due to a fog or mist issuing from commercial traps, may be caused by the high flow rate of carrier gas blowing out small particles of solidified esters. Commercial GC-2 traps were modified, and several other designs were tried, but they did not prove entirely successful. The author designed and constructed a new trap that overcame previous difficulties. This trap, shown in Figure 1, has two exit tubes with roughened internal surfaces. This system permitted up to 87% of the injected sample to be recovered as a pure material.

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[Received May 24, 1962—Accepted March 10, 1963]

Tuna Fatty Acids: II. Investigations of the Composition of Raw and Processed Domestic Tuna

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Abstract

Oils from raw, precooked, and canned tuna were analyzed by gas-liquid chromatography. The analyses showed the presence in tuna oil of 20 fatty acids having chain lengths ranging from 14 to 22 carbon atoms and containing 0 to 6 double bonds. The polyunsaturated fatty acids underwent little change during processing from the raw to the canned product.

Introduction

YELLOWFIN *(Neothunnus macropterus)*, skipjack *(Katsuwonus pelamis)*, albacore *(Thunnus germo)*, and bluefin *(Thunnus thynnus)* are the principle species used in the domestic tuna industry. The production of canned tuna has increased at such a rate that tuna is now the most important canned seafood of the U. S. (1). Most tuna is packed in vegetable oil, but some is packed in an oil-free water broth.

Tuna by-products—which include dark meat, scraps, and precook oils—are also used extensively. The dark meat is used in the preparation of pet foods; the scraps and other refuse, in the production of meals and fish solubles for poultry feed; and the oil, in the manufacture of such products as paint.

Tuna light meat is the portion of the fish that is processed as food for humans. Although this high-protein food is canned in large quantity, relatively little is known about the fatty acid composition of the flesh oil. Even less is known about the structures of the isomers of the constituent unsaturated fatty acids. [In a recent investigation of bluefin fatty acids, the author isolated and determined the structural isomers of the monoenoic fatty acids from this fish. (In press.)] Igarashi and co-workers (2–4) have reported the composition of many of the phospholipids of "Kuromaguro" [oriental or black tuna *(Thunnus orientalis)*], and Katada (5,6) has recently published analyses of the sphingolipids of black tuna. (The true identity of black tuna is still undecided. Some ichthyologists believe it to be similar to *Thunnus thynnus:* others believe it to be a separate species.)

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Knowledge of the fatty acid composition is desirable for an important fish such as tuna, not only because of the value of the fish as a foodstuff, but also because of possible new industrial applications of the oil. In light of recent dietary and medical emphasis on the role of fatty acids in human physiology (7,8), it is important to know the fatty acid composition and the structures of the isomers of the fatty acids of this group of fish. Since the treatment that the oil in the flesh undergoes during processing may influence flavor and quality of the oil, it is also desirable to determine whether processing destroys unsaturation or otherwise alters the oil.

The objectives of the present study therefore were: 1.) To determine what fatty acids are present and their relative amounts in the oils from the light and dark flesh of albacore, bluefin, yellowfin, and skipjack tuna. 2.) To study the effects of processing on the oils in regard to (a) destruction of unsaturation and (b) differential extraction of fatty acids. 3.) To determine to what extent tuna oils contribute to the fatty acids in (a) the drained oil and (b) the residual oil in a vegetable oil pack of tuna.

Experimental Work

The study was made with samples of two kinds: laboratory and commercial (Fig. 1).

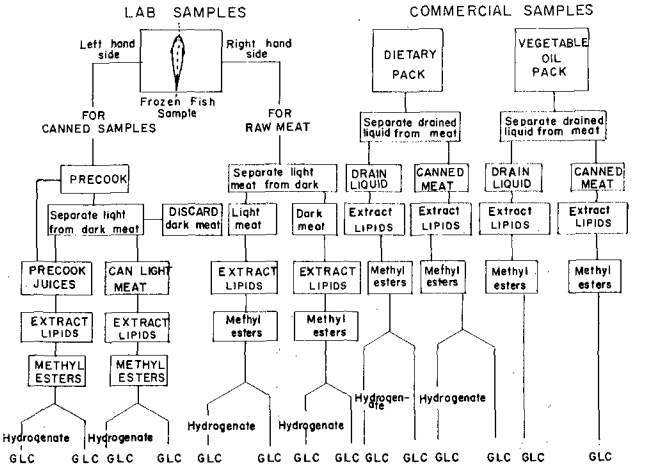

FIG. 1. Composition studies of raw and processed domestic tuna.

TABLE I
Comparison of Chain Lengths Calculated from Analyses of Nonhydrogenated and Hydrogenated Skipjack Precook Oil Methyl Esters

Fatty acid–carbon atoms: double bonds	Relative amount of fatty acids in oil	
	Original fatty acids	Fatty acids after hydrogenation
	Area %	*Area %*
14:0	7.7	7.0
15:0	1.1	0.9
16:0	23.4	
16:1	7.2	
16:2	0.5	
16–total	31.1	32.6
17:0	1.0	1.2
18:0	3.0	
18:1	14.6	
18:2	1.9	
18:4	tr	
18:3–20:?	1.8	
18–total	19.3	20.5
19:0	tr	tr
20:1	2.3	
20:4	3.5	
20:5	13.2	
20–total	19.0	18.5
21:0	tr	tr
22:1	tr	
22:3	0.6	
22:4	0.7	
22:5	0.8	
22:6	17.6	
22–total	19.7	19.6

Preparation of Laboratory Samples

Laboratory samples were prepared from whole raw fish caught off the western coast of South America and kept frozen until used. For each species, two fish were sampled. The species sampled and the total weights of the samples taken were as follows: yellowfin, 27 lb; skipjack, 12 lb; bluefin, 60 lb; and albacore, 23 lb.

In the sampling process, the frozen fish were sawed in half (sagittal section). The right hand sections were combined as a source of raw meat samples; the left hand sections, as a source for preparing canned meat samples.

Raw meat samples. Light meat was separated from dark meat, and oil was extracted quantitatively from the light- and dark-meat samples by the method of Bligh and Dyer (9). Methyl esters for gas-liquid chromatography (GLC) were prepared directly from the oil by the alcoholysis procedure of Gauglitz and Lehman (10).

GLC analyses were conducted with a RSCO 600-Series apparatus equipped with a beta ionization detector utilizing a Sr^{90} source. A 10′ × ¼″ OD column containing 20 g of 2.7% DEGS on acid and base washed and siliconized (11) Chromosorb W (80–100 mesh) was employed. The column was maintained at 174C, with argon flowing at the rate of 30 ml/min, and the volume of the sample was 0.13–0.2 μl.

Peaks appearing on the chromatogram were identified by (a) comparing logarithmic plots of relative retention time (methyl stearate = 1) against chain lengths or degree of unsaturation with plots prepared from known standards and (b) comparing hydrogenated with nonhydrogenated samples. The area percent for each component was determined by triangulation.

Dark-meat samples were handled in the same way as the light-meat samples.

Canned-meat samples. The left-hand sections were precooked, a species at a time, by placing them in large trays and exposing the meat for 1 hr to wet steam at 3–5 psi in an upright retort. The precooked juices were collected, and the remaining meat was treated separately.

The oils were extracted from the aqueous mixture of the precook juices with petroleum ether. After the extract had been dried over anhydrous sodium sulfate, the solvent was removed at reduced pressure, the residual oils were converted to methyl esters and the methyl esters were divided into two groups, one of which was hydrogenated. The methyl esters in both groups then were subjected to the GLC analysis described earlier.

The precooked tuna meat was allowed to stand overnight at 2C. The dark meat was discarded, and the light meat was packed into ¼ lb tuna cans. After 10 ml of water had been added to each can, the cans were vacuum sealed at 600 mm Hg, and the canned product was pressure cooked at 116C for 75 min. After the canned product had aged for 3 weeks at room temperature, in order to allow possible equilibration to take place between lipids of the meat and free juices, eight cans were opened, and the excess liquid was allowed to drain from the product.

The oil content in the drained liquid was small, so the oil was discarded.

The meat was solvent extracted, the recovered oils

TABLE II
GLC Analyses of the Oil Solvent-Extracted from Raw, Laboratory-Canned and Commercially-Canned (Vegetable Oil Absent) Tuna Light Meats

Fatty acid carbon atoms: double bond	Fatty Acid Content											
	Albacore			Bluefin			Yellowfin			Skipjack		
	Raw	Lab canned	Commercial (veg. oil absent)	Raw	Lab canned	Commercial (veg. oil absent)	Raw	Lab canned	Commercial (veg. oil absent)	Raw	Lab canned	Commercial (veg. oil absent)
	A.%	*A.%*	*A.%*	*A.%*	*A.%*	*A.%*	*A.%*	*A.%*	*A.%*	*A.%*	*A.%*	*A.%*
14:0	3.7	4.3	3.5	4.5	5.7	5.7	2.6	2.4	2.6	7.0	7.1	6.0
15:0	1.0	1.2	1.0	0.6	0.8	0.5	0.6	0.7	0.8	1.0	1.1	0.6
16:0	29.3	27.1	29.4	22.1	22.6	25.0	27.1	27.4	27.2	24.0	23.6	25.7
16:1	6.3	6.4	5.3	2.8	3.9	6.2	4.4	2.9	2.9	6.3	7.0	5.0
16:2	tr	tr	0.3	0.5	0.2	0.3	0.6	0.8	0.5	0.4	0.4	0.4
17:0	1.2	1.3	0.2	0.8	1.1	0.6	2.1	2.3	1.4	1.1	0.7	0.8
18:0	6.1	5.4	5.5	6.1	6.9	5.1	7.5	8.9	9.7	3.0	3.0	4.5
18:1	16.6	18.3	17.5	21.7	22.4	16.4	17.8	17.8	21.4	16.2	15.0	17.2
18:2	0.7	0.9	0.7	0.8	1.2	0.3	0.9	0.9	1.2	2.1	1.7	2.1
18:4	2.2	2.5	1.7	0.9	1.0	1.9	tr	tr	tr	0.5	tr	tr
Unknown	tr	0.2	0.3	tr	tr	tr	0.7	0.8	.....	.....	.....	0.8
[a]18:3–20:?	0.6	0.8	0.5	tr	tr	0.7	0.4	0.5	.....	1.2	1.8	0.4
19:0	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr
20:1	2.7	3.0	2.1	6.3	5.3	5.4	1.1	1.7	2.0	2.0	2.4	2.2
20:4	1.2	1.4	0.8	1.0	1.2	1.0	3.6	3.6	3.2	3.0	3.4	2.4
20:5	6.5	6.8	5.7	6.4	6.0	9.8	4.6	4.5	4.0	13.2	13.3	9.5
21:0	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr
22:1	2.0	2.1	1.6	5.4	4.0	3.8	tr	tr	tr	tr	tr	0.4
22:3	0.4	0.3	tr	1.2	tr	0.5	1.0	0.4	1.2	0.7	0.5	0.6
22:4	1.0	0.8	0.9	1.1	1.3	0.7	2.4	1.5	1.6	1.0	0.7	1.3
22:5	0.8	0.8	0.5	1.4	1.2	1.3	1.3	0.7	1.2	1.5	0.7	1.0
22:6	17.6	16.4	20.4	17.1	15.2	15.0	22.2	22.2	19.0	17.3	18.2	20.0

[a] Indicates overlapping peaks on recorder chart.
A = area.
tr = trace amount.

were converted to their methyl esters, and the methyl esters were analyzed by GLC.

Preparation of Commercial Samples

Commercial samples of canned dietary pack (no added vegetable oil) and canned vegetable oil pack were obtained for each species. The fish used in the preparation of the commercial vegetable oil pack were from the same lots of fish used in the preparation of the laboratory samples; however, the commercial dietary packs were prepared from different lots of fish. Lipids were extracted from the canned products according to the scheme shown in Figure 1, converted to methyl esters, and analyzed by GLC.

Results and Discussion

A portion of each sample was hydrogenated and the GLC data for the hydrogenated samples were compared with the data for the corresponding nonhydrogenated portion. Table I presents this data for skipjack precook oil. The close correlation between hydrogenated and nonhydrogenated skipjack precook oil methyl esters exists for the rest of the analyses and helps substantiate the reliability of the GLC data.

Relative Amounts of Fatty Acids Present in Tuna Light Meat

Table II gives the fatty acid analyses for the various samples of the raw light-meat tuna, laboratory-canned tuna, and commercial dietary pack tuna (no added vegetable oil).

Although analyses are reported separately for each of the four species, the data cannot be taken as a basis for species differentiation because the results are based on the analysis of only two fish per species.

The data for the laboratory and the commercial samples show surprisingly good agreement. Furthermore, the agreement between species also is good, in general. The data indicate that in oil from light-meat tuna, 20 fatty acids with carbon atom to double bond ratios ranging from 14:0 to 22:6 comprised more than 98% of the oil. The principal fatty acids were the following: 14:0; 15.0; 16:0; 16:1; 18:0; 18:1; 20:1; 20:4; 20:5; and 22:6; with the three fatty acids—16:0; 18:1; and 22:6—constituting over 60% of the oil and with three other fatty acids—16:1; 18:0; 20:5—constituting about half of the remaining fatty acids. The principal difference among the species was for the fatty acid 22:1, which ranged from 1.6 to 5.4% in albacore and bluefin but was present in little more than trace amounts in yellowfin and skipjack.

In general, the monoenoic fatty acid content of the light meat ranged from 24 to 37%; oleic acid, 16 to 22%; C20:5 fatty acid, 4 to 13%; and C22:6 fatty acid, 15 to 22%.

Fatty acid analysis of the raw dark meat are presented in Table III. The data roughly is parallel to the analyses for the light-meat fatty acids (Table II).

It has been suggested that tuna dark meat may function in somewhat the same manner as the kidney. Braekkan (12) found that the B-vitamin content (with the exception of niacin) of bluefin dark meat was significantly higher than that in the light meat; likewise, the oil content of the dark meat was generally higher than the light meat (12). In the fish used in the present investigation, however, it was observed that the oil content of the dark meat was not significantly different from that of the light meat (Table IV).

TABLE III
GLC Analyses of the Fatty Acid Content of the Oil from Raw Tuna Dark Meats

Fatty acid carbon atoms: double bond	Fatty acid content			
	Albacore	Bluefin	Yellowfin	Skipjack
	A.%	*A.*%	*A.*%	*A.*%
14:0	3.7	4.0	2.8	4.1
15:0	1.2	0.7	0.8	0.9
16:0	24.5	25.7	24.4	19.8
16:1	3.9	3.5	4.5	4.1
16:2	0.2	0.4	0.2	0.5
17:0	1.0	0.7	1.6	1.0
18:0	5.9	12.5	7.7	4.6
18:1	18.0	14.5	17.9	10.7
18:2	0.9	0.6	1.4	2.3
18:4	2.1	1.0	tr	1.1
Unknown	0.3	tr	0.8	.....
[a] 18:3–20: ?	0.7	0.5	0.6	0.7
19:0	tr	tr	tr	tr
20:1	2.8	3.2	2.2	1.3
20:4	1.5	1.4	3.0	3.3
20:5	7.3	5.5	4.3	10.0
21:0	tr	tr	tr	tr
22:1	2.5	2.1	0.6	tr
22:3	tr	tr	0.5	1.1
22:4	1.2	1.1	3.4	1.7
22:5	0.7	1.9	1.4	1.7
22:6	21.6	21.0	22.1	30.0

[a] Indicates overlapping peaks on recorder chart.
A = area.
tr = trace amount.

TABLE IV
Oil Content of Tuna Samples

Species	Oil content of samples (wet basis)			
	Raw meat		Canned light meat	
	Light	Dark	Lab	Dietary
	%	%	%	%
Albacore	7.5	4.3	7.3	6.2
Bluefin	5.0	5.0	5.0	4.2
Yellowfin	0.6	0.7	0.5	0.7
Skipjack	0.9	1.0	0.8	0.8

Effects of Processing on Unsaturation

The commercial processing of tuna involves various steps in which the fish is precooked, cooled in air overnight, packed into cans, and pressure cooked. Some degradation of higher unsaturated fatty acids or differential separation of fatty acids might be anticipated under these conditions. However, neither the laboratory nor the commercially canned tuna indicated that this happened; the composition of the raw and the canned products were about the same (Table II), and the precook oils in the laboratory-canned pack and the dietary pack drainage oils (Table V) were about the same as the corresponding oils extracted from the meat (Table II).

TABLE V
GLC Analyses of Fatty Acid Content of Drainage Oils from Laboratory-Precook and Commercially-Canned (Dietary Pack) Tuna

Fatty acid carbon atoms: double bond	Fatty acid composition of drainage oils							
	Albacore		Bluefin		Yellowfin		Skipjack	
	Lab precook	Dietary pack	Lab precook	Dietary pack	Lab precook	Dietary pack	Lab precook	Dietary pack
	A.%	*A.*%	*A.*%	*A.*%	*A.*%	*A.*%	*A.*%	*A.*%
14:0	4.2	4.0	5.9	4.0	4.1	4.0	7.7	6.1
15:0	0.9	1.2	0.6	0.4	1.1	0.8	1.1	0.7
16:0	25.0	30.8	27.9	20.2	25.1	26.0	23.4	23.7
16:1	6.8	5.1	3.7	4.2	6.7	3.0	7.2	6.3
16:2	0.8	0.3	0.4	0.7	0.3	tr	0.5	0.4
17:0	1.8	1.4	0.8	1.1	0.9	1.5	1.0	0.4
18:0	4.6	5.6	5.2	5.1	6.4	7.2	3.0	4.8
18:1	15.1	18.4	17.4	14.9	19.2	20.8	14.6	15.8
18:2	0.7	0.7	0.8	0.8	1.1	2.1	1.9	2.1
18:4	3.2	1.1	1.2	2.2	tr	tr	tr	tr
Unknown	tr	0.4	tr	tr	1.0	1.2	.....	0.3
[a] 18:3–20: ?	0.8	0.5	0.8	1.4	0.5	1.2	1.8	tr
19:0	tr	tr	tr	tr	tr	tr	tr	tr
20:1	3.6	3.5	4.4	6.8	2.8	3.6	2.3	2.3
20:4	1.3	0.9	1.7	1.9	2.5	2.0	3.5	3.3
20:5	7.3	5.8	6.2	10.3	6.4	5.3	13.2	10.7
21:0	tr	tr	tr	tr	tr	tr	tr	tr
22:1	3.0	2.3	3.9	4.4	tr	tr	tr	tr
22:3	0.3	0.4	1.1	1.0	0.5	1.9	0.6	1.4
22:4	0.9	0.9	0.5	1.7	0.9	1.7	0.7	1.4
22:5	0.7	0.6	1.1	1.1	1.1	1.7	0.8	1.8
22:6	18.6	16.4	16.3	17.6	19.6	16.0	17.6	17.7

[a] Indicates overlapping peaks on recorder chart.
A = area.
tr = trace amount.

Contribution of Tuna Oil in Vegetable Oil Pack

Most commercially packed tuna contains added vegetable oil, usually soybean, resulting in a quantity of vegetable oil many times greater than that of the natural tuna oil. The present study showed that under these conditions, an analysis of the drained or extracted oil from such a pack reflects almost entirely the fatty acid content of the vegetable oil and that the fatty acid characteristics of the fish oils are obscured. For that reason, the data on the analyses of the vegetable-oil pack tuna are not presented.

Conclusions

1.) Twenty fatty acids comprising more than 98% of those present in the oil of albacore, bluefin, yellowfin, and skipjack tuna were identified. The principal fatty acids were: 14:0; 15:0; 16:0; 16:1; 18:0; 18:1; 20:1; 20:4; 20:5; and 22:6; with the three fatty acids—16:0; 18:1; and 22:6—constituting over 60% of the oil and with three other fatty acids—16:1; 18:0; and 20:5—constituting about half the relative weight of the remaining fatty acids. Except for the three fatty acids—20:5; 22:1; and 22:6—the data for the light and dark meats of all four species were closely comparable.

It was observed that the oil content of the light and dark meat did not appear to differ significantly.

2.) There was no marked degradation of unsaturation nor was there differential extraction of fatty acids due to processing.

3.) Owing to the preponderantly greater amount of vegetable oil present, the relative amount of tuna oil, which is important from a dietary standpoint, in either (a) the drained oil or in (b) the rendered oil in a vegetable oil pack could not be determined by the GLC techniques used in these studies.

ACKNOWLEDGMENTS

Procurance of the raw tuna and necessary arrangements made with the California Tuna Industry for preparing the dietary samples by Clarence Carlson; assistance in preparation and canning of the laboratory samples by L. G. McKee.

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[Received July 7, 1962—Accepted December 14, 1962]

Bleaching Off-Colored Cottonseed Oils with Activated Alumina: A Preliminary Cost Study[1]

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Abstract

The majority of off-colored cottonseed oils can be bleached to a prime color with 4% by weight or less of activated alumina in a single operation. Increasing the amount of alumina beyond 4% makes it possible to bleach the most difficult-to-bleach off-colored cottonseed oils. Although additional research is required to establish the process and optimize conditions, results of a preliminary cost study indicate that this method should be competitive on a large scale with rerefining followed by conventional earth bleaching.

A flow sheet is given. Investment and operating costs are reported for bleaching for six months annually in plants having daily capacities of 100,000, 500,000, and 1,000,000 lb of cottonseed oil, prime and/or off-colored, in batches of 6,000, 30,000, and 60,000 lb, respectively.

It is estimated that alumina bleaching of off-colored oil, with solvent extraction of oil from spent alumina, would cost as little as 0.4¢/lb in the largest plant, 0.5¢/lb in the medium plant, and 1.2¢/lb in the small plant. These costs are calculated on the basis of the use of 4% alumina by weight of oil for off-colored oil during one-fourth of the season, in combination with 1%, 2%, or 4% of alumina for prime oil during three-fourths of the season.

Costs could be lowered by reducing oil losses and losses of alumina in regeneration, increasing filtration rates, and lowering alumina price as a result of additional research on its preparation. Lowered cost would make the method more attractive for *prime* oils as well.

Introduction

IT IS ESTIMATED that in recent years, off-colored, bleach-resistant oils might have constituted as much as 25% of cottonseed oil production in the U. S. This is equivalent to 450 million lb of the 1.8 billion lb of cottonseed oil produced annually. These oils are either rerefined, overrefined, or blended with lighter oils. Because of costs, the trend in the industry is to do as little rerefining as is necessary and to blend as much as possible. Since blending is dependent upon the availability of an adequate supply of sufficiently light oils, there is a real need to develop a process for bleaching the off-colored oils at lower cost. In early work by Swift et al. (4), activated alumina was used in combination with activated carbon, bleaching earth, and deodorization to reduce unsaponifiable matter, color, and tocopherol content of cottonseed oil. Later, Fisher and Bickford (1) reported that natural antioxidants and color bodies could be removed from vegetable oils by adsorption on activated alumina and carbon. Subsequently, Pons et al. (3), reported that activated alumina was found to be a superior adsorbent for removing red color bodies from cottonseed oil.

[1] Presented at the AOCS meeting in New Orleans, La., 1962.
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