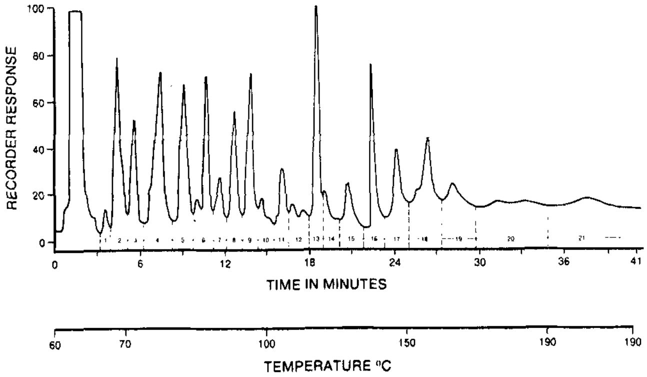

FIG. 1. Gas chromatogram of the acidic volatile decomposition products from thermally oxidized trilinolein.

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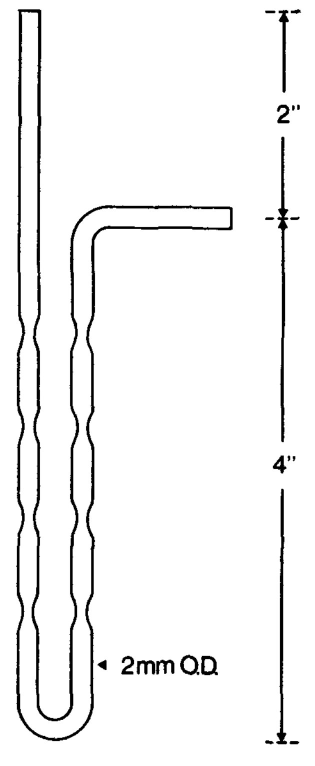


FIG. 2. "Hairpin" trap used for the collection of volatile samples from a gas chromatograph.

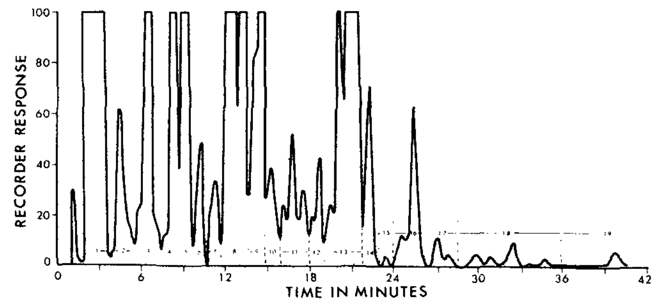


FIG. 3. Gas Chromatogram of nonacidic volatile decomposition products produced by thermally oxidized trilinolein.

subsequent chromatographies were accumulatively collected in "hairpin" traps.

The "hairpin" trap consists of a 30 cm. long piece of 1-3 mm Pyrex glass tubing. This piece of tubing is bent into the shape shown in Figure 2. Restrictions are drawn every 2 cm. in the collecting portion of the tubing. A GC fraction is collected by inserting the side arm of the trap, which is cooled in an appropriate low temperature bath, into the exit septum of the gas chromatograph.

Each subfraction resulting from the second chromatography was then chromatographed for a third time with the Aerograph 1520 using a 20 ft. x 1/8 in. I.D. stainless steel column packed with 15% DEGS on Chromosorb W 70/80 mesh.

Fractionation of Nonacidic VDP by Gas Chromatography

The nonacidic VDP were separated into 19 fractions by gas chromatography using an Aerograph A-90P with a 10 ft. x 3/8 in. I.D. aluminum column packed with 15% SE-30 on Anakrom ABS 70/80 mesh (Figure 3). The temperature was nonlinearly programmed from 80-200 C and held at 200 C for 27 min. The He flow rate was 80 ml/min. Each of the 19 fractions was accumulatively collected in one trap according to the method of Deck et al. (14).

Each of the 19 preliminary fractions of the nonacidic VDP was rechromatographed with an Aerograph 1520 using a 10 ft. x ¼ in. I.D. aluminum column packed with 15% Carbowax 20M on Anakrom ABS 70/80 mesh. The He flow rate was 66 ml/min. The conditions used for the rechromatography of each preliminary fraction were chosen for maximum resolution. These subfractions and subsequent ones were accumulatively collected in "hairpin" traps.

Each subfraction resulting from the second chromatography was then chromatographed for a third time with the Aerograph 1520 using a 20 ft. x 1/8 in. I.D. stainless steel column packed with 15% SE-30 on Anakrom ABS 70/80 mesh.

Identification of Gas Chromatographic Fractions

The subfractions obtained from the third chromatographies of the acidic and nonacidic VDP of trilinolein were in most instances pure compounds as indicated by the peak shapes from the third chromatography and by the subsequent IR and mass spectra. These subfractions were identified by a combination of IR and mass spectrometry and GC retention time according to the method of Kawada et al. (16). A compound was considered to be positively identified if both the IR and mass spectra agreed with the published spectra. An identification was considered tentative if only one type of spectral data was available.

Peak Size

The peak area of each subfraction was obtained according to the method of Krishnamurthy and Chang (7).

RESULTS AND DISCUSSION

A total of 133 compounds was either positively or tentatively identified from the acidic and nonacidic VDP produced by trilinolein during simulated deep-fat frying (Tables I and II). The identified compounds consisted of 8 saturated acids, 24 unsaturated acids, 4 hydroxy acids, 2 keto acids, 3 dibasic acids, 5 aromatic compounds, 15 hydrocarbons, 11 alcohols, 1 cyclic ether, 5 esters, 9 saturated aldehydes, 15 unsaturated aldehydes, 13 ketones, 8 saturated lactones, 7 unsaturated lactones, and 3 miscellaneous compounds.

A number of compounds not previously identified in the VDP of fats and oils were found in this study. The infrared and mass spectra of some of these compounds are presented in Figures 4-7.

Figure 4 shows the infrared spectrum of the subfraction identified as 6-hydroxyhexanoic acid, lactone. The broad, strong absorption band at 5.8μ is due to the ester carbonyl group within the 7-membered ring (17). The series of strong absorption bands between 7.75 and 9.50μ is due to the C-O-C group of the ester linkage. The IR of this fraction is in excellent agreement with the published IR of 6-hydroxyhexanoic acid lactone (18).

Figure 5 shows the IR and mass spectra of the subfraction identified as 4-hydroxy-2-octenoic acid, lactone. The strong carbonyl absorption at 5.70μ and the weak double bond absorption at 6.20μ are characteristic of γ-lactones unsaturated in the 2, 3 position (19). Strong absorption bands due to lactone C-O-C stretching are apparent between 8.0 and 10.0μ. The mass spectrum shows a moderate intensity molecular ion at m/e 140 and a base peak at m/e 83 (M-57). The latter peak is probably due to the loss of the alkyl side chain with charge retention on the unsaturated lactone ring, analogous to the case of saturated γ-lactones (20). Other major peaks at m/e 55, 41, and 29 are ions probably derived from lactone ring fragmentation.

TABLE I

Compounds Positively Identified in the Volatile Decomposition Products of Thermally Oxidized Trilinolein

Fraction[a]	Identified as	Peak size[b]
Saturated acids		
A-3-3-1	Propanoic acid	M
A-4-4-1	Butanoic acid	S
A-5-7-1	Pentanoic acid	M
A-6-7-1	Hexanoic acid	XL
A-7-2-1	Heptanoic acid	L
A-9-7-1	Octanoic acid	M
A-20-4-1	Nonanoic acid	M
A-14-7-2	Decanoic acid	M
Unsaturated acids		
A-17-3-1	*trans*-2-Butenoic acid	S
A-7-2-3	*trans*-2-Pentenoic acid	L
A-7-3-3	*trans*-3-Pentenoic acid	M
A-10-9-1	*trans*-2-Heptenoic acid	XL
A-11-8-1	*trans*-2-Octenoic acid	S
A-12-2-1	*trans*-2-Nonenoic acid	XL
A-13-10-1	*trans*-3-Nonenoic acid	L
A-14-11-1	*trans*-2-Decenoic acid	XL
A-10-10-2	*cis*-2-Heptenoic acid	S
A-10-10-3	*cis*-3-Heptenoic acid	S
A-11-9-1	*cis*-2-Octenoic acid	S
A-11-9-2	*cis*-3-Octenoic acid	S
A-13-12-2	*cis*-2-Nonenoic acid	L
A-12-3-1	*cis*-3-Nonenoic acid	S
A-15-7-1	*cis*-2-Decenoic acid	S
A-16-5-1	*cis*-3-Decenoic acid	S
A-7-3-2	5-Hexenoic acid	S
A-9-6-1	6-Heptenoic acid	L
A-10-10-4	7-Octenoic acid	S
A-16-6-4	*cis*-2-*trans*-4-Octadienoic acid	M
A-17-10-3	*trans*-2-*cis*-4-Decadienoic acid	M
A-18-8-1	*trans*-2-*trans*-4-Decadienoic acid	M
Hydroxy acids		
A-16-3-2	β-Hydroxyhexanoic acid	S
A-14-6-1	α-Hydroxyheptanoic acid	M
A-16-4-1	α-Hydroxyoctanoic acid	S
Dibasic acids		
A-21-10-1	Pimelic acid	XS
A-20-7-1	Suberic acid	S
A-19-7-2	Adipic acid	S
Saturated hydrocarbons		
N-6-6-4	Nonane	S
N-9-2-2	Decane	M
N-13-7-3	Tetradecane	S
N-17-2-1	Pentadecane	XS
N-17-2-2	Dexadecane	XS
Unsaturated hydrocarbons		
N-5-1-2	1-Octene	S
N-5-1-3	*trans*-2-Octene	S
N-5-1-4	*cis*-2-Octene	S
N-5-2-3	1-Nonene	S
N-10-2-3	a *trans*-Undecene	S
N-10-1-3	a *trans*-Dodecene	S
Alcohols		
N-4-3-1-1	Ethanol	M
N-3-5-1	1-Butanol	L
N-4-5-1	1-Pentanol	XL
N-6-9-1	1-Hexanol	S
N-7-8-2	1-Heptanol	S
N-9-6-1	1-Octanol	L
N-8-8-4	2-Octanol	M
N-8-6-4	3-Octanol	S
N-16-12-2	1-Undecanol	M
N-18-3-1	1-Dodecanol	S
N-8-6-2	1-Octen-3-ol	XL
Cyclic ether		
A-19-1-4	1,4-Dioxane	L
Esters		
N-2-2-1	Ethyl acetate	XL
N-3-6-1	Butyl acetate	S
N-7-6-2	Hexyl formate	XS
N-19-4-6	Methyl nonanoate	S
N-19-6-1	Methyl dodecanoate	XS
Saturated aldehydes		
N-2-3-1	Butanal	S
N-3-3-1	Pentanal	XL
N-5-6-1	Hexanal	XL
N-5-7-7	Heptanal	XL
N-9-8-1	Octanal	M
N-10-5-1	Nonanal	S
N-12-15-1	Decanal	M
N-12-13-2-2	Dodecanal	XS
Unsaturated aldehydes		
N-6-7-2	*trans*-2-Hexenal	M
N-8-5-1	*trans*-2-Heptenal	XL
N-10-6-1	*cis*-2-Heptenal	S
N-12-2-1	*trans*-2-Octenal	XL
N-11-13-1	*cis*-2-Octenal	S
N-12-9-3	*trans*-2-Nonenal	M
N-12-6-2	*cis*-2-Nonenal	XS
N-14-2-3	*trans*-2-Decenal	M
N-8-7-1	*trans*-2-*cis*-4-Heptadienal	M
N-14-1-6	*trans*-2-*cis*-4-Nonadienal	M
N-13-10-1	*trans*-2-*trans*-4-Nonadienal	XL
N-16-6-5	*trans*-2-*cis*-4-Decadienal	XS
N-13-11-1	*trans*-2-*trans*-4-Decadienal	XL
Ketones		
N-8-3-4	2-Heptanone	L
N-8-11-1	3-Heptanone	S
N-8-9-3	2-Octanone	M
N-13-15-3	3-Octanone	S
N-9-4-4	2-Nonanone	S
N-16-4-1	3-Nonanone	S
N-15-1-8	3-Decanone	M
N-12-3-3	2-Undecanone	M
N-13-17-6	2-Dodecanone	XS

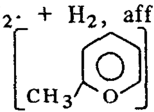
TABLE 1 (Continued)

Fraction[a]	Identified as	Peak size[b]
Saturated lactones		
N-8-9-1	4-Hydroxypentanoic acid lactone	M
N-9-12-1	4-Hydroxyhexanoic acid lactone	S
N-12-12-2	4-Hydroxyoctanoic acid lactone	M
N-16-8-5	4-Hydroxynonanoic acid lactone	S
N-16-10-9	4-Hydroxydecanoic acid lactone	S
N-19-5-1	5-Hydroxydecanoic acid lactone	S
N-11-12-1	6-Hydroxyhexanoic acid lactone	S
Unsaturated lactones		
N-8-11-2	4-Hydroxy-2-hexenoic acid lactone	M
N-11-9-2	4-Hydroxy-2-heptenoic acid lactone	XS
N-12-7-2	4-Hydroxy-2-octenoic acid lactone	XS
N-13-4-4	4-Hydroxy-2-nonenoic acid lactone	XL
N-16-8-6	4-Hydroxy-2-decenoic acid lactone	S
Aromatic compounds		
N-8-8-2	Benzaldehyde	M
N-11-3-8	Hexylbenzene	S
N-17-5	4-Phenylbutanal	XS
N-17-8-7	5-Phenylpentanal	S
N-9-4-4	Butylbenzene	S
Miscellaneous compound		
N-8-3-6	2-Pentylfuran	XL

[a] A = Acidic VDP and N = Nonacidic VDP. The first, second and third numerals indicate the position of the GC fraction during the first, second and third chromatographies, respectively.

[b] XS = Extra small; S = Small; M = Medium; L = Large; XL = Extra Large.

The IR and mass spectra of the subfraction identified as 1,4-dioxane are shown in Figure 6. The strong absorption band at 9.00μ is characteristic of the C-O-C linkage (17). The IR of this fraction is in excellent agreement with the published IR of 1,4-dioxane (18). The mass spectrum shows a strong molecular ion peak at m/e 88 and the base peak at m/e 58 (M-30). The latter peak is due to expulsion of CH_2O, probably as a formaldehyde molecule, analogous to the situation with tetrahydropyran (20). Other major peaks occur at m/e 57, 43, 31, 29, and 28. The mass spectrum of this fraction is in good agreement with the published mass spectrum of 1,4-dioxane (21).

Figure 7 shows the IR and mass spectra of the methyl ester of the subfraction identified as *cis*-2-Octenoic acid. The conjugated *cis* double bond is indicated by the absorption bands at 3.30 and 6.10μ, and the lack of a strong absorption band around 10.3μ, indicative of *trans* C-H bending (17). The strong bands at 5.81 and 8.55μ are due to the conjugated carbonyl and C-O-C groupings of the ester (17). The mass spectrum shows a moderately intense molecular ion at m/e 156. The ion at m/e 125 (M-31) is probably due to loss of the $\cdot OCH_3$ group (20). The base peak at m/e 111 (M-45) may be due, at least in part, to the loss of $CH_3CH_2\cdot + H_2$, affording the stable, aromatic pyrylium ion  (20). The ions at m/e 87 and 74 are indicative of esters unsaturated in the 2,3-position as well as saturated esters (20).

The large number of volatile compounds identified from thermally oxidized trilinolein indicates that extensive thermal oxidative decomposition of the triglycerides can take place during deep-fat frying of highly unsaturated oils. This decomposition takes place through initial formation of hydroperoxides as a result of attack by oxygen directly on the trilinolein or on free linoleic acid, as proposed by Farmer (22). Subsequent decomposition of the hydroperoxides by means of a free radical pathway produces aldehydes, alcohols, ketones, etc. Secondary oxidation of these compounds may then take place leading to the production of carboxylic acids and related compounds as discussed by Horner (23).

In the case of aldehydes, secondary oxidation can take place either at the carbonyl group or in the vicinity of any double bonds present. The large amount of hexanoic acid arose as a result of secondary oxidations, especially the oxidation of hexanal. Two of the terminal, unsaturated fatty acids found in the present study, 6-heptenoic and 7-octenoic acids, were also found in the VDP of hydrogenated cottonseed oil (9). The large amount of 1,4-

TABLE II

Compounds Tentatively Identified in the Volatile Decomposition Products of Thermally Oxidized Trilinolein

Fraction[a]	Identified as	Peak size[b]
Unsaturated acids		
A-13-12-3	*cis*-4-Nonenoic acid	S
A-17-9-3	*cis*-3-Undecenoic acid	S
Hydroxy acid		
A-18-6-3	β-Hydroxyoctanoic acid	S
Keto acids		
A-18-6-3	γ-Ketoheptanoic acid	S
A-21-11-1	4-Ketopimelic acid	XS
Unsaturated hydrocarbons		
A-3-4-2	*trans*-1,3-Octadiene	S
A-4-4-2	*trans*-1,3-Nonadiene	XS
N-16-1-1	a *trans, trans*-Tetradecadiene	S
N-16-1-2	a *trans, cis*-Tetradecadiene	S
Aldehydes		
N-12-10-1	3,4,5-Trimethylheptanal	M
N-8-3-3	*cis*-3-Hexenal	M
N-8-4-1	*trans*-4-Hexenal	S
Ketones		
N-11-4-1	*trans*-3-Nonen-2-one	S
N-11-4-2	2-Methyl-3-octen-5-one	S
N-17-5-6	*trans*-3-Undecen-2-one	S
N-19-3-2	3-Dodecanone	XS
Lactones		
N-10-12-2	5-Hydroxyhexanoic acid lactone	S
N-16-5-2	4-Hydroxy-3-nonenoic acid lactone	XL
N-16-13-2	5-Hydroxy-2-nonenoic acid lactone	M
Miscellaneous compounds		
N-8-8-1	4-Methoxy-3,3-dimethylbutanal	S
N-9-9-1	1-Methoxy-3-hexanone	L

[a] A = Acidic VDP and N = Nonacidic VDP. The first, second and third numerals indicate the position of the GC fraction during the first, second and third chromatographies, respectively.

[b] XS = Extra small; S = Small; M = Medium; L = Large; XL = Extra Large.

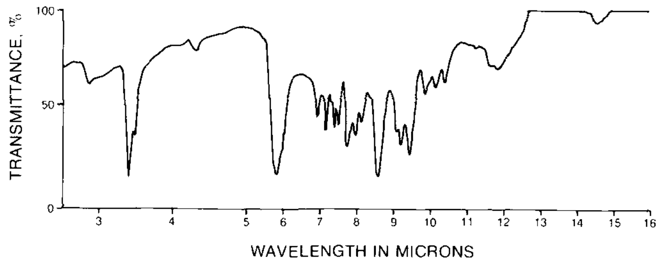


FIG. 4. IR spectrum of subfraction identified as 6-hydroxyhexanoic acid lactone.

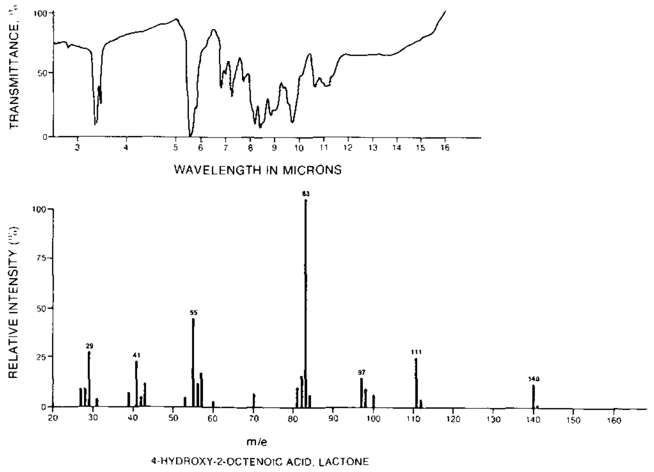


FIG. 5. IR and mass spectra of subfraction identified as 4-hydroxy-2-octenoic acid lactone.

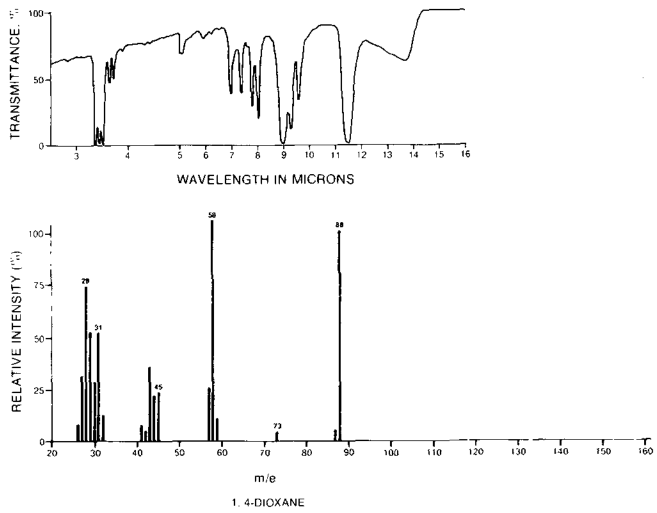


FIG. 6. IR and mass spectra of subfraction identified as 1,4-Dioxane.

dioxane found in the acidic VDP from trilinolein indicates the importance of studying the VDP of fats and oils from the standpoint of potential toxicological effects from eating deep-fat fried foods or from inhaling the smoke generated during frying. 1,4-Dioxane has been shown to have toxic effects on animals and on humans, both by ingestion and inhalation (24, 25).

In the autoxidation of the 1,4-diene system of linoleate, Swoboda and Lea (26) accounted for the formation of the major volatile components from decomposition of the 9 and 13-hydroperoxides formed at the α-positions of the conjugated system. The conjugated system is formed by the shift of either double bond in the nonconjugated linoleate system. The large amounts of hexanal and the 2,4-decadienals arise, at least in part, by decomposition of the appropriate conjugated hydroperoxides of linoleate. Five minor hydroperoxides may also arise from linoleate, according to Swoboda and Lea (26), to eventually form other VDP. The compounds found in the present study which may have resulted from decomposition of these minor hydroperoxides are: *trans*-2-octene, the 2-heptenals, pentanal, and the 2-octenals. No VDP corresponding to the decomposition of the hydroperoxides at position 8 of the linoleate was found. Although the 3-nonenals, expected from the decomposition of the 10-hydroperoxide, were not found, the *cis* and *trans*-3-nonenoic acids were found in the VDP.

Chang et al. (27) proposed that 2-pentyl furan was formed in reverted soybean oil through the oxidative decomposition of linoleate. The large amount of this compound found in the present work supports this proposal. The aromatic compounds identified supports the hypothesis of Krishnamurthy and Chang (7) that aromatic ring formation from long chain unsaturated fatty acids is feasible during deep-fat frying.

A number of the compounds identified in the present study could not have arisen as a result of the decomposition of the hydroperoxides of linoleate. Either these compounds were formed as a result of secondary reactions of the initially formed VDP, or from the thermal oxidation of trilinolein in which the double bond system has migrated along the carbon chain, forming various isomers of linoleate.

Although in the case of deep-fat frying, the latter situation is probably remote, Emken et al. (28) and Scholfield et al. (29) have demonstrated double bond migration in unsaturated fatty acid esters as a result of certain heat and processing treatments.

A large number of lactones were found, including γ- and δ-isomers and, for the first time, an ε-isomer, 6-hydroxyhexanoic acid lactone. The γ-lactones, both saturated and unsaturated, were found in the largest amounts (Tables I and II).

The identification of the unsaturated lactones was based upon the IR, mass spectra and retention times of the authentic compounds published by May, et al. (30). They reported that they contributed to the deep-fat fried flavor, particularly, the C_9 γ-unsaturated lactones.

ACKNOWLEDGMENT

Public Health Service Research Grant, HE-07610, from the National Heart Institute. Pure trilinolein contributed by Procter & Gamble Company.

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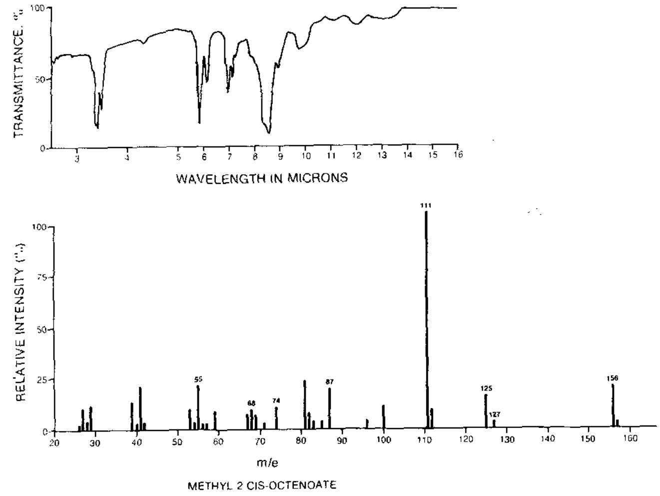

FIG. 7. IR and mass spectra of subfraction identified as the methyl ester of *cis*-2-octenoic acid.

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[Received June 2, 1977]