TABLE I
Comparison of Volatile Compounds from Autoxidized Methyl Linoleate at PV 100–150 and PV 1000[a] mmol/kg

Compounds[b]	PV 100–150	PV 1,000
carbon dioxide	+	+
methyl formate	+	+
pentane	+	+
acetone	+	0[c]
butanal	+	+
2-methyltetrahydrofuran	+	—
2-pentanone	+	+
pentanal	+	+
hexanal	+	+
amyl formate	+	+
2-heptanone	+	+
methyl hexanoate	+	+
1,1-dimethoxyhexane	—	+
2-heptenal	+	+
methyl heptanoate	+	+
2-octenal	+	+
methyl octanoate	+	+
2-ethyl-4-pentyldioxolane	—	+
geometric isomer of 2-ethyl-4-pentyldioxolane	—	+
pentyl hexanoate	+	+
1-methoxy-1-pentoxyhexane	—	+
2-propyl-4-pentyldioxolane	—	+
geometric isomer of 2-propyl-4-pentyldioxolane	—	+
2-butyl-4-pentyldioxolane	—	+
geometric isomer of 2-butyl-4-pentyldioxolane	—	+
cis-2,4-dipentyldioxolane	—	+
trans-2,4-dipentyldioxolane	—	+

[a] Compounds previously identified from autoxidized methyl linoleate, PV 1,000 (1).
[b] Compounds identified in low and high level oxidations are listed in order of increasing GLC retention times.
[c] Not identified, but could be present in very low levels and not be identified by the GC-MS technique used. (GC retention times of acetone and pentane are similar.)

distilled water. The glass wool used was purified by extensive washings with carbon tetrachloride followed by distilled water and then dried in a vacuum desiccator. Gas-liquid chromatographic analysis of headspace gas samples (5–15 ml) from the purified glass wool using a gas chromatograph equipped with a flame ionization detector showed the absence of volatile compounds. Also, blanks were run consisting of purified glass wool exposed to oxygen for seven to nine days and then vacuum distilled at ambient temperature at 20–60 μ. Only a trace of water (estimated volume < 20 μl) was obtained.

Table I contains a comparison of the volatile compounds identified from autoxidized methyl linoleate at peroxide levels of 100–150 and those obtained previously at PV of 1,000 mmole/kg. Each listed compound was identified by comparison of its gas chromatographic and mass spectral data with those of an authentic compound.

2-Methyltetrahydrofuran was found in the less oxidized system but not in the more highly oxidized system. However, this compound could be either absent or present in a low level in the more highly oxidized system since it might not separate from the larger amount of 2-pentanone, as the GLC retention times of the two are close. Identification of 2-methyltetrahydrofuran by means of our MS-GC technique in an abundance of 2-pentanone would be impossible due to the similarities of the mass spectral features of these compounds.

The mechanism of formation of 2-methyltetrahydrofuran is not known. Tetrahydrofurans have been reported to be formed by oxidation of hydrocarbons (2), (3)–specifically 2-methyltetrahydrofuran from pentane (4) but at temperatures above 300 C.

1,1-Dimethoxyhexane, 1-methoxy-1-pentoxyhexane and 2,4-dialkyldioxolanes were shown to be absent in the more mildly oxidized system (Table I) by the MS-GS technique employed. This finding of the absence of these compounds is consistent with our hypothesis that certain secondary reactions consuming aldehydes are responsible for 2,4-dialkyldioxolanes and acetals.

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Nature of Residual Lipids in Menhaden Fish Protein Concentrate

RECENTLY WE DESCRIBED the residual lipids of a fish protein concentrate (FPC) prepared from red hake (*Urophycis chuss*) (1). Hake is a relatively low-fat fish. However, most of the fish that could be utilized for FPC are high in fat; hence we have now analyzed the residual lipids in an FPC prepared from menhaden, a fatty fish. Menhaden (*Brevoortia tyrannus*) has been used for many years in the manufacture of fish meal. FPC was made from fresh menhaden fish by extraction with 2-propanol in the laboratory glassware unit (2) (Bureau of Commercial Fisheries at College Park, Maryland) and furnished to us as batch M-3. The residual lipids were extracted and analyzed by the methods previously used except that non-lipid contaminants were removed by Sephadex chromatography (3).

The menhaden FPC, M-3, was found to have approximately the same amount of residual lipid (chloroform-soluble material) as did the hake FPC, batch GO-1 (Table I). Thin-layer chromatography (TLC) showed that the menhaden lipid was almost

TABLE I
Lipid Content of FPC

Sample	Weight g	Number of days extracted	Yield[a] g	Yield[a] %
GO-1 (hake)	250	14	0.28	0.11
M-3 (menhaden)	250	15	0.38	0.15

[a] The lipids were extracted in a Soxhlet apparatus (chloroform-methanol, 2:1) (2), and the chloroform-soluble material was purified by Sephadex chromatography (3). Aliquots were evaporated to dryness and weighed on a Cahn electrobalance, Model G.

TABLE II
Comparison of Fatty Acid Composition of FPC Residual Lipids With Those of Fresh Fish Oils

Fatty acid chain length	No. of double bonds	Hake FPC[a] %	Fresh hake[b] %	Menhaden FPC[c] %	Fresh menhaden[d] %
12	0	0.9		tr	tr
14	0	3.8	2.2	9.2	8.0
	1	tr			
15	0	0.8	0.2	0.8	0.5
16	0	25.2	17.8	28.2	28.9
	1	8.8	8.5	10.9	7.9
	2		1.0		0.8
17	0	1.2	0.6	0.6	1.0
18	0	9.3	3.9	4.9	4.0
	1	22.2	26.7	13.7	13.4
	2	2.5	1.2	2.6	1.1
	3		0.5	2.4	0.9
	4		2.8		1.9
19	0	1.0	0.7	0.2	0.9
20	0	1.6	0.2		
	1	2.5	0.9		0.9
	2	0.8	0.4	3.6	0.5
	3	1.1	0.1		
	4	1.0	1.2	0.8	1.2
	5	4.5	15.5	8.9	10.2
22	0	3.2			
	1		0.2	0.6	1.7
	2	1.1		tr	
	3	1.8	0.7		
	4	1.6	0.3	tr	0.7
	5	0.7	1.5	1.1	1.6
	6	2.5	10.5	12.3	12.8

[a] Sample GO-1 B (2).
[b] Analysis of fresh red hake fatty acids by Bureau of Commercial Fisheries, College Park, Maryland.
[c] Sample M-3.
[d] Gruger et al. (4).

entirely triglyceride, with small amounts of free fatty acids, mono- and diglycerides and phospholipid (PL). Phosphorus analysis indicated that total PL amounted to about 4% of the total lipid. Hake product lipids had been found to contain from 20% to 35% PL. This finding is the main qualitative difference between the residual lipids of the two types of FPC.

The M-3 fatty acid methyl esters had approximately the same composition as fresh menhaden oil (Table II) whereas those from the GO-1 sample were found to differ somewhat from those of fresh hake. The lower content of highly unsaturated fatty acids in the hake product suggests that the difference may reflect some oxidative losses during handling.

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