

to make certain adaptations; cholesterol can become esterified with short chain fatty acids, and a more or less normal cholesterol metabolism can be resumed.

c) Essential fatty acids are required for phospholipide synthesis. Phospholipides are probably necessary for transport of cholesterol esters. In the absence of essential fatty acids there may be reduced phospholipide synthesis and therefore interference with cholesterol ester mobilization and transport. It is possible that short chain fatty acids by combining with cholesterol are able to spare essential fatty acids for phospholipide synthesis.

On a 15% cottonseed oil diet the ratio of cholesterol esters to phospholipide in the liver was 1 to 2.6; on a diet deficient in fat and essential fatty acids the ratio was 1 to 1.3; on the essential fatty acid-deficient diet containing hydrogenated coconut oil, the ratio was 1 to 1.9.

d) It is also possible that essential fatty acids are involved in certain enzyme systems which regulate cholesterol metabolism. Tulpule and Williams (27) found that EFA deficiency affected the activity of certain enzyme systems and that one of the sites of action of EFA is the phosphate esterification system, coupled with the oxidation of reduced cytochrome C.

The absolute necessity for essential fatty acids has not been proven in man. It is known that certain human skin diseases are helped by a diet supplemented with essential fatty acids. Diets containing vegetable oils rich in essential fatty acids are now being advised by many investigators for the reduction of elevated serum cholesterol levels. Although it is improbable that a human dietary regime is entirely deficient in essential fatty acids, it is possible that the requirements for EFA are elevated in certain

disease conditions. Certainly there is a need for much more investigation in this promising field.

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## Nutritional Quality of Frying Fats in Commercial Use<sup>1</sup>

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THERE ARE two currents of thought and investigation in the problem of fats and nutrition. The first deals with the general problem of the effect of the amount and type of fat in the diet on nutrition and health. This is the major problem, and the other papers in this symposium are devoted to a discussion of this problem. There is however a second consideration, and that involves changes that may take place in fats during processing and use which might affect the nutritional properties of otherwise adequate fats. These considerations involve principally modifications resulting from hydrogenation and heat treatment.



Daniel Melnick

An earlier report (1) from this and Deuel's laboratory describes the changes which occur in the hydrogenation of fats and presents data in support of the complete biological utilization of fatty acid iso-

mers. Alfin-Slater and associates (2) have recently reported on the nutritive value and safety of hydrogenated fats, following a most comprehensive investigation with rats involving studies of 46 consecutive generations, three longevity studies, carcass analyses, and histopathological examination of the tissues.

The present paper deals with the changes that take place in heated fats and more specifically in fats during frying operations. That commercially fried products represent a significant portion of the foods consumed by the American public is supported by the findings in one industry alone; about one-eighth of all the potatoes raised in this country are consumed in the form of potato chips.

Nutritional and toxicological studies of the fats absorbed by fried foods are unfortunately scanty in number. In Table I are listed conclusions drawn from reports from the most active laboratories in this field. In the studies from Deuel's laboratory (3) attempts were made to simulate commercial frying operations in testing both the frying fat after eight hours of continuous use and the last batch of potato chips fried in

<sup>1</sup>Presented in the symposium on Fats in Nutrition and Health at the 48th Annual Meeting, American Oil Chemists' Society, New Orleans, April 30, 1957. For a more extensive review of the subject the reader is referred to a paper published in *J. Am. Oil Chemists' Soc.*, **34**, 351-356 (1957). In this paper and in another submitted to the same Journal will be found additional experimental details and findings to support conclusions drawn.

TABLE I  
Nutritional and Toxicological Studies on Heated Oils

Laboratory (Year)	Test material	% in diet	Results
Deuel (1951)	a) Margarine oil used 8 hrs. at 400°F. in continuous fryings	11.5	Nontoxic
	b) Potato chips fried in the oil	23	Nontoxic
Crampton (1951-56)	a) Limpid vegetable oils batch-heated at 527°F. under CO <sub>2</sub> for 6-15 hrs.	10-20	Toxic
	b) Cyclized monomeric acids (from linolenic)	10-20	Toxic
	c) Dimers and higher polymers (from linoleic)	10-20	Unabsorbed
Kaunitz (1955-56)	Limpid CSO air-blown at 200°F. for 200-300 hrs.	10-20	Toxic
Kummerow (1956)	Oils air-blown at 392°F. for 24 hrs.		
	a) corn oil; drop of 16 in iodine number	20	Toxic
	b) margarine oil; drop of 9 in iodine number	20	Nontoxic
	c) butter oil; drop of 5 in iodine number	20	Nontoxic

this oil. On the basis of the growth responses of the test animals and values for efficiency of food utilization it was reported that there were no differences whatsoever between the nutritive values of the fresh and heated fats.

Crampton and associates (4-8) were interested in making oils rich in the polyunsaturated fatty acids, particularly those rich in linolenic acid, more stable flavorwise so that they could be incorporated in human dietaries. They subjected oils, such as linseed oil, to thermal polymerization changes by using the conditions noted in Table I. Such oils were toxic. In studies with a variety of vegetable oils Crampton showed that it was the cyclized monomeric acids derived from linolenic acid which imparted toxicity to his oils. The dimers and higher polymers derived from linoleic acid were, according to Crampton, not toxic in the usual sense of the word but were simply unabsorbed; they contributed to the diarrhea noted. Crampton never extended conclusions beyond the scope of his findings. A temperature of 527°F. is about 100°F. above the smoke point of frying oils and so is never encountered in the frying industries.

The test oils employed by the next two groups of workers listed in Table I were also heat-abused in the laboratory under conditions which bear no similarity to practical operations in the frying industries. There is no question that Kaunitz and associates (9-11) obtained a toxic oil when limpid cottonseed oil was air-blown at 200°F. for 200 to 300 hrs. But this is essentially a marked exaggeration of the A.O.M. stability test! We know that limpid cottonseed oil under these conditions becomes unpalatable after 12 to 15 hrs. of heating with the attainment of a peroxide value of 100 m.e./kg.

Despite the fact that Kummerow and coworkers (12) selected the same temperature employed in commercial frying operations, their air-blown oils are no more pertinent to practical operations than those used by Kaunitz and associates. In commercial fryings there is a constant replenishment with fresh oil of the oil absorbed by the food, and frying operations are not conducted with aerated oils but rather with oils under a blanket of an inert gas, the steam generated by the food being cooked. Nevertheless, even under these unrealistic laboratory conditions, unquestionable evidence of toxicity was obtained only with the corn oil; in this case there was a large decrease in iodine value, a drop of 16.

In order clearly to delineate the question of polymer development in frying oils the data shown in

Table II are presented. Cottonseed oil was heated under carbon dioxide until there was a drop of about 5 in iodine value. The same original oil was air-blown at the same temperature to effect a comparable drop in iodine value; this required a heating period of only one-tenth as long. In both polymerized oils there were increases in viscosity. The flavor picture with these oils is worthy of emphasis. The thermally polymerized oil is fairly good in flavor and, when diluted in fresh oil, scores good in flavor. On the other hand, the air-blown oil that has been thermally oxidized is repulsive in flavor, and its objectionable characteristics are detected even when diluted to 1% concentration in the fresh control oil. Thus oils containing thermal polymers lack the safeguard of unacceptable flavor to cause a consumer to reject such

TABLE II  
Evaluation of Oils Heated in the Laboratory

Test	Control CS salad oil (A)	CO <sub>2</sub> blown at 600°F. for 16 hrs. (B)	Air-blown at 600°F. for 1.5 hrs. (C)
Iodine No. (Wijs).....	114.0	109.4	107.7
I No. drop.....	—	4.6	6.3
Nd at 40°C.....	1.4645	1.4661	1.4671
Viscosity, cp. at 40°C.....	29.5	42.7	45.4
Free fatty acids, %.....	0.05	0.80	0.60
Flavor (100%).....	Good	F. good	Repulsive
Flavor 10% in (A).....	—	Good	Very poor
Flavor 1% in (A).....	—	Good	Poor
Free tocopherol, %.....	0.040	0.003	0.008
Peroxide values, m.e./kg.			
Fresh held in dark <sup>a</sup> .....	0.4	2.7	3.9
After 48 hrs. at R. T.....	1.4	9.8	19.0
After 96 hrs. at R. T.....	3.9	18.5	36.5
After 192 hrs. at R. T.....	7.2	27.5	41.0

<sup>a</sup> Oils stored under air in half-filled, loosely capped jars.

oils. Whereas the oxidative polymers *per se* are also free of objectionable flavor, oils containing such polymers are definitely unpalatable and, when held at room temperature, continue to deteriorate at an accelerated rate.

Fatty acid compositions (13) of the oils heated in the laboratory are presented in Table III. Attention is directed to the fact that only in the thermally polymerized oil are there found conjugated dienoic acids. Conjugation is known to be a precursor of polymer formation (14). The absence of conjugated dienoic acids in the thermally oxidized oil is attributed to their disappearance because of ease of oxidation (1).

When a dienoic acid, such as linoleic acid, is heated, migration of the double bonds occurs. This is followed by a reaction under an inert atmosphere between the conjugated dienoic acid and the nonconjugated acid to form a polymer (15). In the course of this reaction there occurs a reduction in double bonds or degree of unsaturation. Degree of unsaturation is measured by iodine value. Thus one has at hand a precise method for determining whether thermal polymers may have formed in an oil used in the frying of foods. Whereas an appreciable drop in iodine value

TABLE III  
Fatty Acid Composition of Oils Heated in the Laboratory

Test	Control CS salad oil	CO <sub>2</sub> blown at 600°F. for 16 hrs.	Air-blown at 600°F. for 1.5 hrs.
Iodine No. (Wijs).....	114.0	109.4	107.7
Conjugated dienoic, %.....	0.00	2.76	0.00
Conjugated trienoic, %.....	0.03	0.00	0.07
Linoleic acid, %.....	52.3	44.1	47.6
Oleic acid, %.....	21.5	27.8	23.9
Saturated acids, %.....	21.8	20.9	24.1

TABLE IV  
Summary of Changes in Iodine Values of Fats Employed in the Commercial Manufacture of Potato Chips

No. of chippers	Type of fat	Iodine value		Laboratory		
		Initial	Observations	A	B	Avg.
9	Corn oil	124.5	Change on use Significance	-1.22 p=0.10	-1.29 p=0.001	-1.26 p=0.01
9	Cottonseed cooking oil	110.0	Change on use Significance	-1.74 p=0.01	-1.51 p=0.001	-1.63 p=0.01
18	Winterized cottonseed oil	112.5	Change on use Significance	-0.88 p=0.02	-0.74 p=0.02	-0.81 p=0.02
36	Above unhydrogenated oils	113.7	Change on use Significance	-1.18 p=0.01	-1.07 p=0.001	-1.12 p=0.001
10	Lightly hydrogenated cottonseed oil	87.3	Change on use Significance	-1.21 p=0.05	-1.00 p=0.10	-1.11 p=0.10
27	Shortenings	67.1	Change on use Significance	-0.57 p=0.01	-0.47 p=0.01	-0.52 p=0.001
16	Oil-and-shortening-blends	92.2	Change on use Significance	+1.64 None	+1.23 None	+1.62 None

(Wijs) justifies further work to determine whether reactions other than polymerization may have been responsible, no decrease in iodine value is proof that no such polymerization changes could have occurred.

It should be mentioned that the cyclized monomeric acids capable of being derived from linolenic acid are not of practical concern. None of the vegetable oils employed in commercial frying operations contain linolenic acid. It is true that limpid soybean oil contains about 8% of this fatty acid, but no frier in this country can use in his operations unhydrogenated soybean oil because of flavor instability. Soybean oil shortenings contain no linolenic acid. Thus, simply stated, the question of thermal polymers in the frying industries is one concerned solely with the possibility of polymers being formed from the linoleic acid in the frying oils.

About this time last year the author was appointed by the National Potato Chip Institute to head a committee to investigate the possibility of thermal polymers being present in the frying oils employed by the potato chip industry. Ora Smith, director of research for the industry, wrote to the chip manufacturers throughout the country asking for their cooperation in a survey of the fats and oils in use. Both fresh and equilibrium oils were requested. The latter oils were those which had been in continuous use for a period of at least four days. These two oils from each Chipper was then sent under code to two laboratories for determinations of the iodine value. As mentioned earlier, if there were no decrease, then one would have to conclude that there were no thermal polymers in the frying oils employed by the industry.

In Table IV is a summary of the changes in iodine value of fats employed in the commercial manufacture of potato chips. Eighty-nine Chippers participated in this survey. Thirty-six of them used limpid unhydrogenated oils. The remainder used either lightly hydrogenated cottonseed oils, shortenings, or oil-and-shortening blends. The initial iodine values are listed as well as the changes in the values by each of the two laboratories. There are also included values showing the statistical significance (16, 17) of the changes noted. When  $p=0.05$ , it is concluded that there are 95 chances out of 100 that the observed change is a real one. Statisticians hold that this probability must be satisfied or no significance can be assigned to the change noted. There is no question that the changes in iodine value obtained in analyzing the three types of unhydrogenated oils and the shortenings are all highly significant from the statistical standpoint. Despite the fact that the lightly hydrogenated cottonseed oils and the oil-and-

shortening blends contain more linoleic acid than the shortenings, no significant changes in iodine value were found; this is attributed to difficulties in properly sampling these oils.

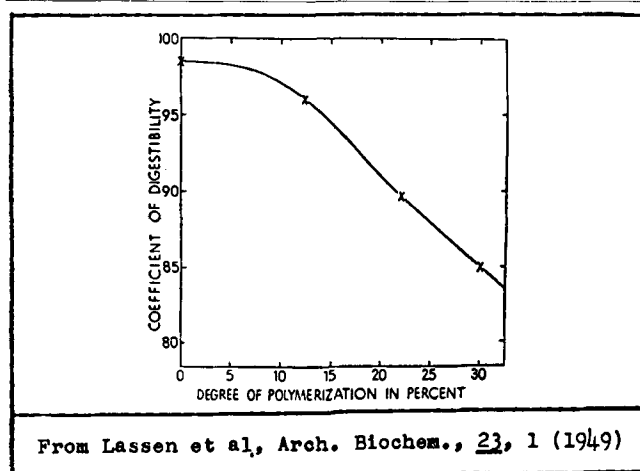


FIG. 1. Decrease in digestibility of a highly unsaturated oil as the degree of polymerization increases; the latter plotted as the percentage drop in iodine value.

In Figure 1 are data plotted by Lassen and associates (18) showing the decrease in digestibility of a highly unsaturated oil as the degree of polymerization increases; polymerization has been plotted as the percentage drop in the iodine value. It will be noted that a drop of about 5% in iodine value is required before there is a measurable decrease in the coefficient of the digestibility of the oil. In studies completed, but as yet not reported, Alfin-Slater (19) has noted that growth, reproduction, lactation, and longevity are not impaired when cottonseed oil is heat-polymerized to effect a drop of 5% in iodine value. Digestibility of the oil is slightly reduced from about 96% to 93%. Hence it must be concluded that any change in digestibility attributable to an iodine value drop of about 1%, found in the present study, is beyond the realm of measurement and is therefore of no nutritional significance. This conclusion is most conservative since it presupposes that the very small drop in iodine value noted in the present survey of frying oils in commercial use may have actually been due to polymer formation. Such however is not the case, as will be shown by the results of subsequent studies.

In Table V are shown the changes in the fatty acid composition (13, 20) of typical fats employed in the commercial manufacture of potato chips. Small increases in the free fatty acid value are noted, but no

TABLE V  
Changes in Fatty Acid Composition of Typical Frying Fats Employed in the Commercial Manufacture of Potato Chips

Chipper No.	Fat tested			Fatty acid composition, %						
	Type	State	Iodine value	Free	Trans	Conjugated		Linoleic	Oleic	Saturated
						Dienoic	Trienoic			
24	Corn oil	Fresh	124.8	0.02	2.3	0.00	0.06	54.7	28.4	12.4
		Heated	122.4	0.20	3.6	0.95	0.08	51.8	29.8	13.0
62	Cottonseed-cooking oil	Fresh	111.6	0.06	2.0	0.00	0.11	50.3	22.6	22.6
		Heated	109.4	0.18	2.5	0.85	0.11	47.3	24.5	22.8
23	Cottonseed-winterized oil	Fresh	114.7	0.04	2.4	0.00	0.00	52.3	22.2	21.1
		Heated	112.5	0.20	2.3	1.00	0.04	50.0	22.4	22.2
28	Lightly hydrogenated cottonseed oil	Fresh	85.6	0.04	26.1	3.04	0.04	20.2	47.6	24.3
		Heated	85.1	0.33	27.4	3.12	0.06	19.4	49.5	23.5
74	Shortening	Fresh	69.1	0.03	37.1	0.57	0.02	7.8	60.0	27.2
		Heated	67.6	0.20	34.3	0.43	0.02	7.1	60.0	28.0
51	Oil-and-shortening blend	Fresh	88.4	0.03	22.1	0.38	0.00	26.3	44.7	24.2
		Heated	87.0	0.11	22.8	0.57	0.02	25.3	44.6	25.1

significant increases in the concentrations of *trans*-acids occur. The small decreases in linoleic acid content reflect the small decreases in iodine value. Of major interest is the finding that there are small but definite increases in the conjugated dienoic acid content of the unhydrogenated oils. In the hydrogenated fats there are present some conjugated dienoic acids at the very start, particularly in the lightly hydrogenated cottonseed oils. These accumulate during the early stages of hydrogenation and then disappear as hydrogenation continues (1). There is nothing objectionable in the conjugated dienoic acids from the nutritional standpoint, as shown in studies conducted in Deuel's laboratory (1). Such acids are not anti-metabolites for linoleic acid but are readily and completely metabolized to carbon dioxide and water. The presence of the conjugated dienoic acids in all the heated fats however is very good proof that one is not concerned here with thermal oxidative polymers since under the conditions leading to the formation of the latter no conjugated dienoic acids survive.

Other analyses conducted on the typical fats employed in the commercial frying operations have furnished more proof that no significant changes in composition occur in the fats. The Solids Content Index (21) of each of these fats, obtained by the highly precise dilatometric procedure, shows no difference between fresh and heated oils; the distribution of solids to liquid in each paired grouping over a wide range of temperature remains remarkably constant, and this would not be the case if an increase in *trans*-acids had occurred.

Flavor considerations and the retention of conjugated dienoic acids in the heated oils have eliminated from consideration thermal oxidative polymers but not the thermal nonoxidative polymers. A very sensitive tool was then sought to measure with precision the minimal formation of the thermal nonoxidative polymers. An adaptation of the dilatometric method proved to be highly satisfactory for this purpose.

When cottonseed salad oil is held at 32°F. for a sufficient period of time, some solid fat slowly crystallizes from the solution as shown in Table VI. When the same oil, but thermally polymerized under carbon dioxide to effect an iodine value drop of about 5, is similarly stored in the cold, there is an immediate precipitation of a large amount of solid fat with a slow progressive increase thereafter. When as little as 10% of the thermally polymerized oil is added to the fresh oil so that the over-all change in iodine value amounts to 0.5, there is an immediate deposition of solid fat in quantity greatly in excess of that

expected from the 10% addition alone of the thermally polymerized oil. Apparently coprecipitation occurs in this system. Measurements of the Solid Content Index in this range of values are reproducible to within 0.2 S.C.I. units so that with this tool the significance of less than 0.1 drop in iodine value can be evaluated.

Using this highly sensitive dilatometric approach, evidence was sought for the presence of thermal polymers in six of the more labile oils employed in the commercial manufacture of potato chips. These oils are characterized as labile since they contain about 50% linoleic acid. They are listed in Table VII in decreasing order of change in iodine value. On the basis of the data presented in the preceding table, one would expect an increase in the Solids Content Index in the heated oils at an earlier time if thermal polymers were present. This however does not occur. Indeed, the reverse is noted. In every paired grouping of oils held at 32°F. there is found with the heated oil a delay or no change in the precipitation of solid fat.

This then is proof that the small decrease in iodine value of about 1% noted in the present survey of potato chip frying oils is not caused by the presence of thermal polymers. Undoubtedly, slight oxidation of linoleic acid occurs, and the volatile degradation products escape through the stack; this is believed to be responsible for the slight lowering of iodine value noted. Further tests on these heated oils have shown that the heated samples have lower interfacial tension values when layered over water. Such evidence points to the presence of emulsifiers developed during the frying operations, which delay crystallization of solid components in the heated oils.

The findings presented in this paper cover only operations in the potato chip industries. Studies similar to this one should be conducted on oils employed

TABLE VI  
Dilatometric Studies on an Oil Heated in the Laboratory and then Held in the Cold

Hours at 32°F.	Solids Content Index at 32°F.		
	Control CS salad oil (A)	CO blown at 600°F. for 16 hrs. (B)	90% (A) + 10% (B)
16	0.0	11.6	3.9
20	0.9	11.8	4.0
24	1.2	12.0	4.3
28	2.1	12.5	4.4
32	2.3	13.0	4.6
40	3.0	13.5	4.8
48	3.7	14.4	4.9
60	3.9	14.4	5.1
72	4.7	14.4	5.2
96	5.5	14.4	5.8

in other frying operations, especially when limpid unhydrogenated oils are used. The frying of potato chips, insofar as heat-damage of the frying oils is concerned, is a relatively mild treatment. There is such a rapid turnover in oil, *i.e.*, constant replenishment with fresh oil to compensate for the oil absorbed by the potato chip, that undesirable by-products do not accumulate in the frying oils. The free fatty acid value in this series of heated oils averaged 0.27%, as shown in Table VIII, an increase of only 0.22%

TABLE VII

Dilatometric Studies on Winterized Cottonseed Oils Employed in the Commercial Manufacturing of Potato Chips

Chipper No.	State of the oil	Iodine value		Solids Content Index at 32°F.					
		Found	Change	Oils held at 32°F. for period of (hrs.)					
				16	20	28	40	60	96
68	Fresh	113.2	—	0.0	0.0	0.6	4.8	5.7	6.9
	Heated	110.7	-2.5	0.0	0.0	0.0	2.5	5.1	5.7
22	Fresh	111.9	—	3.2	3.8	4.5	6.5	7.1	7.5
	Heated	110.2	-1.7	1.8	2.5	4.0	6.5	7.2	7.7
10	Fresh	114.0	—	0.5	2.7	4.2	5.9	6.9	7.1
	Heated	112.5	-1.5	0.0	0.6	1.5	3.3	6.6	6.8
27	Fresh	114.7	—	0.0	0.0	0.0	0.0	2.5	2.8
	Heated	113.6	-1.1	0.0	0.0	0.0	0.0	1.5	3.2
26	Fresh	112.1	—	0.0	0.0	0.1	0.9	4.5	4.9
	Heated	111.2	-0.9	0.0	0.0	0.0	0.5	4.0	4.6
32	Fresh	113.9	—	0.0	0.0	0.0	0.3	1.8	3.5
	Heated	113.6	-0.3	0.0	0.0	0.0	0.3	2.3	3.4

over that of the fresh oil. The total free fatty acid content seldom exceeds 0.5%, and there is very seldom, if ever, the need to discard the frying oil. Indeed in the present survey only three oils out of the 89 tested had a free fatty acid value in excess of 0.5%. It is also worth remembering that the rapid and almost complete volatilization of the water from potato chips during frying is, in essence, continuous steam deodorization and refining of the frying oil throughout its use.

### Summary and Conclusions

Publications on fats heated in the laboratory have been critically reviewed, and many of these have been shown to present findings irrelevant to commercial frying operations. Just about two weeks before the delivery of this paper Rice and associates (22) independently reported before the American Institute of

TABLE VIII

Summary of Changes in Free Fatty Acid Values of Fats Employed in the Commercial Manufacture of Potato Chips

No. of Chippers	Type of fat	Free fatty acid values of the fat, %			
		Fresh	Heated	Change	Standard deviation
9	Corn oil	0.05	0.44	+0.39	±0.34
9	Cottonseed cooking oil	0.04	0.21	+0.17	±0.06
18	Winterized cottonseed oil	0.05	0.24	+0.19	±0.11
10	Lightly hydrogenated cottonseed oil	0.05	0.29	+0.24	±0.23
27	Shortening	0.06	0.24	+0.18	±0.18
16	Oil-and-shortening blend	0.05	0.29	+0.24	±0.15
89	All the above	0.05	0.27	+0.22	±0.17

Nutrition on the nutritive value of frying oils in commercial use. They too were critical of the results of studies of highly unsaturated fats heated under unrealistic, arbitrarily selected, laboratory conditions. Conclusions based upon such laboratory studies should not be extended beyond the scope of the findings reported.

The present report has presented the results of a nation-wide survey of the frying oils in use on a continuing basis in the potato chip industry. Whereas a 1% decrease in iodine value has been found in the heated oils, this change has been shown to have no nutritional significance. The constancy in composition of the frying oils, heated as compared to fresh, and the results of physico-chemical studies support the conclusion that neither thermal polymers nor thermal oxidative polymers are present in the oils employed in the commercial frying operations. No *trans* fatty acids develop during the heating operations.

The changes in linoleic acid content, the essential fatty acid in vegetable oils largely responsible for the important noncaloric functions of fat in the diet (23, 24), are too small to have nutritional significance. This permits the same conclusions to be made in regard to the nutritive value of the heated oils, *i.e.*, of the triglycerides contained therein, as have been drawn from studies conducted on the corresponding fresh oils.

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