Fatty Acid Composition as a Basis for Identification of Commercial Fats and Oils

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ABSTRACT

Gas liquid chromatographic analyses of 200 samples of commercial fats and oils were compared to the standard ranges specified by the Food and Agriculture Organization/World Health Organization Codex Alimentarius Committee on Fats and Oils at its seventh session, London, England, March 25, 1974. Only six samples fell notably outside the standard ranges. On the basis of this information, the U.S. delegation is offering for consideration by the Committee at its next meeting a method for using the fatty acid composition of a fat or oil to determine its authenticity.

INTRODUCTION

At the seventh session of the Food and Agriculture Organization/World Health Organization Codex Alimentarius Committee on Fats and Oils in London, March 1974, a majority of the delegates supported the view that gas liquid chromatographic (GLC) analysis of component fatty acids of fats and oils provides useful evidence of their authenticity. Accordingly, a single range of values for each fatty acid in 10 commercial fats and oils (Table I) was agreed upon (1). These ranges were established by the Committee members through negotiations and represent their collective experience and judgment. Comments on the validity of the ranges were to be solicited from member nations. The ranges were based on specifications originally presented to the Codex Committee by the AOCS (2) but were modified substantially by the delegates. The validity of the original specifications was tested by comparing them with 200 examples of GLC analyses taken from the literature (3). This same set of data has now been compared to the Codex Committee's final specifications. It is conceivable that some of these literature sources may have contributed to the delegates' decision, but the extent of such influence, if any, is unknown. When the broad extent of nationalities and degrees of experience represented by the Committee is considered, the data base should not be severely prejudiced.

EXPERIMENTAL PROCEDURES

To facilitate handling of the data, computer programs were written to compare proportions of fatty acids in each of the 200 samples with the corresponding ranges given for each fat. If the value for a particular fatty acid fell outside the specified range for a fat, the absolute value of the difference between this value and the nearest range limit was calculated. The total of these differences for all fatty acids was considered as the "deviation" of the sample from that particular fat. After all 10 deviations were established, they were ranked in ascending order. The oil giving least deviation was first choice; the second least, second choice; etc. A correct first choice meant that the sample was correctly identified.

RESULTS AND DISCUSSION

Data from computerized matching of the 200 samples are summarized in Table II. Of these, 194 samples were correctly identified as first choice. In the other six, it appeared as second choice. The data also show that 194 samples deviated $< 2\%$ from Codex specifications.

Almost all cottonseed oils deviated some from Codex ranges. Usually, deviation was from 0.1 to 0.2% and appeared with the C14:0 or C16:1 acids. Accuracy of the GLC data for these components may have led to these minor errors. Noteworthy, from the data in Table II, is the apparent overlap between specifications for sunflower and safflower oils as shown by the nine sunflower oils that gave zero deviation from either Codex range. A slight modification in safflower oil specifications would entirely eliminate such an ambiguity. Likewise, two samples of sesame oil also gave zero deviation from sunflower oil ranges. This overlap is even less serious inasmuch as a test can be used to identify sesame oils (4). In fact, this test is a part of the Codex Alimentarius Commission's Recommended International Standard for edible sesame seed oil (5).

On the basis of our results, the United States Codex delegation is offering for consideration a method to use the fatty acid composition of an oil to determine whether or not it should be accepted as labeled. The method is:

1. Compare GLC determined percentage of each fatty acid in an oil or fat sample with the corresponding range established by the Codex Alimentarius Fats and Oils Committee.
2. For each acid, record in absolute percentage the amount by which the sample composition falls outside the prescribed range.
3. Add all the deviations to obtain the arithmetic sum.
4. If the arithmetic sum is 2% or less, accept the sample as having the claimed identity.
5. If the total deviation is $> 2\%$, compare the sample in question with the other nine fats and oils for which GLC determined fatty acid compositions have been agreed upon by the Codex Committee.
6. Accept the claimed identity for the sample if step 5 does not lead to a smaller total deviation from specified fatty acid composition ranges for one of the other fats or oils.

The 2% maximum total deviation indicated in step 5 was arbitrarily selected, but with it, the application of steps 1-4 led to correct identification of 194 out of 200 samples in an evaluation of the method. Subsequent application of steps 5 and 6 would have permitted acceptance of the remaining six samples.

The following examples illustrate the proposed method:

Example 1. A sample labeled peanut oil (Arachis) by GLC contained 7.4% palmitic acid, 5.3% stearic acid, 35.7% oleic acid, 44.4% linoleic acid, 0.9% arachidic acid, 0.6% eicosenoic acid, 5.1% behenic acid, and 0.6% lignoceric acid.

Steps 1-3. Comparison with prescribed ranges:

Acid	% in sample	Codex range	Deviation
16:0	7.4	6.0-15.5	
18:0	5.3	1.3-6.5	
18:1	35.7	36-72	0.3
18:2	44.4	13-45	
20:0	0.9	1.0-2.5	0.1
20:1	0.6	0.5-2.1	
22:0	5.1	1.5-4.8	0.3
24:0	0.6	1.0-2.5	0.4
Total	100.0		1.1

TABLE I

Fatty Acid Composition of Fats and Oils Determined by Gas Liquid Chromatography[a]

Fatty acid	Arachis	Cottonseed	Lard and rendered pork fat	Maize	Mustard seed	Premier Jus and edible tallow	Safflower seed	Sesame seed	Soybean	Sunflower seed
C<14	<0.1	<0.1	<0.5	<0.1	<0.5	<0.1	<0.1	<0.1	<0.1	<0.1
C14:0	<0.1	0.5-2.0	0.5-2.5	<1.0	<1.0	1.4-6.3	<1.0	<0.5	<0.5	<0.5
C14:1			<0.2			0.5-1.5				
C15:0			<0.1			0.5-1.0				
C15:ISO			<0.1			<1.5				
C16:0	6.0-15.5	17-29	20-32	8.0-19	0.5-4.5	20-37	2.0-10	7.0-12	7.0-12	3.0-10
C16:1	<1.0	0.5-1.5	1.7-5.0	<0.5	<0.5	0.7-8.8	<0.5	<0.5	<0.5	<1.0
C16:2						<1.0				
C16:ISO			<0.1			<0.5				
C17:0			<0.5			0.5-2.0				
C17:1			<0.5			<1.0				
C17:ISO										
C18:0	1.3-6.5	1.0-4.0	5.0-24	0.5-4.0	0.5-2.0	6.0-40	1.0-10	3.5-6.0	2.0-5.5	1.0-10
C18:1	36-72	13-44	35-62	19-50	8.0-23	26-50	7.0-42	35-50	19-30	14-65
C18:2	13-45	33-58	3.0-16	34-62	10-24	0.5-5.0	55-81	35-50	48-58	20-75
C18:3	<1.0	0.1-2.1	<1.5	<2.0	6.0-18	<2.5	<1.0	<1.0	4-10	<0.7
C20:0	1.0-2.5	<0.5	<1.0	<1.0	<1.5	<0.5	<0.5	<1.0	<1.0	<1.0
C20:1	0.5-2.1	<0.5	<1.0	<0.5	5.0-13	<0.5	<0.5	<0.5	<1.0	<0.5
C20:2			<1.0		<1.0					
C20:4			<1.0			<0.5				
C22:0	1.5-4.8	<0.5	<0.1	<0.5	0.2-2.5		<0.5	<0.5	<0.5	<1.0
C22:1	<0.1	<0.5			22-50					<0.5
C22:2					<1.0					
C24:0	1.0-2.5	<0.5		<0.5	<0.5					<0.5
C24:1					0.5-2.5					<0.5

[a]These ranges, tentatively adopted by the Food and Agriculture Organization/World Health Organization Codex Alimentarius Committee on Fats and Oils, refer to typical commercial samples of bona fide fats and oils. A range of <0.1% indicates that the fatty acid is not normally present in a quantifiable amount, whereas a blank indicates that the fatty acid is not normally present.

TABLE II

Comparison of 200 Gas Liquid Chromatographically Determined Fatty Acid Compositions with Tentative Codex Alimentarius Ranges

Fat or oil	Number of samples	Correct first choice	Deviation from correct oil (absolute value)						
			0	<1	<2	<3	<4	<5	<6
Arachis	14	14	7	2	3	1			1
Cottonseed	53	53	1	46	4	1		1	
Lard and rendered pork	7	7	6	1					
Maize	35	35	34	1					
Mustard seed	4	4		1	2		1		
Premier Jus and edible tallow	4	2			3		1		
Safflower seed	14	14	13	1					
Sesame	7	3	2[a]	5					
Soybean	22	22	21	1					
Sunflower seed	40	40	39[b]	1					
Total	200	194	123	59	12	2	2	1	1

[a]Two samples had zero deviation from both sesame and sunflower specifications.

[b]Nine samples had zero deviation from both sunflower and safflower specifications.

Step 4. Total deviation is < 2.0%, so the sample is accepted as having the identity indicated on its label.

Example 2. A sample labeled cottonseed oil contained 0.9% myristic, 19.8% palmitic, 0.4% palmitoleic, 2.1% stearic, 16.1% oleic, and 60.7% linoleic acids.

Steps 1-3.

Acid	% in sample	Codex range	Deviation
14:0	0.9	0.5-2.0	
16:0	19.8	17-29	
16:1	0.4	0.5-1.5	0.1
18:0	2.1	1.0-4.0	
18:1	16.1	13-44	
18:2	60.7	33-58	2.7
18:3	--	0.1-2.1	0.1
Total	100.0		2.9

Step 4. Because total deviation from so-called cottonseed oil is > 2.0%, steps 5 and 6 must be applied.

Step 5.

Fat	Total deviation
Arachis	44.7[a]
Cottonseed	2.9
Lard and rendered pork	68.0[a]
Maize	4.5
Mustard seed	85.8[a]
Premier Jus and tallow	72.0[a]
Safflower seed	9.8
Sesame seed	39.2[a]
Soybean	17.7
Sunflower seed	10.2

[a]These could have been eliminated by inspection to reduce the amount of work involved in carrying out step 5.

Step 6. The lowest deviation, 2.9%, was for cottonseed oil. Therefore, the sample should be accepted as having the identity claimed on its label, which was cottonseed.

One problem that may arise, if the proposed identification method is accepted, concerns an oil which may be correctly identified as labeled by application of steps 5 and 6, but which has a lowest total deviation so large that its source may still be in question. Consequently, an upper limit of total deviation should be given also. On the basis of our data, the limit could be set somewhere near 5% total deviation and still ensure that almost any oil would be correctly identified. Hopefully, if the method described here is accepted, the Codex Committee will consider establishing such an upper limit. It is also important to note that this method applies only to crude or refined and bleached oils and cannot be applied to oils that have been modified by hydrogenation, winterization, etc., or to mixtures of oils.

REFERENCES

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