MASKING UNDESIRABLE FLAVORS IN FISH OILS'

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ABSTRACT

The odor of fish oil used medicinally may require masking by flavoring materials. During a search for suitable masking materials, fresh, specially refined menhaden oil having a minimum of flavor was stored with and without added flavoring materials for 5 days at 75° F and for longer periods at several lower temperatures. In initial preliminary screening tests with 66 different flavoring materials. the masking of rancid or other unpalatable flavors developing in the stored oil was evaluated by a small panel. In later tests, a large consumer-type panel consisting of untrained laboratory personnel was used to determine the preference for the flavors of those materials that worked best in the screening test and that were of a type approved by the U.S. Food and Drug Administration for use in foods. Several flavoring materials showed promise, particularly those having the flavor of root beer, lemon, wintergreen (methyl salicylate), and wild cherry.

When fish oil is used medicinally-for example, as a cholesterol depressant or as a source of vitamins-the presence of "fishy," rancid, or other flavor components may make it unpalatable. One way of overcoming this problem is to add some flavoring material that will mask the undesirable flavor. It is important that the added flavor be one that is pleasing to most consumers so that we do not merely substitute one undesirable flavor for a slightly more undesirable one. The aim of this work therefore was to find flavoring materials, approved for food use, that would adequately mask the unpleasant flavor components developing in stored menhaden oil (the fish oil produced in largest quantity in the United States) and that would also have a flavor pleasing to most customers.

We carried out this study in two experiments. In Experiment 1, reasonably palatable menhaden oil refined by a special technique was stored for 5 days at room temperature with and without added flavoring materials. A small trained panel was used to determine the efficiency of over 66 different flavoring materials

in masking the developing undesirable flavor components in the stored oil. Then, in Experiment 2, those materials giving the best masking action, excluding any not approved for use with food, were further tested by a larger consumertype panel for hedonic rating of the different flavors resulting from adding the selected flavoring materials to the oil before it was stored.

EXPERIMENT 1-MASKING TESTS

The first series of tests were set up as a rapid screening of many flavoring materials to see which ones best masked the undesirable off flavors that develop in stored menhaden oil. At this stage, no consideration was given to individual preference for the flavor additive, which was investigated in Experiment 2.

MATERIALS

Menhaden oil was specially refined by a combination of clay bleaching, molecular distillation, and treatment with massive quantities of silica gel (Stansby and Jellinek, 1965). This treatment yielded an oil that was free of fishy and rancid flavor components but that still retained some small burnt flavor.

Sixty-six flavor additives that were screened initially included synthetics and isolates.

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undiluted and in alcoholic solution, essential oils, and imitation flavoring compositions. The additives were obtained from many sources.

METHODS

In the initial work, 5-ml samples of menhaden oil, with or without flavor additive, were held in covered plastic petri dishes (100 by 15 mm) for 5 days at 75° F (22° C). Preliminary tests indicated that 1 part by volume of flavoring material per 400 parts by volume of oil gave about the proper concentration with most flavor preparations, so this concentration was used throughout the work. With the large surface of oil exposed to air in this test, a fairly advanced stage of rancidity was reached in 5 days.

In some of the later work in which larger samples were required, oil in both 200-ml (8-oz) and 25-ml (1-oz) stoppered bottles was stored: (1) at 40° F (4° C) for 8 to 12 weeks, (2) at -20° F (-29° C) for 8 to 12 weeks, and (3) at room temperature—that is, at 75° F (22° C) for 4 weeks.

For various aspects of this work, especially in preliminary observations, paired comparison tests, duo-trio tests, ranking tests and descriptive tests—all described by Jellinek (1964) were used. For most of this work, however, descriptive tests and ranking tests were used; only the descriptive and ranking tests will be tabulated in this report.

Samples of oil were tasted by immersing the tip end of a plastic spoon into the oil and placing the spoon into the mouth. This procedure avoided coating the lips with oil, and it eliminated much of the objection of some panel members to an undesirable oily feel during protracted examination of many samples.

The panel used in this part of the work consisted of 5 to 7 laboratory workers all of whom had had some previous experience with odors and flavors of fish oils. Several preliminary training sessions were held in order for them to become familiar with and be able to use consistent terminology for the odors and flavors to be encountered in the experimental work.

RESULTS

Initial Screening Test

In an initial screening test of all 66 flavoring compounds, a degree-of-masking scale was used as follows: ++, very good masking; +, good masking; \pm , good masking when first tasted but did not mask aftertaste; ?, questionable masking; —, negative masking—that is, the taste was not masked (Table 1). The flavoring materials are listed in alphabetical order within each category.

Main-Examination Test

We then made a more detailed examination, paying greater attention to the type of flavor which sometimes varied at different stages during the storage of the oil. The following comments show certain limitations of many of the flavor additives.

Flavoring materials with "green" or "floral" flavor properties.—Because fresh fish oil has a "green-grassy" or "green-cucumber" odor and flavor, two green standards had been worked out in earlier experiments (Stansby and Jellinek, 1965). These standards were cis-hexen-3-ol-1 and Green Aroma Hr (coded sample of Haarmann and Reimer, Holzminden, West Germany," probably: nona-2-enal or nona-2-dienal or a mixture of both (Forss, Dunstone, Ramshaw, and Stark, 1962). These two green standards were tried as masking agents.

Cis-hexen-3-ol-1 contributed a definite greengrassy note to the menhaden oil and also showed very good masking abilities in the exposed menhaden oil.

Green Aroma HR contributed a green-cucumber note to the fish oil but seemed to have prooxidant qualities. In comparison with the control sample, the menhaden oil got rancid faster and reached a higher intensity. These observations seem to be similar to those made about 20 years ago on butter with a high content of di-

¹ The use of trade names is merely to facilitate descriptions; no endorsement is implied.

TABLE	1.—Effectiveness	of	66	flavoring	materials	in	masking	the	fishv	flavor	and	odor	of	menhaden	oil
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Category and substance	Flavor type and descriptions	Masking effect	Category and substance	Flavor type and descriptions	Masking effect
Synthetics and isolates			Essential oils-Con.		
Acetoin Agrumen aldehyde Anethol, U.S. Pharmacopoeia (U.S.P.) Benzaldehyde i-Butyl quinoline	butterlike fruity-citrus spicy-anise sweet-bitter almond green-spinachlike or earthy (asparagus)	± ++ 	Lemon oil, U.S.P. Lime oil Orange oil, U.S.P. Peppermint oil Root beer oil Spegrmint oil, rectified	fruity-lemon fruity-lime fruity-orange cooling-peppermint wintergreen group cooling-spegremint	+ + - ++
Carvol Cinnamic aldehyde Citral Ethyl phenyl acetate Ethyl salicylate Eucalyptol Eugenol Foretine Geranyl acetate Geranyl acetate Geranyl acetate Geranyl propionate cis-hexen-3-ol-1 i-Jasmone Linalool Melonal (2,6-dimethyl-5-hepten-1-al)	spicy-caraway spicy-cinnamon fruity-lemon sweet-honeylike wintergreen cooling spicy-cloves green-mushroomy/earthy green-leafy & floral-rosy green-leafy & floral-rosy green-leafy & floral-rosy green-leafy & floral-rosy green-leafy & floral-rosy green-leafy & floral-rosy green-leafy floral floral furty-melony		Spearmint oil, rectified Imitation flavoring compositions Blood Orange Flavor Imitation Boysenberry Flavor Butter Aroma Cherry Bouquet Imitation Cranberry Flavor Fruity Bouquet Green Aroma HR Imitation Honey Flavor Imitation Lemon Juice Flavor #51.124A Lemon Mint Flavor Lime Mint Flavor Imitation Melon Base Orange Mint Flavor	cooling-spearmint fruity-baysenberry butterlike fruity-cherry or sweet ch fruity-cranberry fruity green-cucumber sweet-honeylike fruity-lemon fruity-lemon fruity-lime fruity-orange	- + + + + + + + + + + + + + + + + + + +
Menthol Methyl nonyl ketone Methyl salicylate Styralyl acetate -Undecalacton Vanillin Essential oils Anise oil, U.S.P. Bay oil Caraway oil Caraway oil Casia oil Cinnamon oil, U.S.P. Clove oil Ginger oil	cooling green-? wintergreen green fruity-peach sweet-vanilla spicy-anise spicy-bay spicy-caraway spicy-caraway spicy-casia spicy-cosia spicy-clove spicy-ginger	 +++ ? +++ +++ +++ 	Tetrarame Orange Violet Flavor Imitation Essence Arrack Imitation Butterscotch Oil Brandy Flavor Imitation Oil Soluble Flavor Wild Cherry #12009 Imitation Oil Chocolate Imitation Essence Cacca Malt True Concentrate Imitation Aroma Maple Imitation Oil Rum, Jamaica #017 Tutti Frutti Oil	fruity-citrus floral-violet fruity-brandy sweet-caramel brandy-like sweet-caramel-vanilla fruity-cherry sweet-caramel-chocolate sweet-malt-chocolate sweet-malt sweet-maple fruity-rumlike fruity-candy	+ + + + + + + + + + + + + + + +

acetyl. Butter with a high diacetyl content has more butter flavor and turns rancid faster than does butter with a low diacetyl content. For this reason, export butter has to be washed carefully to lower the content of diacetyl (Moncrieff, 1951).

In addition to these two green standards, some other chemicals with green character or floral character, or both, were tried but without success. They are listed among the synthetics and isolates above. Some of them—namely, i-butyl quinoline, foretine, i-jasmone, methyl nonyl ketone, and styralyl acetate—contributed sharpness and a biting note to the menhaden oil.

Geraniol, geranyl acetate, geranyl butyrate, and geranyl propionate contributed a green, somewhat leafy note combined with sharpness. In addition, a flowery "rosy" note was observed. These materials have been recommended repeatedly by aroma companies. However, the materials do not seem to be suitable for use with menhaden oil. The same observation was made with "violet" flavor.

Flavoring materials with "fruity" flavor properties.—Citral added a refreshing lemon odor and flavor to fish oil; but in addition, it is sharp in taste with the sharpness lingering in the aftertaste. Repeat tests with other concentrations confirmed that citral alone is too sharp. In combination with fruity flavoring compositions, it might help to introduce a refreshing note.

Melonal (2,6-dimethyl-5-hepten-1-al) contributed a fruity refreshing note, lingering in the aftertaste. In addition, it gave a greenness similar to that described by some panel members as "green-melony." Unfortunately, the sample turned rancid rapidly toward the end of the 5-day period of storage when melonal was added.

 γ -Undecalactone (peach lactone), when used alone, is too artificial in odor and too sharp in flavor.

Lemon oil made the odor of the menhaden oil aromatic-fruity. It was retested with good results.

Lime oil gave a refreshing citrus taste to the menhaden oil, contributed some astringency, but it did not linger in the aftertaste. The aftertaste was more lemon-"candy"-like. (It was probably not pure lime oil; artificial flavoring material probably had been added, giving this "candy"like flavor.)

Orange oil has good masking properties.

Blood Orange Flavor, Lemon Juice Flavor, Lemon Mint Flavor, Lime Mint Flavor, Orange Mint Flavor, Tetrarome Orange had good masking properties.

Imitation Boysenberry Flavor and Imitation Cranberry Flavor had masking properties for the odor rather than for the flavor. In addition, the flavor was quite artificial and unpleasant. The fruits that were imitated were not recognizable as such either in odor or in flavor. We tried to obtain a natural cranberry oil, but it is not available on the market.

Cherry Bouquet was outstanding in the masking properties of rancidity and oily feeling.

Wild Cherry Flavor also had very good masking properties.

Fruity Bouquet had very good masking properties; the flavor was fruity-candylike.

Imitation Melon Base, in alcoholic dilutions, gave a fruity-melon type of odor. In undiluted form, it smelled fruity and honey sweet. The oily feeling was well masked in the flavor with fruitiness and sweetness lingering in the aftertaste. After the oil was exposed for 1 day, there was in addition some lingering sharpness.

Imitation Oil Rum had good masking properties.

Flavoring materials with "butterlike" flavor properties.—Acetoin contributed a butterlike flavor and feeling, lingered in the aftertaste. The butter feeling is perceivable longer in the aftertaste than is the oily feeling. Unfortunately, the sample became rancid after only 3 days, indicating some prooxidant effect.

Butter Aroma showed good masking properties but only at the start of the test.

Flavoring materials with "sweet" flavor properties.—Benzaldehyde (bitter almond flavor) alone is not satisfactory. It has to be used in a flavoring complex such as Cherry Bouquet.

Ethyl phenyl acetate and Imitation Honey Flavor mask the oily feeling. In addition they contribute a honey flavor.

Vanillin was tried in different concentrations, but the results obtained were not satisfactory.

Flavoring materials with "spicy" flavor properties.—Anethol contributeed an anise flavor. It masked the oily feeling very well and changed it into a pleasant anise flavor (similar to that of cough drops flavored with anise oil or anethol).

Carvol and eugenol were not suitable. They added a sharp, somewhat burnt note to the menhaden oil.

Anise oil showed very good masking properties.

Bay oil, caraway oil, clove oil, and ginger oil were not suitable. They added a sharp, somewhat burnt note to the fish oil.

Cassia oil and cinammon oil were equally suitable.

Flavoring materials of the "wintergreen" group.—Ethyl salicylate, methyl salicylate, and root beer oil all masked the oily feeling very well.

Flavoring materials with "cooling" flavor properties.—Menthol, peppermint oil, and spearmint oil contributed initially only a bland taste and pleasant oily feeling to the menhaden oil. Samples with eucalyptol had a sharp note in flavor. All samples had a rancid flavor at the end of the 5-day storage test at room temperature.

Reduction in the List of Potential Masking Materials

The number of potential flavoring materials for masking the objectionable flavor components in the menhaden oil, based upon the preliminary screening tests, was too large to subject them all to a large-scale consumer-type test and therefore had to be decreased. The most promising flavoring materials for masking were:

Synthetics and isolates:

Anethol Ethyl phenyl acetate Ethyl salicylate cis-hexen-3-ol-1 Methyl salicylate

Essential oils: Anise oil, U.S.P. Cassia oil Cinnamon oil, U.S.P. Lemon oil Orange oil, U.S.P. Root beer oil

Imitation flavoring compositions: Blood Orange Flavor Butter Aroma Cherry Bouquet Fruity Bouquet Imitation Honey Flavor Imitation Lemon Juice Flavor #51.124A Lemon Mint Flavor Lime Mint Flavor Orange Mint Flavor Tetrarome Orange Imitation Oil Rum #017 Imitation Oil Wild Cherry #12009 Tutti Frutti Oil

Because using more than a dozen flavoring materials in the large-scale consumer tests was not desirable, we considered limiting further the number of flavoring materials for additional study. The results of this phase of the examination permitted us to eliminate additional materials, based upon the following observations.

Ethyl phenyl acetate was described as perfumelike by some consumers and was therefore disliked by them.

Ethyl salicylate was partly liked, partly dis-

liked by different panel members. The scores for methyl salicylate were higher.

Cis-hexen-ol-1 tested in samples stored at 40° F and -20° F for 8 weeks did not result in observable rancidity. The samples still had a greengrassy character. The control sample stored at 40° F was rancid; the control sample stored at -20° F was almost odorless and was faint cucumber green in flavor.

Even though cis-hexen-3-ol-1 masked the oily feeling and rancidity, it was not liked by the panel.

Cassia oil and cinnamon oil have much the same flavor; cinnamon oil was therefore arbitrarily selected for further experiments.

To reduce the number of fruity flavors, we made a preliminary consumer test. From six flavors—Blood Orange, Lemon Juice, Lemon Mint, Lime Mint, Orange Mint, and Tetrarome Orange—Lemon Juice Flavor was chosen for further experiments.

Fruity Bouquet with its candy, fruitlike flavor was disliked by some panel members who described it as being perfumelike.

Cherry Bouquet not only was outstanding in the masking properties of rancidity and oily feeling but received the highest rating in the consumer test.

Only flavoring materials allowed by the U.S. Food and Drug Administration could be considered. The Cherry Bouquet and Fruity Bouquet are perfume compositions and are not permitted as food additives.

Butter Aroma was not satisfactory. As was already experienced with acetoin, samples flavored with a butter-aroma composition turned rancid faster in the storage test at 40° F than did the control. The 40° F sample was definitely rancid after 8 weeks of storage, although the samples held below freezing were not.

Imitation Honey Flavor was too artificial in flavor and therefore disliked by most of the panel.

Masking of Fish Oil with Pronounced Burnt Flavor

As was described in Materials, menhaden oil was refined both by the regular three-stage (clay bleaching, molecular distillation, silica gel) process and by a modified two-step process in which the silica-gel procedure was eliminated but in which the collection of distillate during molecular distillation was restricted. Such a procedure greatly simplified refining but resulted in a menhaden-oil product containing considerable burnt-flavor component. Flavoring materials giving best results with the latter oil did not necessarily give optimum results with the silica gel refined oil.

Of the flavoring materials found to be most successful for the ordinarily refined procedure, those with a fruity-type flavor-especially Cherry Bouquet and Fruity Bouquet-masked the burnt flavor better than did such flavors as root beer oil, methyl salicylate, or anethol. None of these compounds was able to mask the burnt flavor completely when added to the freshly distilled oil. After 6 weeks of storage at 40° F (4° C) samples masked with Cherry Bouquet or Fruity Bouquet had no burnt flavor, but considerable remained when root beer oil or methyl salicylate was used, and an intermediate amount remained when anethol was used.

Flavoring materials having good masking properties and giving promise for good consumer acceptability were as follows: anethol, anise oil, cinnamon oil, lemon oil, Lemon Juice Flavor, methyl salicylate, orange oil, root beer oil, Imitation Oil Rum, Imitation Oil Wild Cherry, and Tutti Frutti Oil.

When considerable burnt flavor is present in menhaden oil, such as occurs when the two-stage modified refining process is used, none of the masking agents completely obscures this burnt flavor. Certain fruity flavors such as Cherry Bouquet or Fruity Bouquet are the most effective in masking this flavor, with methyl salicylate or root beer oil being less effective.

EXPERIMENT 2-CONSUMER-TYPE TESTS

The next stage of the investigation was to determine consumer hedonic rating for the several masking substances that had been found to be almost equally suitable for disguising the objectionable flavors developing in menhaden oil during storage.

MATERIALS

The same materials-both menhaden oil and flavor additives—were used in this phase.

The flavors used were those that in Experiment 1 had given the best masking results. Any flavors that were used in Experiment 1 and that were not approved for food additive use by the U.S. Food and Drug Administration were eliminated in this part of the work. Table 2 shows the identity and source of these substances.

TABLE 2.-Flavor components rated in the consumer test.

Flavor material	Suppliers ¹
Anethol, U.S.P.	1,4
Anise oil, U.S.P.	3
Cinnamon oil, U.S.P.	3
Lemon oil, U.S.P.	4
Imitation Lemon Juice Flavor #51.124A	2
Methyl salicylate	1,4
Orange oil, U.S.P.	4
Root beer oil	3
Imitation Oil Rum #017	4
Imitation Oil Wild Cherry #12009	4
Tutti Frutti Oil	4

Felton Chemicals Co., Inc., Brooklyn, N.Y.
Firmenich, New York, N.Y.
Fiorasynth Laboratories, Inc., New York, N.Y.
Fritzche Brothers, Inc., New York, N.Y.

METHODS

A modified molecular distillation procedure was used. This procedure eliminated the necessity for carrying out the time-consuming treatment with silica gel. The oil was distilled only until the pot residue amounted to 10 % (as contrasted to 3 % in the usual methods). This decreased amount of distillation resulted in much less burnt flavor (although in considerably more burnt flavor than when a silica-gel treatment was used). The oil could be used. however, directly without silica-gel treatment when sufficiently effective masking agents were used.

Oils were stored at (1) 40° F (4° C) (2) at -20° F (-29° C) for 4 to 12 weeks.

The panel for the consumer-type test consisted of Bureau of Commercial Fisheries personnel who had no previous experience in taste testing. The 15 to 20 participants were asked to rate

Name Date Please indicate by check mark ($$) how much you like or dislike each sample and rank it in order of preference (1-best, 2-second best, etc.).								
Hedonic Scale	Scale	Code	Code	Code				
Like very much	1							
Like moderately	2							
Neither like nor dislike	3							
Dislike moderately	4							
Dislike very much	5							
Comments:								
Too weak								
Satisfactory								
Too strong								
Flavor recognized as:								
Ranking order:								

FIGURE 1.-Score sheet for the consumer test.

two to three flavored samples on a hedonic scale, using the score sheet shown in Figure 1.

Five ml of oil was rated by the panel 5 days a week for about 3 months. It was necessary to determine whether a flavor, liked initially, was still liked after continuous intake. Likes and dislikes were expressed on an hedonic scale of 5 points (Fig. 1).

RESULTS

Table 3 summarizes the consumer ratings over the period of test.

Some consumers were consistent in their rating throughout the entire period of the test. Other consumers were consistent only with some flavors but were inconsistent with others.

DISCUSSION

Table 3 shows that all of the tested flavors received high ratings (1 or 2) as main ratings by the consumer-type panel. It should be noted that these high ratings were not given by the consumer panel as a whole but rather by groups within the panel. It also should be noted that there is a split of like and dislike for the same flavor.

This split is understandable, and it may be due to different causes. Individuals with various background show widely different flavor preferences. For example, Europeans who are used to methyl salicylate being used primarily for flavoring medicinal products or in disinfectants would doubtlessly have rated this substance lower for fish-oil masking than did our American panel. In parts of Asia where anise seed

TABLE 3.-Consumer ratings for different flavored fish oil.

Flavoring material added to fish oil	Hedonic ratings ¹							
Anethol	1,	2,		(4)				
Anise oil	(1)	2,		(4)				
Cinnamon oil	1,	2,	(3)	4				
Lemon oil	(1)	2,	(3)	(4)				
Imitation Lemon Juice Flavor #51.124A	1,	2,		(4)				
Methyl salicylate	1,	2,	3,		(5)			
Orange oil	(1)	2,	3,	(4)				
Root beer oil	1,	2,	(3)	(4)				
Imitation Oil Rum #017	(1)	2,	(3)	(4)	(5)			
Imitation Oil Wild Cherry #12009	1,	(2)	З,	(4)				
Tutti Frutti Oil	1,	2,		(4)				

¹ Rating numbers not in parentheses were the ones given by most consumers; those in parentheses were the ones given by fewer membersfor example, lemon oil was liked moderately (Rating 2) by most consumers, but some gave a higher rating (1) and some gave a lower rating (3) or (4). is grown, frequently anise is disliked as a flavoring material. In addition to differences due to nationality, childhood experience has a definite influence on likes and dislikes. Orange oil is a good example. Panel members who had been forced during childhood to take orange-flavored cod-liver oil as a medicine objected to orange flavor. As can be observed in daily food intake where some persons prefer sweet foods and others prefer acid foods, there is likewise the same split of like and dislike with sweet flavoring materials (cinnamon oil and Tutti Frutti Oil) and with acid flavors (Lemon Juice Flavor).

Even though the consumer-type panel as a whole did not rank the flavors in a definite order of preference, a pronounced like for any of the offered flavors by smaller consumer groups is obvious. This finding should encourage trial of these fish-oil masking flavors with larger consumer groups.

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