

## FLAVOR AND OXIDATIVE STABILITY OF SUNFLOWER OIL

By

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Sunflower oil is characterized as a semidrying oil (1,2) because of its high iodine value of about 138. Nevertheless, its major use has always been as an edible oil. Only two polyunsaturated acids contribute to the high iodine value of sunflower oil; namely, linoleic acid at 70% levels and trace amounts (0.2-0.3%) of linolenic acid. In a review on the composition of 11 commercial varieties of sunflowers, Earle *et al.* (3) concluded that fatty acid composition is not significantly influenced by variety. Craig (2) has shown a wide range in fatty acid composition of oils from individual sunflower seeds in both inbred lines and commercial varieties. Oils low in linoleic acid have been reported by Kinman and Earle (4). Only 46.4% linoleic acid in oil from the Krasnodarets variety was found by Earle *et al.* (3) and Craig (2) observed that some single seeds of the Peredovik variety contained less than 50% linoleic acid. Sunflower oils with a lower content of linoleic acid may have somewhat different keeping qualities and stability characteristics than the sunflower oil reported in this paper.

The potato chip industry continues to increase its production year after year and additional supplies of oil are required. Since U.S. sunflower oil might enter this market, a cooperative test was undertaken by the Red River Valley Potato Processing Laboratory, East Grand Forks, Minnesota; Old Dutch Foods, St. Paul, Minnesota; and the Northern Regional Research Laboratory, Peoria, Illinois, to investigate the suitability of sunflower oil for potato chip frying.

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<sup>2</sup> A laboratory cooperatively operated by the Eastern Utilization Research and Development Division, Agricultural Research Service, U.S. Department of Agriculture; Minnesota Agricultural Experiment Station; North Dakota Agricultural Experiment Station; and the Red River Valley Potato Growers Association.

Examination of oil stability may be directed toward the oil itself, toward food products prepared from the oil, or toward an oil extracted from a food product. We conducted studies in each category and examined four different lots of commercially prepared sunflower seed oils. The data obtained are presented here.

#### METHODS OF EVALUATION

Organoleptic evaluation of sunflower oils was conducted at the Northern Laboratory by methods described by Moser *et al.* (5). Oils used for this study were solvent-extracted and commercially refined during 1968 U.S. production. The oils were deodorized (3 hr at 210° C) in all-glass laboratory-size (4 liter) equipment. Oils were bleached when required with 1% bleaching earth. All antioxidants and sequestering agents were added on the cooling side of deodorization, usually at the 0.02% level. Three lots of sunflower oil were investigated for stability characteristics with almost identical results and the fourth lot was utilized in potato chip frying experiment.

Oils for potato chip frying were entirely commercially processed; i.e., refined, bleached, winterized, and deodorized. The sunflower oil had an IV of 138, a linoleic acid content of 72%, and was brilliantly clear with Lovibond 1.0 yellow and 0.1 red. Antioxidant mixture G-50 (Griffith Laboratories) was added to the oil before use at the recommended rate of 8 oz per 1000 lb of oil. The cottonseed-corn oil mixture (70:30) came from a commercial chip fryer, was prepared to his specifications, and contained the G-50 antioxidant mixture.

Potato chips were processed in the stainless steel, continuous experimental fryer developed at the Potato Processing Laboratory. Two 20-hr (two and one-half 8-hr days) frying runs (1000 lb of potatoes each) were made. The first run utilized a 70:30 cottonseed-corn oil mixture, a standard frying oil for the chip industry, and the second run was made under identical conditions with sunflower oil. Chips from Kennebec potatoes were fried at the rate of 50 lb/hr. Frying conditions were 375° F with continuous circulation of the oil (not filtered) through the heaters and fryer. Temperature drop during frying was 7° to 12° F. Slices, 0.05 inch thick, were immersed in the frying oil for 1-1/2 min and allowed to drain for 1-1/2 min before bagging. Chips (unsalted) and oil were sampled every 2 hr during the runs. Make-up oil was added after each sampling period.

Flavor data on freshly deodorized oil and on 60° C oven-aged oil were provided by a 20-member taste panel. Except for the initial evaluation of fresh oil, all results were obtained by comparison of two samples. Statistically significant differences in flavor scores are noted by the usual signs. Another test to evaluate edible oil stability is the Active

Oxygen Method (AOM) (6). In this test, oils are heated to 97.8° C and air is bubbled through them at a rate of 2.33 ml/min. The amount of oxidation is indicated by the peroxide levels determined after specified times of aeration.

### Oil Evaluation

Table I shows flavor data for sunflower oil treated with several

Table 1. Flavor Evaluation of Sunflower Oil Treated with Commercial Antioxidant Mixtures

Storage Treatment	Taste Panel Flavor Scores				Significance
	Untreated Control	Tenox-2 0.02% <sup>1</sup>	Tenox-6 0.02% <sup>1</sup>	G-50 0.02% <sup>1</sup>	
None	7.7(0.0) <sup>2</sup>	8.0(0.0)	8.2(0.0)	8.1(0.0)	+
4 days at 60°	4.3(10.0)	7.1(9.2)			**
4 days at 60°	4.4(10.5)		6.4(5.6)		**
4 days at 60°	5.6(9.3)			7.1(6.4)	**
8-hr AOM	108	16.3	16.6	23.4	

<sup>1</sup>0.02% active ingredients; Tenox-2 and -6 (Tennessee Eastman) and G-50 (Griffith Laboratories).

<sup>2</sup>Figures in parentheses are peroxide levels at the time of tasting.

commercial antioxidant mixtures. These mixtures may contain up to eight components, including two to three primary antioxidants, plus a metal sequestrant (common to all of the antioxidants used), and a varied combination of carrier-solvents. After 4 days at 60° C, a highly significant improvement in stability is shown for oils containing each mixture over that of the untreated control oil. Peroxide levels that develop upon 4-day storage at 60° C are slightly lower than the control, while the 8-hr AOM peroxide values are markedly lower than the control. Results of investigations conducted with each of the individual components of these antioxidant mixtures show that improved stability results

primarily from metal sequestration rather than from antioxidant synergism (10). Equally good results under the test conditions were obtained by the addition of 0.01% citric acid to the oil. As shown many times for soybean oil (7), trace metal sequestration is one of the most important factors in improving stability of highly unsaturated oils.

The AOM peroxide curves depicted in Figure 1 are evaluations of the same oils for which flavor data was presented in the previous section. Improvement over the control is evident, but there is little difference among the three antioxidant mixtures. Similar tests with these antioxidants in nonbleached oils gave comparable results, except that the nonbleached oils were slightly more stable than the refined-bleached oils.

#### POTATO CHIP EVALUATION

Light color is the prime requirement for a good potato chip and color depends primarily on the quality and condition of the potato. The darkening tendency in the chip is related to the sugar content of the potato. Our fry tests were made at the end of the storage season for northern grown potatoes and the potatoes used were of low quality. However, the specific gravity range and the sugar content of the potatoes were satisfactory. The average of 10 determinations for specific gravity was 1.102 for potatoes used in the cottonseed-corn run and 1.106 for those used in the sunflower oil run. The sugar content of these potatoes (8), given in Table II, comes within the low range accepted for good chip production.

Table II. Sugar Content of Potatoes Used in Chip Frying

Duplicate Sampling of Frying Oil Runs	Sugars in Juice, %		
	Fructose	Glucose	Sucrose
Sunflower	0.03	0.07	0.54
Sunflower	0.03	0.06	1.14
Cottonseed-corn	0.01	0.04	0.58
Cottonseed-corn	0.06	0.12	1.02

There was no significant difference in color between chips fried in sunflower oil and those fried in the cottonseed-corn oil mix. Color of the potato chips are determined by the method of Isleib (9) is given in Figure 2. Color was also measured by a different method at Old Dutch

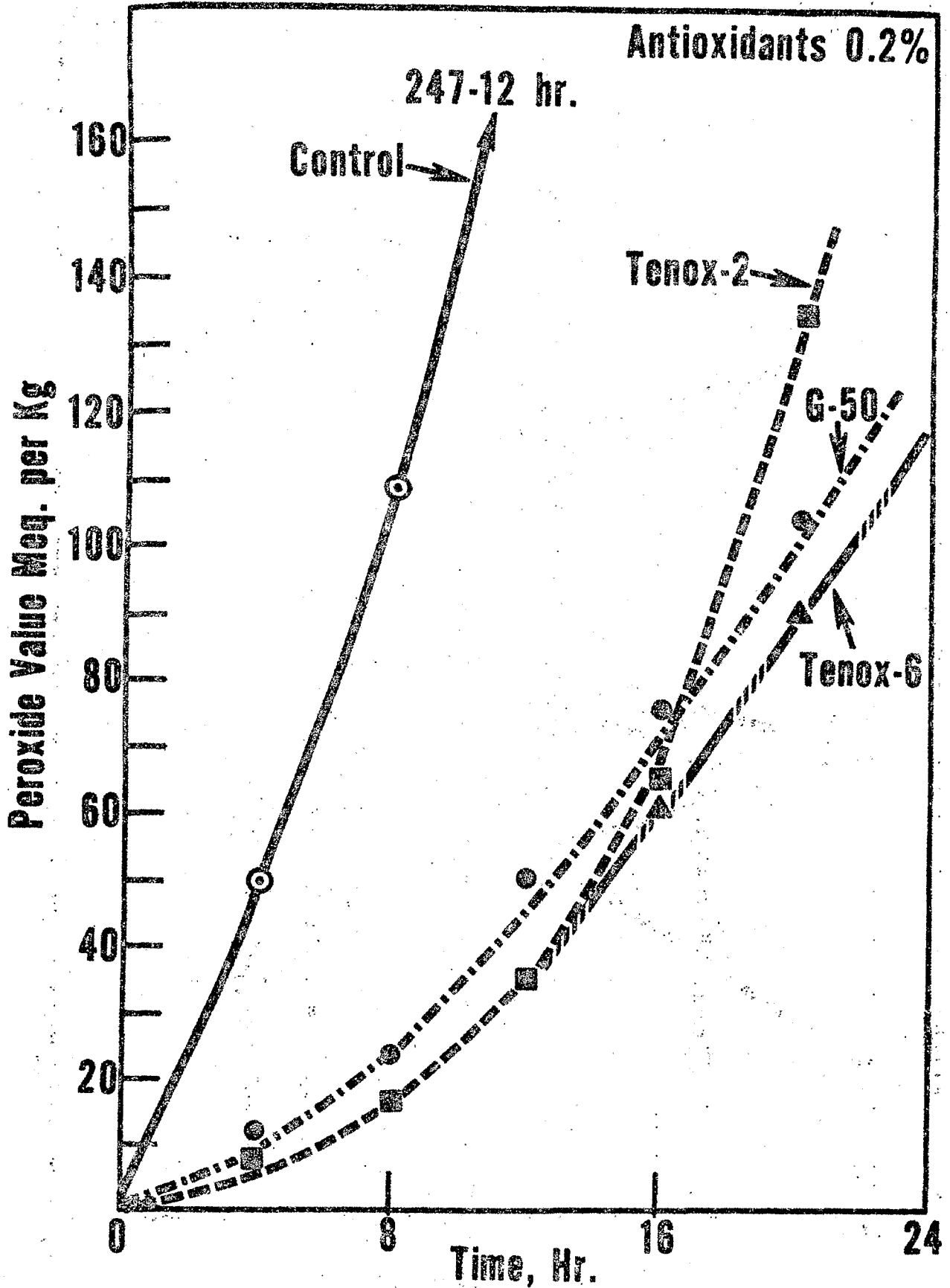


Figure 1. Antioxidant effect on peroxide development in sunflower oil under ACM conditions.

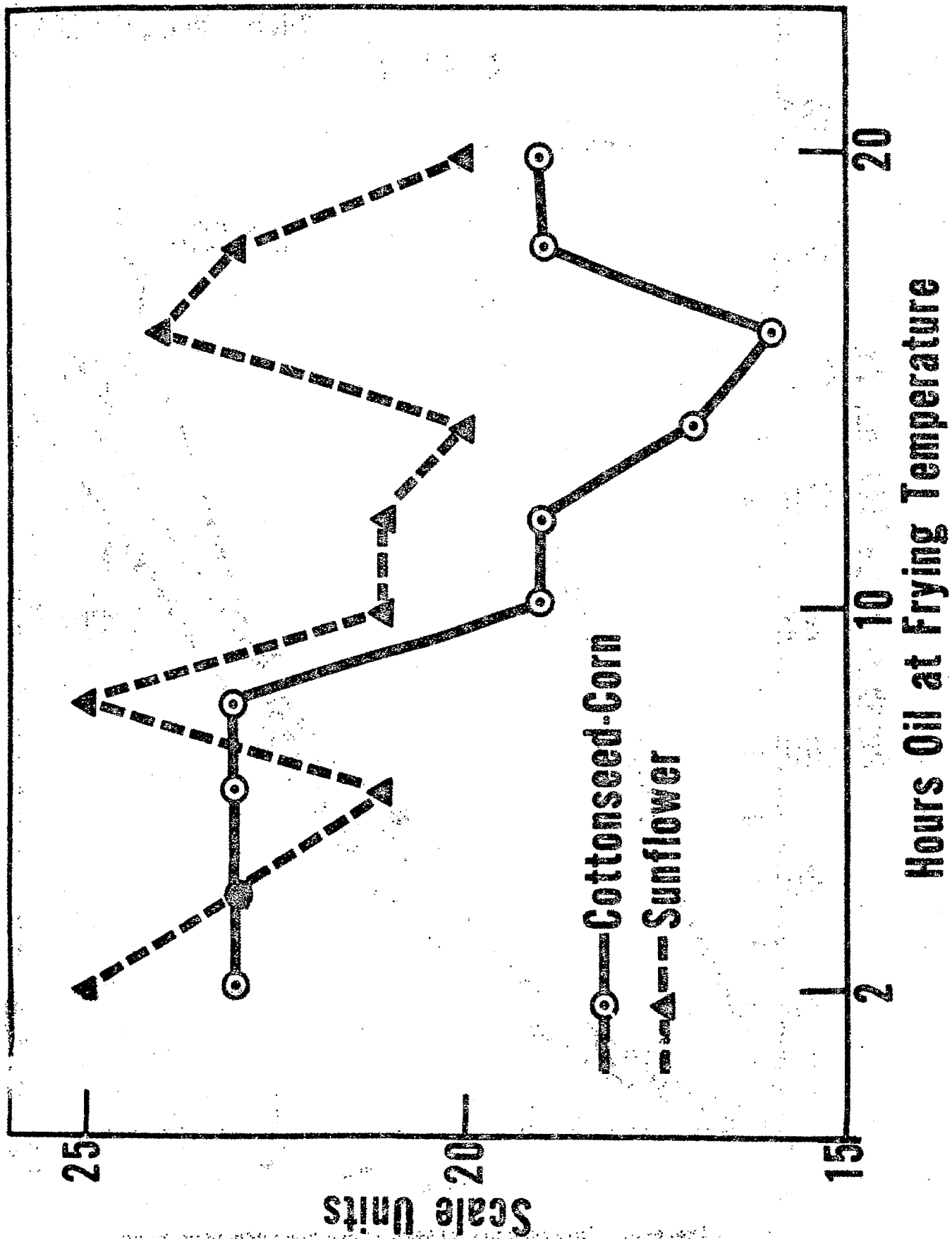


Figure 2. Color of potato chips.

Foods, Inc. with similar results. The slight trend to less color (may not be significant) in chips fried in oils heated as long as 20 hr would indicate that either oil characteristics or oil deterioration within that time has only a minor, if any, effect on chip color.

Flavor evaluations of the potato chips are shown in Table III

Table III. Flavor Evaluation of Potato Chips Stored at Room Temperature

Sample	Frying Time, hr	Flavor Scores		
		Initial	2 weeks	4 weeks
Sunflower	2	7.4 **	7.4 **	6.6 +
Cottonseed-corn	2	5.7	6.5	5.8
Sunflower	8	7.1 +	7.6 +	7.2 **
Cottonseed-corn	8	6.7	7.1	6.1
Sunflower	12	7.2 +	7.4 **	6.3 +
Cottonseed-corn	12	6.8	6.7	5.9
Sunflower	16	7.5 +	7.0 **	6.6 +
Cottonseed-corn	16	6.8	6.0	6.3
Sunflower	20	7.2 +	7.4 +	7.0 +
Cottonseed-corn	20	6.9	7.0	6.5

for room temperature storage and in Table IV for accelerated storage at 60° C. In every evaluation of chips stored at room temperature the taste panel scored those fried in sunflower oil above the ones fried in cottonseed-corn oil. In many comparisons a significant difference was observed in favor of sunflower oil. The results indicate that after 4 weeks of room storage, all chips were acceptable and had satisfactory flavor (Table III).

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Cottonseed-corn	2	5.7	6.5	5.8
Sunflower	8	7.1	7.6	7.2
		+	+	**
Cottonseed-corn	8	6.7	7.1	6.1
Sunflower	12	7.2	7.4	6.3
		+	**	+
Cottonseed-corn	12	6.8	6.7	5.9
Sunflower	16	7.5	7.0	6.6
		+	**	+
Cottonseed-corn	16	6.8	6.0	6.3
Sunflower	20	7.2	7.4	7.0
		+	+	+
Cottonseed-corn	20	6.9	7.0	6.5

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Table IV. Flavor Evaluation of Potato Chips Stored at 60° C

Sample	Frying Time, hr	Flavor Scores		
		Initial	1 week	2 weeks
Sunflower	2	7.4 **	6.7 **	1.3 **
Cottonseed-corn	2	5.7	3.9	6.1
Sunflower	8	7.1 +	6.6 *	1.2 **
Cottonseed-corn	8	6.7	5.6	4.8
Sunflower	16	7.5 +	6.3 **	--
Cottonseed-corn	16	6.8	4.6	1.8 **
Sunflower	20	7.2 +	3.9 **	--
Cottonseed-corn	20	6.9	6.9	4.3

The quality of chips fried in either sunflower or cottonseed-corn oil deteriorated before the second week of storage at temperatures of 60° C (Table IV). When flavor scores of aged chips drop below a value of 5, the majority of taste panel members describe both sunflower and cottonseed-corn oil fried chips as rancid and unacceptable. All chips after storage for 2 weeks at 60° C, regardless of the oil in which they were fried, were rated below 5 by the taste panel with one exception. Chips fried in cottonseed-corn oil after it had been used continuously for 2 hr had a flavor score of 6.1.

A static oil quality condition is never reached in short experimental frying operations because each starts with a large body of fresh oil and because withdrawal of many oil samples necessitates addition of make-up oil at a much higher rate than in commercial practice. This increased dilution with fresh oil is beneficial to chip quality. Previous work at the Northern Laboratory (10) has indicated that the shelf life of experimentally fried potato chips is related to the number of hours the oil has been maintained at frying temperatures.

Oil consumption by the chips was approximately 6% less for the cottonseed-corn run than for the sunflower. The average oil content from 10 samples of chips was 32.8% for the cottonseed-corn run, and 35.0% for the sunflower. Both values are in the desirable range for good chips, and no evidence of excessive oil absorption by the sunflower oil chip is indicated. The operator can control oil absorption in the chip by adjusting frying times and other conditions.

Table V lists chemical data for the two frying oils. Data of this

Table V. Chemical Evaluation of Potato Chip Frying Oils Sunflower (Sun) versus Cottonseed-Corn (C-C, 70:30)

Frying Time, hr	Free Fatty Acids, %		Peroxides, meq/kg		Oil <sup>1</sup> From Chip P.V. meq/kg	
	Sun	C-C	Sun	C-C	Sun	C-C
0	0.02	0.05	9.0	5.0	---	---
2	0.03	0.13	4.0	4.7	6.2	2.4
4	0.06	0.18	7.2	3.2	7.5	2.6
6	0.08	0.22	5.8	3.1	5.5	2.5
8	0.08	0.26	2.1	2.9	6.2	5.0
10	0.10	0.30	5.9	2.5	5.8	5.4
12	0.11	0.32	4.8	1.9	7.9	2.4
14	0.14	0.33	5.2	2.3	6.8	2.4
16	0.15	0.35	4.8	2.3	9.0	1.7
18	0.16	0.43	4.7	3.8	9.2	3.8
20	0.17	0.42	4.1	4.3	5.8	4.3

<sup>1</sup>Oil extracted after chips stored 5 weeks at room temperature (Old Dutch Foods, 1968).

type are routinely collected by commercial fryers in characterizing chip frying oils. Free fatty acid formation in the sunflower oil was at consistently lower levels than in the cottonseed-corn mixture during the 20-hr frying period. Peroxide values of the oils extracted from the chips stored for 5 weeks indicate some autoxidation of the sunflower oil but at a level below which poor flavor responses are indicated.

#### OXIDATIVE EVALUATIONS

Oxidative rancidity in foods results from deterioration of the fat. During the course of fat oxidation some of the fat hydroperoxides will decompose and give rise to typical rancid odors. Among the many products of this decomposition is the hydrocarbon, pentane. One method to measure autoxidation of potato chips is to use gas chromatography to determine the pentane content in the headspace gas of a package. The data in Figure 3 indicate that deterioration of potato chips fried in sunflower oil occurs between the 13th and 14th day of storage at 60° C. This rapid formation of pentane indicates that sunflower oil deterioration progresses rapidly once it starts. Figure 4 shows the pentane headspace analysis for chips fried in cottonseed-corn oil. These chips showed no pentane formation until after 19 days' storage. At 22 days the chips are very rancid and the autoxidized oil had formed considerable pentane.

#### Polymer Formation

Fats and vegetable oils exposed to both air and high temperatures, as in potato chip frying, develop oxidative polymers. Such polymeric materials are undesirable for a number of reasons, including catalysis of further autoxidation of the fat (11). Since these polymeric materials are largely composed of two fatty acid residues polymerized together, they are referred to as dimers. Dimeric materials can be analyzed in heated and oxidized fats by a liquid chromatographic technique (12-14) that separates the dimers and strongly polar materials from the fatty material. Figure 5 depicts results of such a chromatographic separation made on a sample of sunflower oil used in the potato chip frying experiment.

The first and largest peak contains the natural, unoxidized fatty acids; the second peak, the polymeric material; and the third peak, the shorter chain, highly oxidized, and split fatty acid fragments. Figures 6 and 7 plot dimer content versus time of frying for each of the frying oils. Calculations showed that the data up to the 20-hr frying time fit a parabolic curve better than a linear regression curve. Dimer contents for oils beyond the 20-hr frying time would be expected to increase at a constant but unknown rate. Therefore, a dotted curve extends from the highest point of the parabolic curve. To obtain data

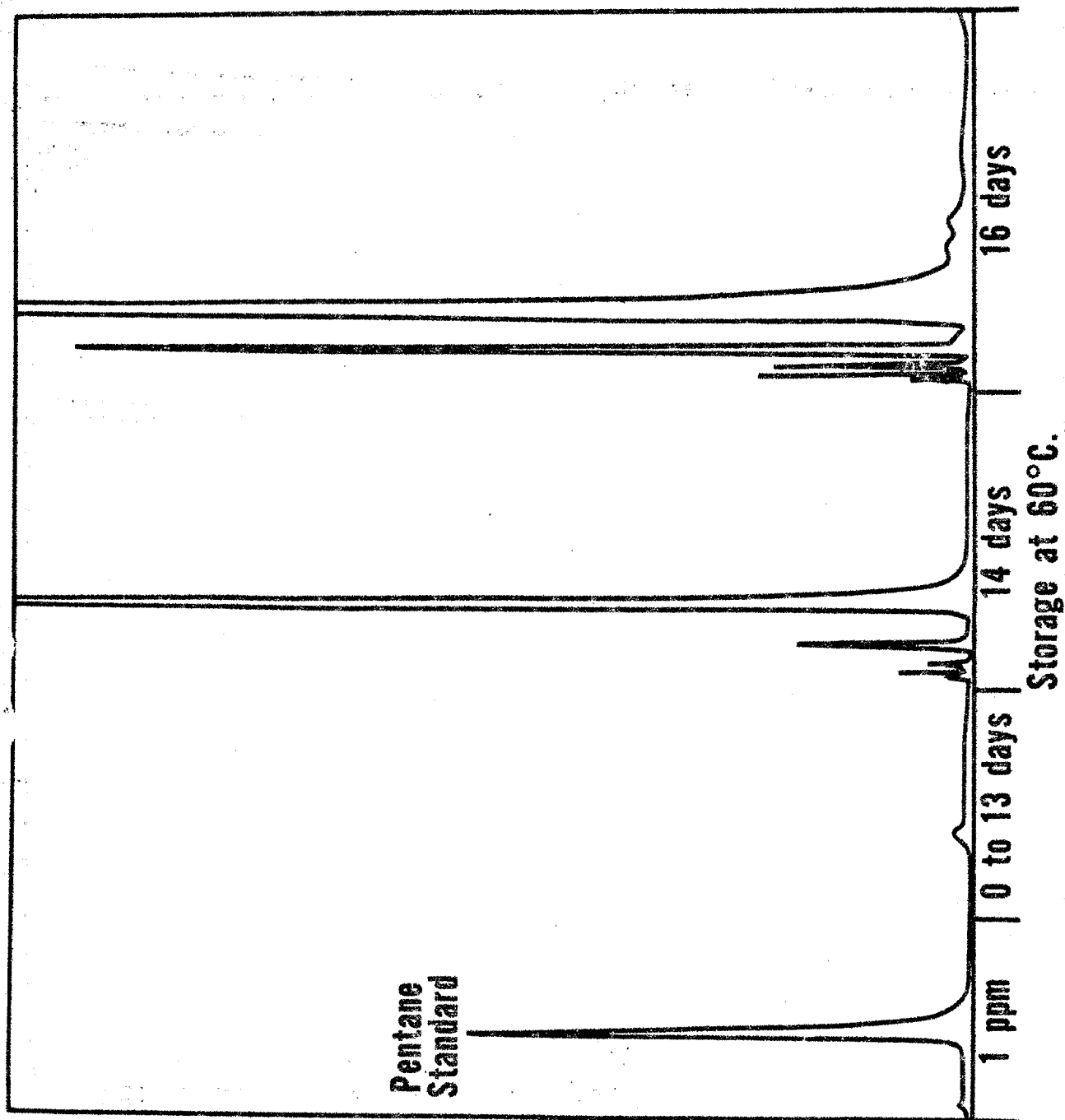


Figure 3. Stability of packaged chips fried in sunflower oil as indicated by pentane accumulation in the headspace gas during 60° C oven storage.

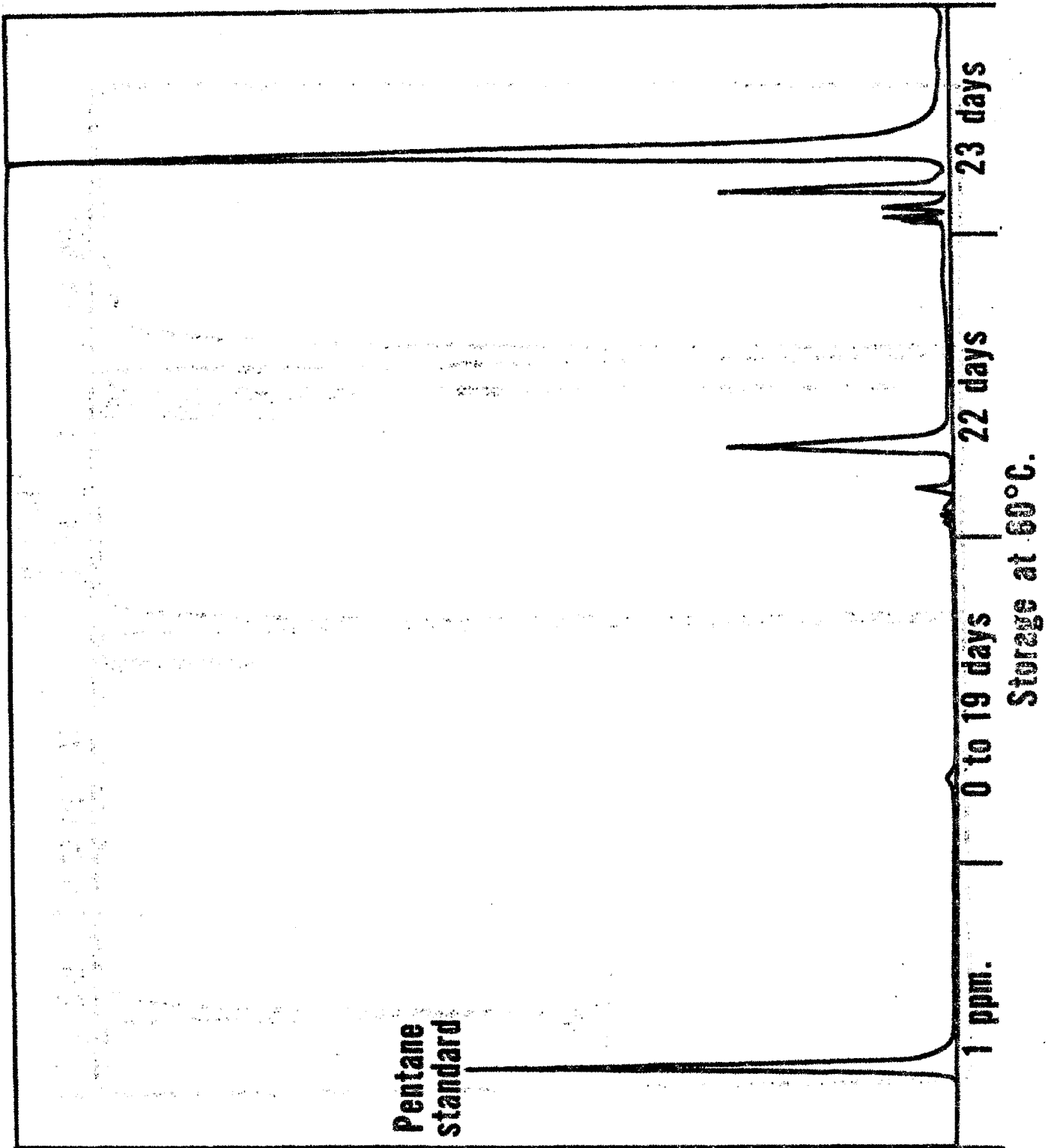


Figure 4. Stability of packaged chips fried in 70:30 cottonseed-corn oil as indicated by pentane accumulation in the headspace gas during 60° C oven storage.

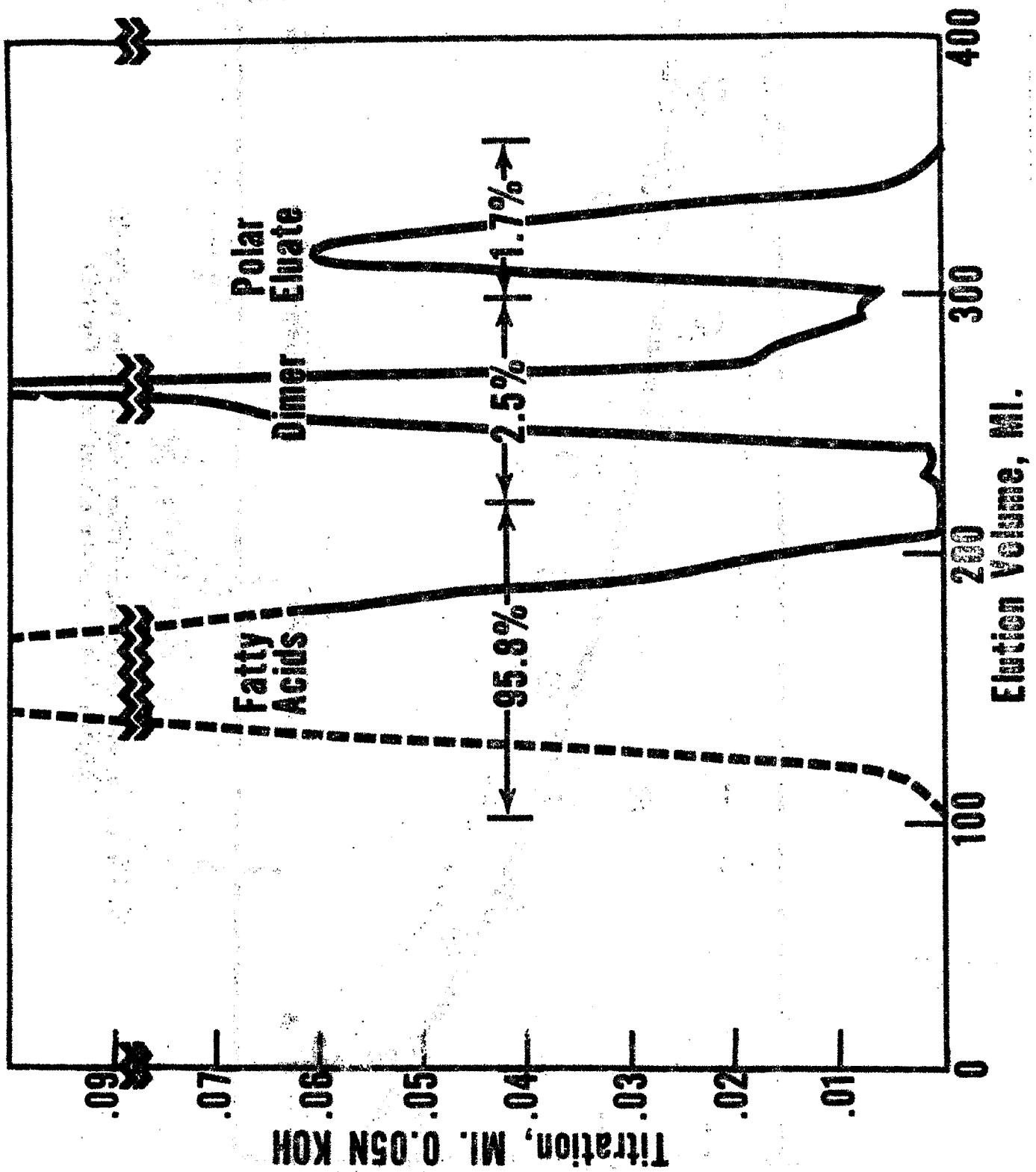


Figure 5. Dimer analysis of sunflower oil after 12 hr of chip frying.

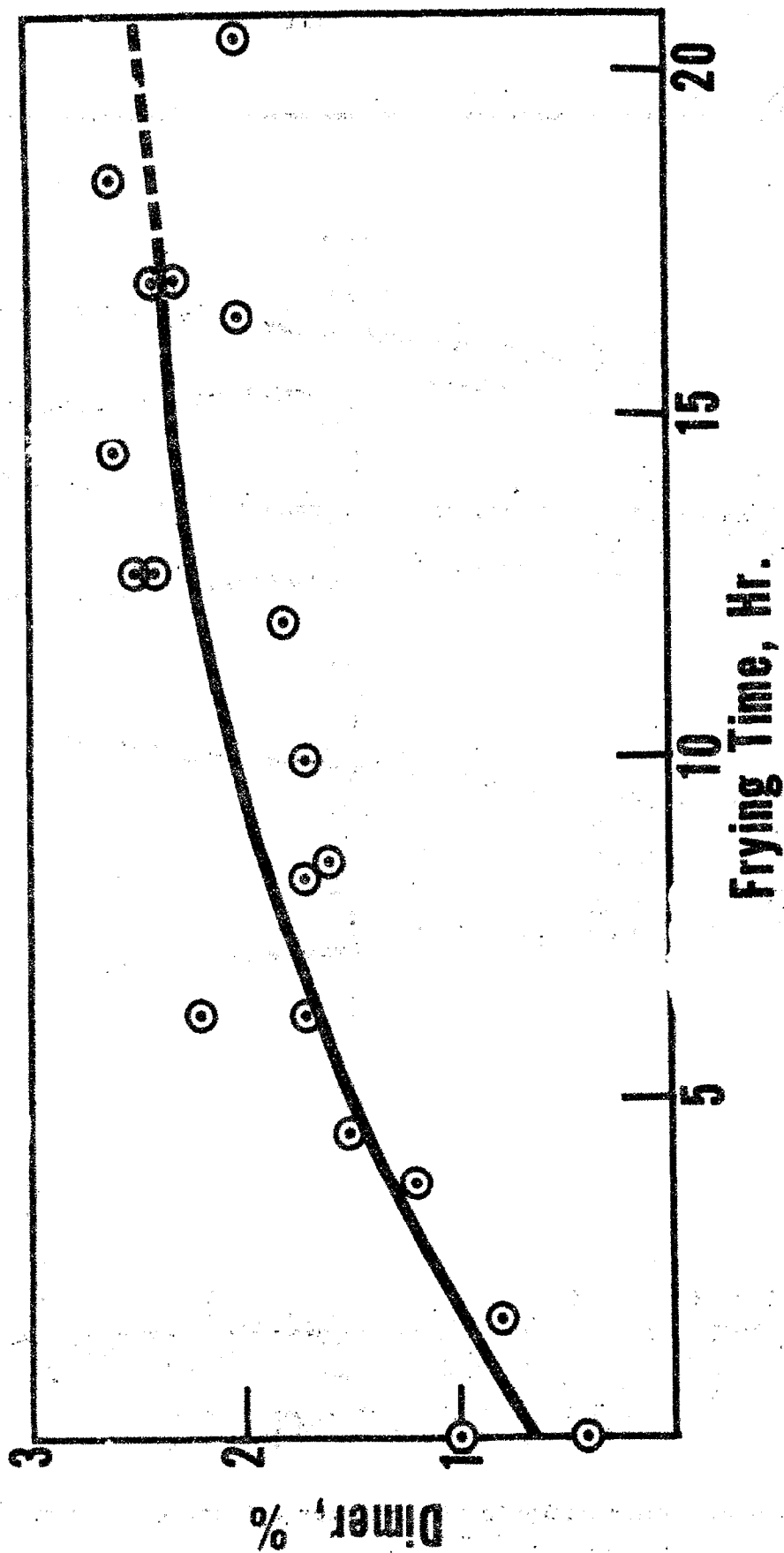


Figure 6. Dimer content of sunflower potato chip frying oil sampled during a 20-hr experimental fry test.

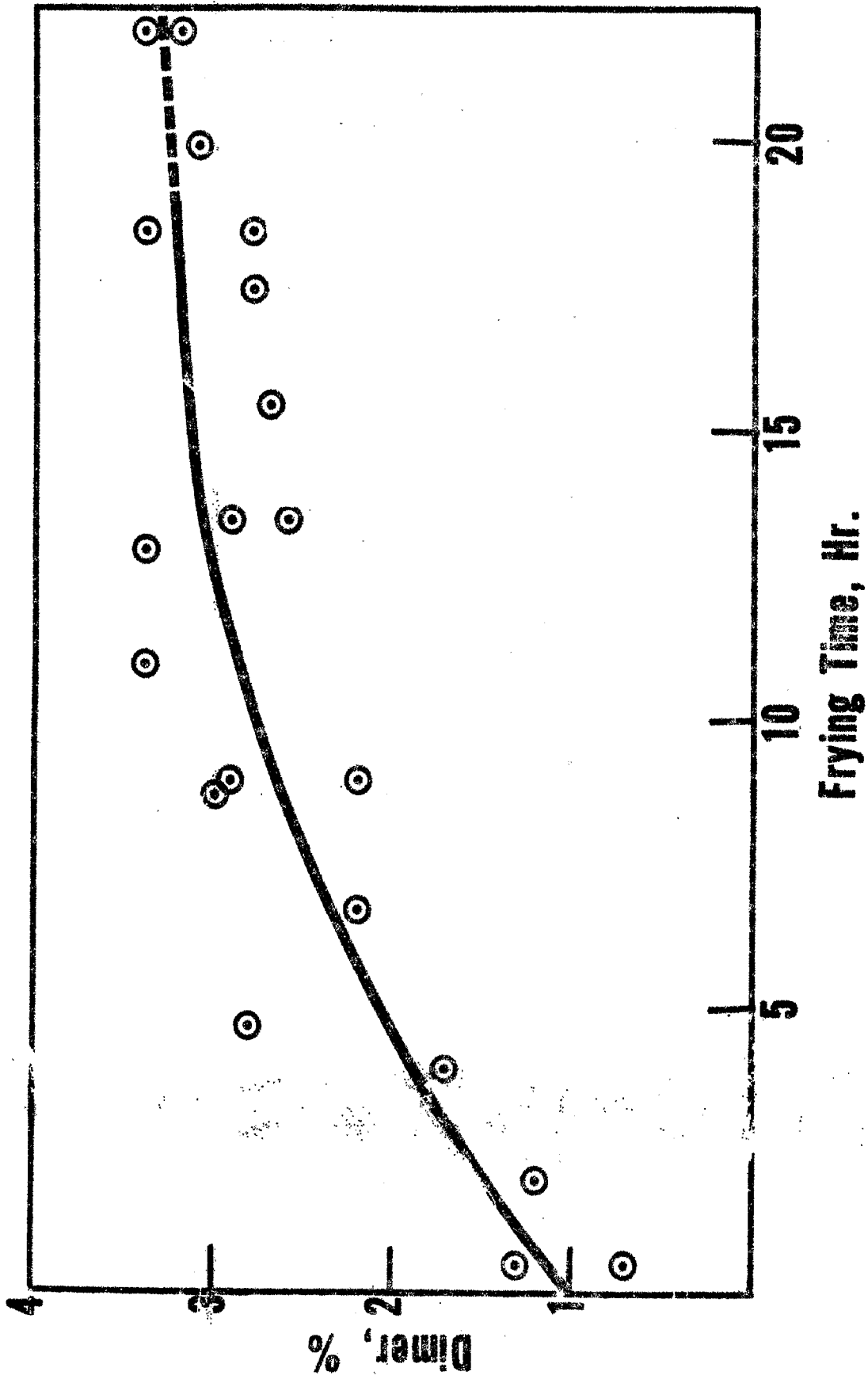


Figure 7. Dimer content of cottonseed-corn (70:30) potato chip frying oil sampled during a 20-hr experimental fry test.





needed to confirm this supposition actual frying would have to be performed for longer than 20 hr. The curve might be changed somewhat if less make-up oil were used than in these experiments.

Figures 6 and 7 indicated that dimer content in the cottonseed-corn oil after frying for 20 hr is 3.3% and in the sunflower frying oil is about 1% lower. This difference of 1% is rather constant over the 20-hr frying period, indicating perhaps that the rates of polymer formation in both oils may be approximately the same.

Table VI presents data on dimers in oils extracted from potato

Table VI. Dimer Analysis on Oils Extracted from Potato Chips

Sample	Frying Time, hr	Oil in Chip, %	Fatty Acids, %	Dimer, %	Polar Eluate, %
Sunflower	2	32.6	98.2	1.0	0.8
	12	33.7	96.2	2.4	1.2
	20	35.6	96.8	1.9	1.1
Cottonseed-corn (70:30)	2	34.2	97.1	2.0	0.8
	12	32.0	94.6	3.2	2.0
	20	32.8	94.5	3.0	2.0

chips. These chips were not aged, and data for the extracted oil compare favorably with that presented for the corresponding frying oils. No evidence was obtained that polymers tend to concentrate in the chip. The amount of polar materials (polar eluate) increased in proportion to the amount of dimer formation, since they are byproducts of the same hydroperoxide decomposition reaction.

#### Oxygenated Acids

The importance of the oxygenated acids to the quality and stability of sunflower oil remains to be established as does the source of these acids. The presence of oxygenated acids in sunflower oil is evidenced by the hydrogen bromide reaction (Durbetaki titration), diene absorption,

infrared spectrophotometry, and thin-layer chromatography (4, 15, 16). The reported content of hydrogen bromide-absorbing acids varies from 0 to more than 4% (17). However, oils from fresh seeds reportedly contain little, if any, of these oxygenated acids (18). Morris *et al.* (15), as well as Kinman and Earle (4), suggest the presence of dimorphecolic acid or its isomers and an epoxy acid. Since then, Miolajczak *et al.* (18) isolated from stored sunflower seed and reported the structure of a saturated and an unsaturated epoxy acid and two conjugated dienolic acids. Since the acids are optically active, it was concluded that they were derived from biological (enzymatic) processes and not from autoxidation during storage.

Hydrogen bromide-reactive materials in potato chip frying oils are low as expected (Table VII). The titration at 0° C may represent only

Table VII. Oxygenated Acids

Sample	Frying Time, hr	HBr Equivalent	
		0°, %	55°, %
Sunflower	2	0.1	0.3
Sunflower	10	0.2	0.8
Sunflower	20	0.2	0.9
Cottonseed-corn	20	0.2	1.1

epoxy acids (calc. as epoxyoleic), whereas the titration at 55° C represents the total of epoxy acids, dienols, and perhaps other structures.

#### TRACE METAL CONTENT

Sunflower oil is sensitive to trace contamination with heavy metals (10). Trace amounts of metals, especially copper, are active oxidation catalysts for unsaturated fats. In Table VIII are reported the copper contents of several sunflower oils as determined by neutron activation analysis. The copper varies considerably; this variation may be real or it may result from errors in either sampling or analysis at these low levels. Analyses were performed by direct irradiation of the oils; no chemical separation, fractionation, or ashing of the oils were done. Interference of an unknown irradiation peak prevented analysis of copper in the 2-hr chip oil. The 10- and 20-hr chip oils were reported to be high in potassium. In Table IX are recorded the results of trace element

Table VIII. Copper Analysis of Various Sunflower Oils (Neutron Activation Analysis)

Sample	Cu, ppm
American salad oil	0.111
German salad oil	0.00535
American refined, undeodorized	0.00951
American refined and bleached	0.0107
Chip frying oil, 2 hr	N.D. <sup>1</sup>
Chip frying oil, 10 hr	0.0585
Chip frying oil, 20 hr	0.0450

<sup>1</sup>Not detected.

Table IX. Trace Element Analysis of a Refined and Bleached Sunflower Oil (Emission Spectra)

Element	Conc., ppm
Aluminum	<0.06
Barium	0.014
Boron	0.03
Calcium	<2.8
Chromium	<0.02
Copper	0.085
Iron	1.33
Magnesium	<1.01
Manganese	0.01
Phosphorus	<1.4
Potassium	<5.6
Sodium	1.71
Strontium	<0.006
Zinc	0.062

determination performed on the ash of another sample of edible sunflower oil by spectroscopic emission analysis.

Since trace metal analysis at the levels reported in Tables VIII and IX are exceedingly difficult to perform, the results are offered primarily to indicate levels rather than to denote absolute figures. We have recently reported the copper levels in soybean salad oil at 0.030 to 0.100 ppm (19).

#### SUMMARY

Evaluation studies on a commercially prepared sunflower salad oil showed that commercial antioxidant mixtures containing metal sequestrants imparted additional flavor and oxidative stability.

A 20-hr fry test comparing sunflower oil to a cottonseed-corn oil mixture demonstrated that sunflower oil potato chips stored at room temperature for 4 weeks received the higher flavor scores at each evaluation. Measurements by two independent laboratories showed that color of chips fried in the sunflower oil was equal to that of those fried in the cottonseed-corn oil mixture. Data on pentane formation indicated that once the induction period is passed, both sunflower and cottonseed-corn oil chips deteriorated rapidly. Oxidative dimer and free fatty acid formation during the 20-hr chip frying test was less in the sunflower oil than in the cottonseed-corn oil mixture. Since sunflower oil is sensitive to trace metal contamination, data on trace metal contents of oils are presented.

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The mention of firm names or trade products does not imply that they are endorsed or recommended by the Department of Agriculture over other firms or similar products not mentioned.

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