AOCS Collaborative Study on Sensory and Volatile Compound Analyses of Vegetable Oils

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ABSTRACT: An AOCS collaborative study was conducted to determine the effectiveness of sensory analysis and gas chromatographic analyses of volatile compounds in measuring vegetable oils for levels of oxidation that ranged from none to high. Sixteen laboratories from industry, government, and academia in Canada and the United States participated in the study to evaluate the flavor quality and oxidative stability of aged soybean, corn, sunflower, and canola (low-erucic acid rapeseed) oils. Analytical methods included sensory analyses with both flavor intensity and flavor quality scales and gas-chromatographic volatiles by direct injection, static headspace, and dynamic headspace (purge and trap) techniques. Sensory and volatile compound data were used to rank each of the oils at four levels of oxidation—none, low, moderate, and high. For soybean, canola, and sunflower oils, 85–90% of laboratories correctly ranked the oils by either analysis. For corn oil, only 60% of the laboratories ranked the samples according to the correct levels of oxidation by either analysis. Variance component estimates for flavor scores showed that the variation between sensory panelists within laboratories was lowest for the unaged oils. As storage time increased, the variance also increased, indicating that differences among panelists were greater for more highly oxidized oils. Between-laboratory variance of sensory panel scores was significantly lower than within-laboratory variance.

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KEY WORDS: Canola oil, corn oil, flavor, gas chromatography, low-erucic acid rapeseed oil, oxidation, sensory evaluation, soybean oil, sunflower oil, volatile compound.

The AOCS Flavor Quality and Stability Committee has sponsored several collaborative studies since its inception in 1967, when it was initially known as the Flavor Nomenclature and Standards Committee. The current committee name was adopted in the late 1980s to reflect the committee's interest in stability measurements in addition to flavor analysis. The first collaborative studies focused on flavor characteristics of oils, but results showed little agreement among collaborators in identifying flavors from pure compounds (1). Subsequent collaborative studies by the committee were designed to include both sensory and gas-chromatographic (GC) volatiles analyses to measure the extent of oxidation. The GC analyses, used in later collaborative studies, evolved from the work of researchers who developed rapid methods for measuring volatile compounds. In the late 1960s, Evans *et al.* (2) presented a rapid method to analyze hydrocarbons, such as pentane, to monitor the oxidation of oils. This involved placing the oil in the heated inlet liner of the gas chromatograph to generate the volatile compounds. By the early 1970s, this simplified method for GC volatiles analysis—direct injection—was perfected, so laboratories could conduct volatile compound analysis easily without time-consuming distillation procedures to collect compounds (3,4). The AOCS collaborative study in 1979 utilized this direct injection technique to measure volatile compounds and to correlate them with sensory analysis on a qualitative-type flavor rating scale for soybean, corn, and hydrogenated soybean oils that were photooxidized to three levels of deterioration (1). Results showed correlation coefficients for the oils that ranged from −0.76 to −0.90. During the next ten years, the methods to measure volatile compounds by GC advanced to include headspace methods, both static and dynamic (purge and trap) (5–7). Also, two sensory scales, one based on overall intensity of flavors and the other on overall flavor quality, were modified for oils as an alternative to the qualitative-type flavor grading scale based on flavor descriptors used in previous collaborative studies (1,8). A collaborative study was conducted in 1989 to determine the validity of the new GC volatiles methods and revised scoring scales. The objective of this study was to determine the effectiveness of these new and modified sensory analyses and GC analyses of volatile compounds in measuring levels of oxidation in four vegetable oils. Oils were oxidized in the dark rather than photooxidized as in the 1979 test. The objectives of this paper are to report the data from the study to show the capability of the collaborators to correctly distinguish the levels of oxidation with these methods; to show relationships between data from sensory and GC volatiles methods; and to report within-laboratory and between-laboratory variance in flavor score data.

METHODS AND MATERIALS

Sixteen laboratories from industry, government, and academia in Canada and the United States evaluated sixteen sam-

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ples of fresh and oxidized vegetable oils for flavor by sensory methodology and for oxidative stability by GC volatile compounds. Fourteen laboratories participated in the sensory panel analysis, and eight groups evaluated the oils for volatile compounds by three capillary GC methods.

Samples of commercially processed citric acid-treated soybean, sunflower, canola (low-erucic acid rapeseed), and corn oils were aged under Schaal oven conditions at 60°C to reach three levels of oxidation—low, moderate, and high—in addition to the unaged (0 time) samples (6). To determine the stages of oxidation, the oils were monitored during storage by periodic peroxide value (PV) analysis with the AOCS titration method Cd 8-53 (8). The oils were aged to reach specific levels of oxidation, rather than being aged for the same number of days.

Fresh and oxidized oils were packaged in 250-mL amber glass, narrow-mouthed bottles. After the headspace was sparged with nitrogen, the bottles were capped with screw-type lids. Oils were shipped to collaborators in dry ice. Each laboratory received coded samples of each oil with instructions to evaluate the products for sensory and/or GC volatiles analysis.

Sensory analysis. The oils were analyzed for intensities of individual flavor descriptors and for an overall rating based either on overall flavor intensity or overall flavor quality. Collaborators were given the choice of using either of two AOCS flavor evaluation methods—intensity scale or quality scale (Cg 2-83; Ref. 8) (Figs. 1 and 2). The intensity scale had been developed for rating the overall flavor intensity of oils that are processed to have as little flavor as possible, such as soybean, canola, sunflower, or cottonseed oil. On the other hand, the quality scale was developed to rate the overall flavor quality of oils, such as olive, peanut, or corn, that have distinct, characteristic, desirable flavors; however, all oils can be rated on the quality scale. For rating soybean, sunflower, and canola oils, six of the laboratories chose the intensity scale, and eight decided to use the quality scale. All groups used the quality scale to rate the corn oil. Testers were also instructed to list all flavors detected in each oil and to rate the intensity of each flavor as none, weak, moderate, or strong (Figs. 1 and 2). These description intensity levels were later converted during data tabulation to numerical scores on a scale of 0 to 3 with none = 0, weak = 1, moderate = 2, and strong = 3.

AOCS FLAVOR INTENSITY EVALUATION

Name______________ Date______________

Directions: Take 5 to 10 mL of warm oil into the mouth; pull air through the oil and exhale through the nose. Overall Intensity Scores: rate samples on 10–1 scale. Descriptions: identify flavors and rate as weak (W), moderate (M), or strong (S).

SCALE		Sample Numbers			
10	Bland				
9	Trace				
8	Faint				
7	Slight				
6	Mild				
5	Moderate				
4	Definite				
3	Strong				
2	Very Strong				
1	Extreme				

DESCRIPTIONS	DESCRIPTION INTENSITY Sample Numbers			
Nutty				
Buttery				
Corny				
Beany				
Hydrogenated				
Burnt				
Weedy				
Grassy				
Rubbery				
Melon				
Rancid				
Painty				
Fishy				
Other __________				
Other __________				

FIG. 1. Scoresheet for overall flavor intensity scale and individual flavor description intensity.

AOCS FLAVOR QUALITY EVALUATION

Name______________ Date______________

Directions: Take 5 to 10 mL of warm oil into the mouth; pull air through the oil and exhale through the nose. Overall Intensity Scores: rate samples on 10-point scale; identify flavors and rate as weak (W), moderate (M), or strong (S).

QUALITY SCALE		OVERALL FLAVOR QUALITY SCORES Sample Numbers			
10	Excellent				
9	Good				
8					
7	Fair				
6					
5	Poor				
4					
3	Very Poor				
2					
1					

DESCRIPTIONS	DESCRIPTION INTENSITY Sample Numbers			
Nutty				
Buttery				
Corny				
Beany				
Hydrogenatd				
Burnt				
Weedy				
Grassy				
Rubbery				
Melon				
Rancid				
Painty				
Fishy				
Other __________				
Other __________				

FIG. 2. Scoresheet for overall flavor quality scale and individual flavor description intensity.

GC volatiles analysis. Collaborators chose one of three GC volatiles methods—direct injection, static headspace, or dynamic headspace (purge and trap). Because GC equipment and standard conditions routinely used by each laboratory varied so widely, the collaborators agreed to use their own GC methods. Table 1 lists the conditions for the three GC methods used by the collaborators. Only those collaborators that provided conditions are listed by alphabetic code for specific GC conditions. Collaborators were asked to provide data

TABLE 1
Conditions for Gas-Chromatographic Volatiles Methods[a]

Conditions	Direct injection	Static headspace	Dynamic headspace
Purge (temperature/time)			125°C/15 min (O) 100°C/20 min (M) 150°C/5 min (H) 175°C/25 min (B) 190°C (C)
Trap (temperature/time)	120°C/10 min (P) 200°C (K)	120°C/30 min (N)	25°C/5–15 min
Desorb temperature			200°C (M) 250°C/5 min (H) (B)
Injector temperature	200°C	200°C	190°C (C) 250°C (B) (H)
Initial column temperature/hold time	35°C/10 min	−50°C/5 min	40°C/15 min
Temperature program rate	4 or 5°C/min	5°C/min	4 or 5°C/min
Final column temperature	220°C	250°C	230°C
Detector temperature	250°C	250°C	250°C

[a] Letters in parentheses indicate codes for those laboratories providing gas-chromatographic conditions.

on total volatiles and on thirteen individual volatile compounds: pentane, propanal, 2-propenal, pentanal, hexanal, 2-heptenal, 2,4-heptadienal, 2-octenal, nonanal, 2-nonenal, 2-decenal, pentylfuran, and 2,4-decadienal. However, most groups did not report all volatiles because of lack of peak identification by either mass spectrometry or by known compound retention time. Only data for total volatiles, pentane, and hexanal were provided by all chromatographers. Because only one laboratory was able to provide data as ppm, all groups were asked to provide volatile compound data as integrator counts.

Statistical analyses. Sensory data were analyzed by two-way analysis of variance, and all data were analyzed for both Pearson product moment correlation coefficients and rank order correlation coefficients (9). Significant differences in data are expressed as the $P \leq 0.05$ level unless indicated otherwise.

RESULTS AND DISCUSSION

PV. The PV data for the oxidized oils showed three levels of oxidation for each oil in addition to the unaged samples (Table 2). Published data have reported ranges in PV for oxidized vegetable oils, including soy, sunflower, and canola, to be 3–5 for low oxidation, 10–12 for moderate oxidation, and 16–18 for high oxidation (6). The oils in this test were oxidized to PV ranges of 3.1–4.6 for low level of oxidation, 10.4–11.9 for moderate, and 17.2–17.9 for high level of oxidation. Each oil type required a different amount of time (days at 60°C) to reach these PV amounts; therefore, attempts to compare relative stability between oil types would not be valid.

Overall flavor intensity or flavor quality scoring. The objective of the sensory analyses was to check the panels' capability of determining the correct levels of oxidation of each oil type. To help fulfill this objective, the committee asked panelists to report overall flavor scores in addition to individual intensity scores for each flavor description because rating oils for an overall flavor intensity or quality score is a standard practice in the oil industry in North America (1,6,8). The Institute of Food Technologists lists both quantitative descriptive analysis (intensity rating scales) and quality rating scales as appropriate for correlating sensory analysis with chemical

TABLE 2
Peroxide Values of Oils at None, Low, Moderate, and High Levels of Oxidation

	Level of oxidation			
Oil type	None	Low	Moderate	High
Soybean	0	4.6	10.4	17.3
Sunflower	0	3.3	11.2	17.2
Canola	0	4.1	11.0	17.2
Corn	0	3.1	11.9	17.9

TABLE 3
Flavor Scores, Standard Deviations, and Ranked Oxidation Levels for Soybean Oils

	Oxidation levels							
Lab code	None	Rank	Low	Rank	Moderate	Rank	High	Rank
Quality scale								
B	7.5 ± 1.3	1	6.8 ± 0.5	2	5.8 ± 1.3	3	4.0 ± 0.8	4
C	6.6 ± 0.5	2	7.0 ± 0.9	1	4.4 ± 1.4	3	4.0 ± 1.2	4
D	8.0 ± 0.8	1	5.7 ± 1.1	2	4.4 ± 1.0	3	3.7 ± 1.0	4
E	7.9 ± 1.2	1	4.3 ± 1.1	2	3.0 ± 1.0	3	2.4 ± 0.8	4
G	7.3 ± 0.6	1	5.8 ± 1.1	2	4.2 ± 1.2	3	3.0 ± 1.4	4
H	6.3 ± 1.3	1	5.9 ± 1.1	2	4.4 ± 0.8	3	3.6 ± 1.0	4
L	8.9 ± 0.7	1	5.3 ± 1.8	3.5	5.6 ± 1.6	2	5.3 ± 1.5	3.5
M	8.3 ± 0.5	1	7.3 ± 1.0	2	6.4 ± 1.1	3	5.1 ± 0.9	4
Average[a]	7.6a	1.1	6.0b	2.1	4.8c	2.9	3.9d	3.9
Intensity scale								
A	8.5 ± 1.0	1	6.8 ± 1.2	2	5.5 ± 1.3	3	3.9 ± 1.9	4
F	7.9 ± 0.7	1	6.3 ± 1.8	2	4.4 ± 1.6	4	4.6 ± 1.5	3
I	8.6 ± 1.0	1	7.6 ± 1.4	2	5.6 ± 1.3	3	3.7 ± 1.0	4
J	8.8 ± 1.6	1	7.7 ± 1.7	2	5.8 ± 1.7	3	4.4 ± 1.5	4
N	8.2 ± 0.4	1	6.9 ± 1.0	2	4.9 ± 1.0	3	3.8 ± 1.4	4
O	7.9 ± 0.8	1	6.3 ± 0.9	2	4.9 ± 1.6	3	4.3 ± 2.0	4
Average[a]	8.3a	1.0	6.9b	2.0	5.2c	3.2	4.1c	3.8
Both scales average[a]	7.9a	1.1	6.5b	2.0	4.9c	3.0	3.9d	3.9

[a]Scores within each row with letters in common are not significantly different ($P \geq 0.05$).

or physical measurements (10). Panelists were instructed not to use the quality scale as a hedonics scale (like- and dislike-type ratings), but rather to rate the quality of the oils based on their own panel's standards for excellent to poor oil quality.

The mean flavor scores for the oils, rated on either of the two scales, ranged from approximately 8.0 for unaged soy, sunflower, and canola oils to 3.0–4.0 for highly oxidized oils (Tables 3–5). Other researchers have also reported the same

TABLE 4
Flavor Scores, Standard Deviations, and Ranked Oxidation Levels for Sunflower Oils

	Oxidation levels							
Lab code	None	Rank	Low	Rank	Moderate	Rank	High	Rank
Quality scale								
B	7.8 ± 1.5	1	7.3 ± 1.3	2	5.3 ± 1.5	4	5.4 ± 1.7	3
C	7.5 ± 0.8	1	6.1 ± 1.4	2	6.0 ± 1.0	3	5.5 ± 1.2	4
D	7.7 ± 0.8	1	6.6 ± 1.0	2	5.9 ± 0.7	3	5.3 ± 1.3	4
E	8.3 ± 1.2	1	5.5 ± 2.1	2	5.1 ± 1.4	3	3.8 ± 1.3	4
G	7.5 ± 0.9	1	4.7 ± 1.0	2	4.4 ± 1.2	3	3.1 ± 1.5	4
H	6.6 ± 0.8	1	5.8 ± 0.8	2	4.7 ± 0.8	3	3.7 ± 0.8	4
L	7.8 ± 1.0	1	6.5 ± 1.4	2	6.4 ± 0.5	3	5.1 ± 1.4	4
M	7.1 ± 0.4	1	6.0 ± 1.0	2	5.9 ± 0.7	3	5.1 ± 0.9	4
Average[a]	7.5a	1.0	6.1b	2.0	5.5c	3.1	4.8d	3.9
Intensity scale								
A	8.5 ± 0.7	1	6.5 ± 2.0	2	5.5 ± 1.8	3	4.6 ± 1.6	4
F	7.5 ± 0.2	1	6.4 ± 1.8	2	6.1 ± 1.5	3	4.8 ± 0.9	4
I	8.7 ± 0.5	1	7.9 ± 1.9	2	6.0 ± 1.2	3	4.8 ± 1.2	4
J	9.3 ± 0.9	1	8.0 ± 1.5	2	6.8 ± 1.6	3	5.6 ± 2.3	4
N	8.3 ± 0.9	1	7.4 ± 0.7	2	5.4 ± 1.1	3	4.2 ± 1.1	4
O	5.9 ± 2.1	3	7.4 ± 0.9	1	6.6 ± 0.9	2	4.5 ± 1.7	4
Average[a]	8.0a	1.3	7.3a	1.8	6.1b	2.8	4.7c	4.0
Both scales average[a]	7.8a	1.1	6.5a	1.9	5.7c	3.0	4.8d	3.9

[a]Scores within each row with letters in common are not significantly different ($P \geq 0.05$).

TABLE 5
Flavor Scores, Standard Deviations, and Ranked Oxidation Levels for Canola Oils

	Oxidation levels							
Lab code	None	Rank	Low	Rank	Moderate	Rank	High	Rank
Quality scale								
B	6.8 ± 1.0	1	5.0 ± 2.4	3	4.5 ± 1.9	4	5.8 ± 2.1	2
C	7.5 ± 0.8	1	6.9 ± 0.8	2	3.6 ± 0.8	3	2.6 ± 1.0	4
D	8.4 ± 1.0	1	6.1 ± 1.3	2	4.9 ± 1.7	3	2.9 ± 0.9	4
E	7.1 ± 1.1	1	5.4 ± 1.5	2	3.4 ± 2.1	3	2.6 ± 1.8	4
G	7.0 ± 1.0	2	7.8 ± 1.0	1	3.3 ± 1.3	3	2.4 ± 1.0	4
H	6.9 ± 0.7	1	5.5 ± 1.0	2	3.4 ± 0.8	3	3.0 ± 1.1	4
L	9.0 ± 0.0	1	7.0 ± 1.2	2	5.2 ± 2.0	3	3.4 ± 1.5	4
M	8.0 ± 1.0	1	6.4 ± 0.8	2	5.3 ± 1.0	3	4.3 ± 0.5	4
Average[a]	7.6a	1.1	6.3b	2.0	3.7c	3.1	3.4d	3.8
Intensity scale								
A	8.4 ± 0.8	1	6.6 ± 1.3	2	4.5 ± 1.4	3	3.5 ± 1.8	4
F	7.9 ± 1.1	1	5.8 ± 0.9	2	5.0 ± 1.3	3	4.4 ± 1.2	4
I	8.8 ± 0.8	1	7.3 ± 1.0	2	4.5 ± 1.0	3	2.8 ± 0.4	4
J	9.5 ± 0.9	1	8.5 ± 1.3	2	5.8 ± 1.6	3	4.1 ± 1.3	4
N	8.2 ± 0.6	1	6.9 ± 0.7	2	4.6 ± 0.7	3	2.7 ± 0.8	4
O	7.8 ± 1.2	1	6.5 ± 1.3	2	5.8 ± 2.2	3	3.9 ± 2.6	4
Average[a]	8.4a	1.0	6.9b	2.0	5.0c	3.0	3.6d	4.0
Both scales average[a]	8.0a	1.1	6.7b	2.0	4.5c	3.1	3.3d	3.9

[a]Scores within each row with letters in common are not significantly different ($P \geq 0.05$).

relationship of oxidation to change in flavor scores for canola (6,11,12), sunflower (6,12), and soybean oils (6). The mean scores were lower for samples rated by overall quality scaling than for those rated by overall intensity scaling. The range in overall quality scores for corn oil showed a much smaller range (6.8–5.2) from the no-oxidation level to the high-oxidation level than the ranges for the other three oils, even though the PV for the corn oils decreased at similar levels to the other oils (Table 6). A two-way analysis of variance showed significant differences ($P \leq 0.05$) between overall scores for subsequent oxidation levels within each oil type except between the moderate- and high-oxidation soybean oils and the none- and low-oxidation sunflower oils when the intensity scale was used. Also, no significant differences were found between the following oxidation levels for corn oil when the quality scale was used: none-low, low-moderate, and moderate-strong. The lack of significant differences between the subsequent oxidation levels in the corn oils was due

TABLE 6
Flavor Scores, Standard Deviations, and Ranked Oxidation Levels for Corn Oils

	Oxidation levels							
Lab code	None	Rank	Low	Rank	Moderate	Rank	High	Rank
Quality scale								
A	7.6 ± 1.2	1	7.1 ± 1.2	2	6.2 ± 1.6	3	5.9 ± 1.2	4
B	5.5 ± 2.4	1.5	4.8 ± 1.0	3	5.5 ± 1.0	1.5	4.7 ± 1.3	4
C	6.4 ± 0.8	3	7.1 ± 0.9	1	6.7 ± 1.0	2	5.8 ± 1.1	4
D	7.4 ± 1.3	1	6.7 ± 0.8	2	6.6 ± 0.8	3	5.9 ± 1.1	4
E	5.7 ± 2.3	2	6.0 ± 1.3	1	5.5 ± 2.1	3	4.5 ± 1.0	4
F	6.9 ± 1.7	1	6.1 ± 0.8	2.5	5.3 ± 1.2	4	6.1 ± 1.4	2.5
G	5.5 ± 1.1	2	6.9 ± 1.3	1	3.9 ± 0.9	3	3.3 ± 1.0	4
H	5.2 ± 1.5	3	6.3 ± 1.1	1	5.4 ± 1.1	3	4.6 ± 1.2	4
I	7.8 ± 2.1	1	6.7 ± 1.5	2.5	5.8 ± 2.0	4	6.7 ± 2.2	2.5
J	7.3 ± 1.9	1	5.8 ± 1.5	3	6.5 ± 1.9	2	5.2 ± 1.3	4
L	7.5 ± 1.0	1	6.3 ± 1.7	2	6.0 ± 0.8	3	5.5 ± 1.3	4
M	7.0 ± 0.6	3	7.1 ± 0.7	1.5	7.1 ± 0.7	1.5	6.0 ± 1.0	4
N	7.9 ± 0.6	1	6.8 ± 0.7	2	5.4 ± 1.2	3	4.3 ± 1.5	4
O	7.6 ± 0.9	1	6.5 ± 1.6	2.5	6.5 ± 1.7	2.5	6.3 ± 1.7	4
Average[a]	6.8a	1.6	6.5ab	1.9	5.8bc	2.8	5.2c	3.8

[a]Scores within each row with letters in common are not significantly different ($P \geq 0.05$).

to errors in the perceptions of some of the panelists in rating the oxidation levels.

The overall intensity and quality scores were used to rank the oils within each oil type for level of oxidation. The mean rankings showed that 85–90% of the panels were able to correctly rank soybean, sunflower, and canola oils (Tables 3–5). The oxidation levels of the corn oils, determined by overall quality score, were correct 50–60% of the time for the none-, low-, and moderate-oxidation levels and 86% of the time for the high-oxidation level (Table 6). Reports from sensory panel managers participating in this study indicated that many panelists were not as familiar with rating corn oil as they were with the other oils tested.

Variance was calculated for the pooled overall scores of all oils, rated on the quality scale and on the intensity scale, both within laboratories and between laboratories. The highest variances were within laboratories, with the variance increasing slightly with increasing oxidation (Fig. 3). Between-laboratory variance for the quality scale was the lowest; however, only slight differences were noted for intensity and quality scales at the moderate and high oxidation levels. Between-laboratory variances, calculated for each oil separately, showed that the highest variances for the intensity scale were at the none- to low-oxidation range (data not shown). On the other hand, testers who used the quality scale showed the highest between-laboratory variances at the higher oxidation levels (data not shown).

Flavor intensity of descriptors. Ratings of intensities of individual flavors as none, weak, moderate, or strong were quantitated by assigning values of 0, 1, 2, or 3, respectively. Thirteen flavor descriptors were listed on the scoresheets (Figs. 1 and 2). The unaged soybean, sunflower, and canola oils were described as predominantly nutty and buttery (Fig. 4A–C). However, the corn oil was initially characterized as predominantly corny and burnt (Fig. 4D). As oxidation increased, the nutty and buttery flavors gradually decreased in intensity in all oils, but the intensities of the corny and burnt notes in corn oil changed little with increasing oxidation. Off-flavor characteristics of each oil type were present and tended to increase with increasing aging. For example, beany and grassy flavors were detected in soybean oil, burnt and grassy flavors were noted in sunflower oil, and grassy flavor was detected at low intensity levels in canola oil. Rancid and painty flavors generally increased with increasing oxidation of each oil type. These results are in contrast to those of Pongracz (7), who reported that the panel could not detect a specific rancid flavor in aged sunflower oils with peroxide values ranging from 4.6 to 12.6. The linolenate-containing oils, soybean and canola, were also described as fishy, which is typical for oils with 8–10% linolenate (6,12). Most of the flavors in corn oil did not change with increasing oxidation, with the exception of small increases in rancid and painty flavors, as well as a high level of off-flavors (musty, bacon, lard) detected in the highly oxidized sample. Pine, weedy, and musty flavors were detected at low intensity levels in sunflower oil as off-flavors (Fig. 4B), and sulfur and cabbage-like flavors were noted in canola oil as off-flavors(Fig. 4C). The markers most sensitive to increasing oxidation levels were buttery (decreasing in intensity) and rancid and painty (increasing in intensities).

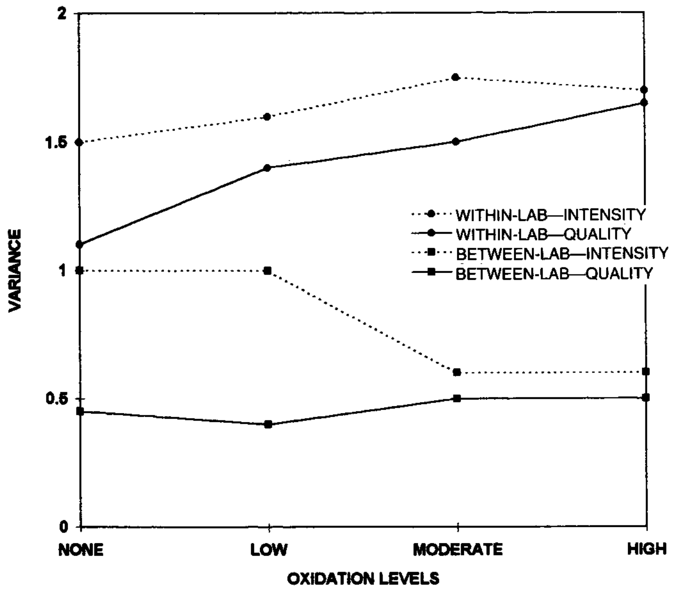

FIG. 3. Between-laboratory and within-laboratory variances for intensity and quality scores pooled over all oil types.

GC volatiles analyses. In conducting GC analyses of volatile compounds, collaborators used a variety of times and temperatures for the various procedures, resulting in a wide range of absolute integrator counts within each oxidized oil type for pentane (Fig. 5) and hexanal (Fig. 6). The objective of the volatile compound portion of the study was to determine the effectiveness of the methods in ascertaining the levels of oxidation of the oils. The direct injection method used by two laboratories—K and P—was able to correctly rank sunflower and soybean oils by both pentane and hexanal analyses. Groups K and P found for canola oil that the low-oxidation sample had more pentane than the moderately oxidized sample, but only P detected more pentane and hexanal in the moderately oxidized corn oil than in the highly oxidized corn oil. These results with direct injection agreed with the data from the previous AOCS collaborative study in the capability of this method to rank oil oxidation (1).

The integrator counts obtained by laboratory N with static headspace for pentane and hexanal showed relatively low absolute counts compared with the groups that used dynamic headspace (Figs. 5 and 6). However, the data from N showed progressively increasing integrator counts with increasing oxidation for all oil types and the capability of this method to properly determine oxidation levels. Data on pentane and hexanal, measured by dynamic headspace, demonstrated that all groups showed increasing integrator counts to match increasing oxidation levels in soybean, sunflower, canola, and corn oils, with the exception of laboratory I for soybean oil and laboratory B for canola oil. By all GC analyses, canola

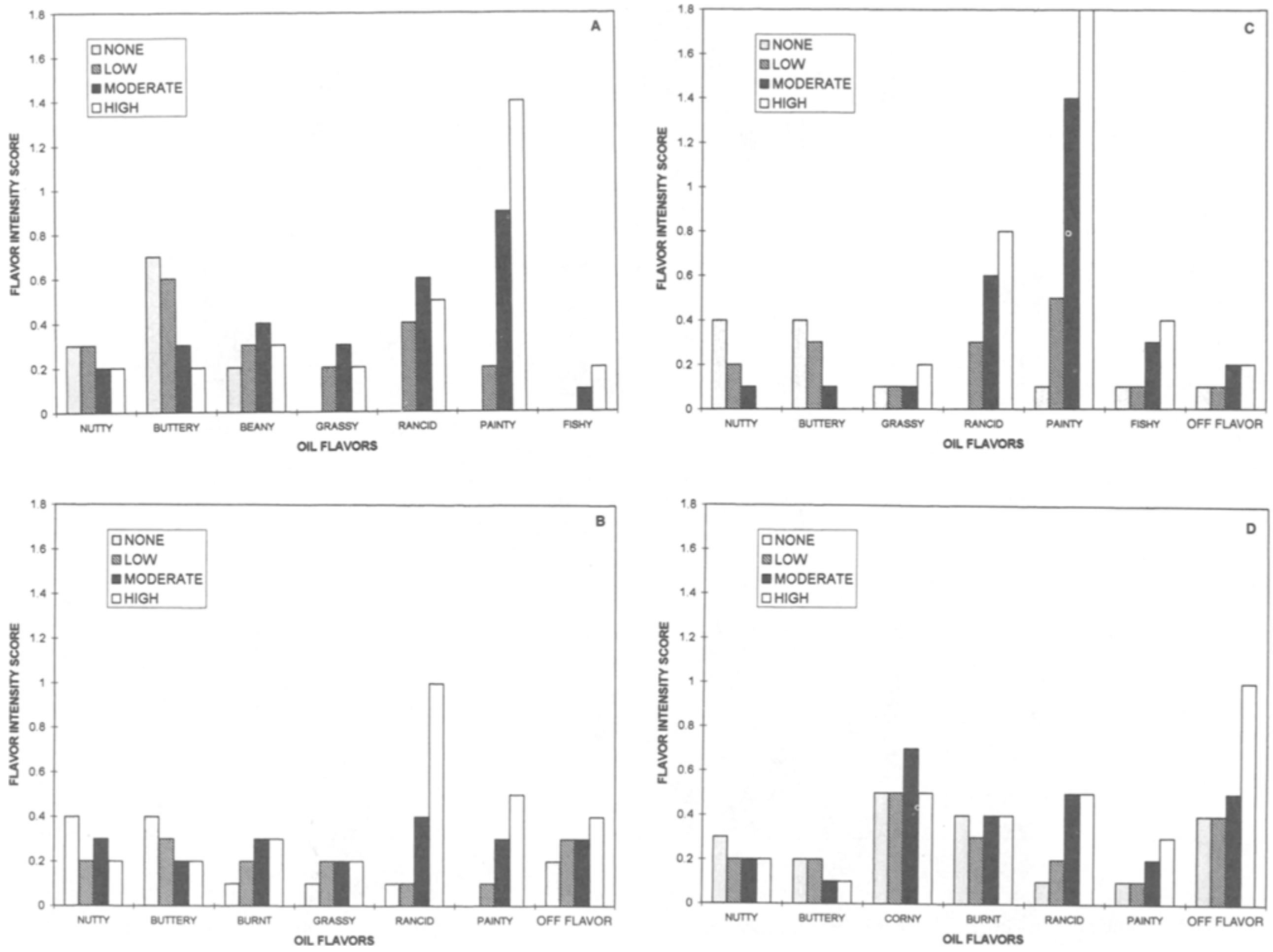

FIG. 4. Intensities of flavor descriptors for soybean (A), sunflower (B), canola (C), and corn (D) oils at four levels of oxidation: none, low, moderate, and high. Scores based on an intensity scale of none = 0, weak = 1, moderate = 2, and strong = 3.

oil had lower levels of pentane and hexanal than the other oils. This result was not unexpected, based on the work of Frankel (13), who reported that pentane and hexanal were degradation products from linoleate oxidation. Canola oil, which had only 21% linoleate compared to 71, 58, and 54 for sunflower, corn, and soybean oils, respectively, would be expected to have less pentane and hexanal formation than the other oils. The canola oil contained 62% oleate, compared to 23, 16, and 26% for soybean, sunflower, and corn oils, respectively; however, volatile compounds, such as octanal, nonanal, and 2-decenal, from oleate oxidation (13) were reported by only one group; therefore, comparisons of volatile compounds formed from oleate decomposition could not be made. The effect of various GC conditions on the amounts of pentane and hexanal reported in the samples was not determined because of the differences in times and temperatures used. Laboratories B, I, and M had the highest levels of these two volatiles; however, the limited information on GC conditions provided by the laboratories did not make it possible to make conclusions about the relationship of volatiles levels and GC conditions.

Relationship of sensory and volatiles data. Correlation coefficients were calculated within sensory data between average overall flavor scores (both intensity and quality scales from Tables 3–6) and intensity of individual descriptors (from Fig. 4). Positive coefficients above 0.90 were calculated between overall flavor score and intensity of buttery flavor, indicating corresponding changes in intensity of buttery flavor

TABLE 7
Pearson Product Moment Correlation Coefficients of Average Overall Flavor Scores[a] with Intensities of Individual Flavor Descriptors[b]

	Flavor descriptors				
Oil type	Nutty	Buttery	Grassy	Rancid	Painty
Soybean	0.92	0.95	−0.77	−0.87	−0.98
Sunflower	0.39[c]	0.94	−0.81	−0.92	−0.92
Canola	0.96	0.97	−0.74	−0.99	−0.99
Corn	0.33[c]	0.92	N.D.[d]	−0.93	−0.90

[a] Flavor score data from Tables 3–6.
[b] Descriptor intensity data from Figure 4.
[c] All coefficients significant at $P \leq 0.05$ except 0.39 and 0.33.
[d] Not determined.

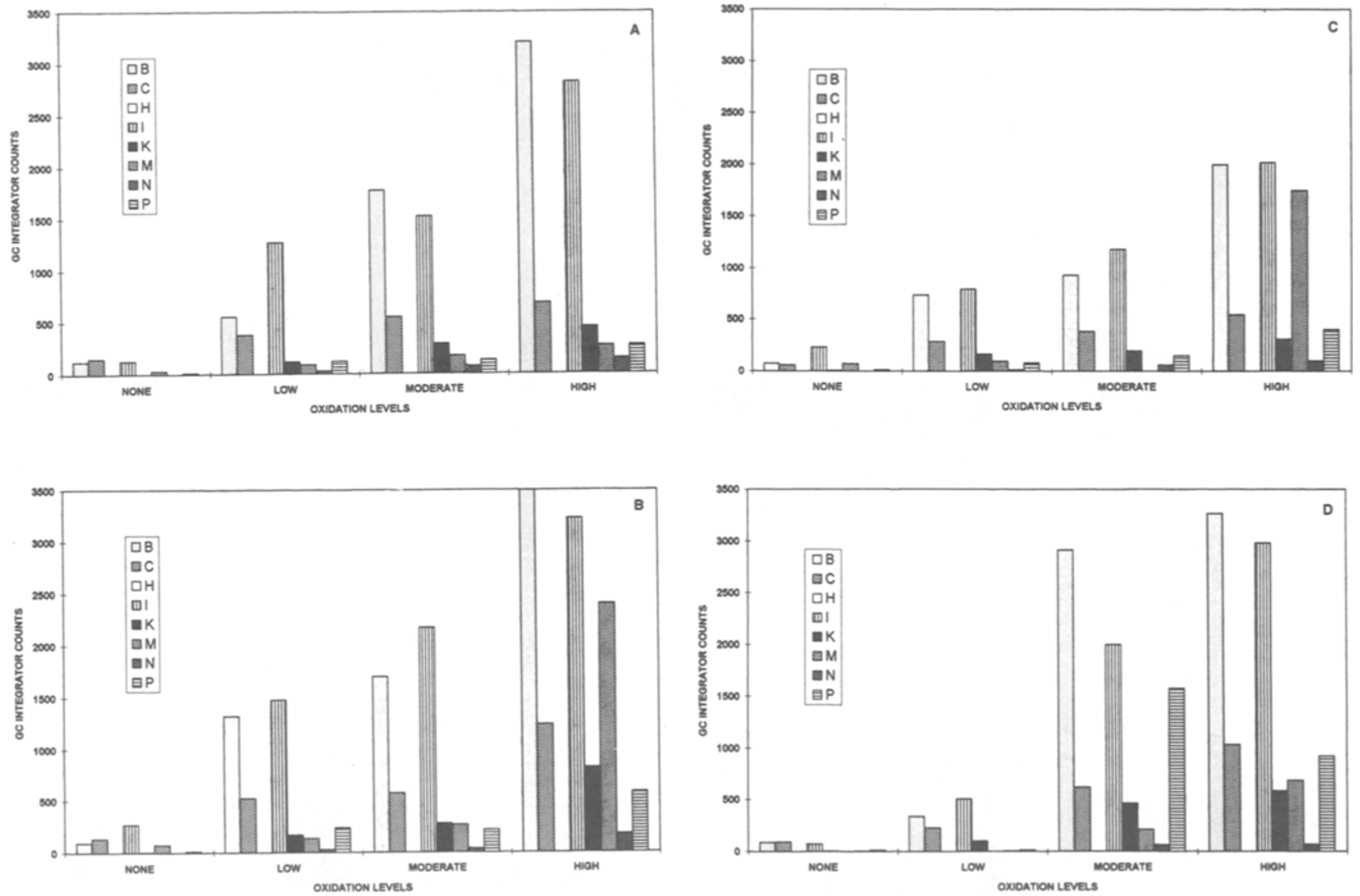

FIG. 5. Levels of pentane in soybean (A), sunflower (B), canola (C), and corn (D) oils at four levels of oxidation: none, low, moderate, and high. Analyses by dynamic headspace GC: laboratories B, C, H, I, M, O; static headspace GC: laboratory N; direct injection GC: laboratories K and P. Laboratory H did not provide data for all oil samples. GC, gas chromatographic.

with decrease in overall flavor score (Table 7). Negative coefficients above –0.90 were calculated for the relationships of increasing painty flavor and decreasing flavor score in all oils. The relationship of rancid flavor and overall flavor was similar to that for painty flavor. The correlation coefficients between grassy flavor intensities and overall scores ranged only from –0.74 to –0.81 for canola, soybean, and sunflower oils. The pattern of increasing and decreasing grassy flavor intensity with decreasing flavor scores contributed to these coefficients. Finally, the correlation between nutty flavor and flavor score was only significant for canola and soybean oils.

Mean ranks of oxidation levels in each oil type by total volatiles, pentane, and hexanal showed that misranking occurred most often for corn oil samples (Table 8). Correlation coefficients between mean ranks of oxidation levels by volatiles and mean ranks of oxidation levels by overall flavor scores (data from Tables 3–6) showed coefficients of 0.99 for soybean and sunflower oils, 0.98 for canola oils, and 0.95–0.97 for corn oils. The mean rankings for soybean and sunflower oils were correct slightly more often than for canola oils, contributing to the slightly lower coefficients for

TABLE 8
Mean Ranking of Volatile Compounds for Oils at None, Low, Moderate, and High Levels of Oxidation and Rank Order Correlation Coefficients with Average Overall Flavor Scores[a]

	Oxidation levels				Correlation coefficients[b]
Oil	None	Low	Moderate	High	
Total volatiles					
Soybean	1.0	2.1	3.1	3.8	0.99
Sunflower	1.0	2.0	3.0	4.0	0.99
Canola	1.0	2.2	2.8	4.0	0.98
Corn	1.3	1.7	3.3	3.7	0.95
Pentane					
Soybean	1.0	2.0	3.0	4.0	0.99
Sunflower	1.0	2.1	2.9	4.0	0.99
Canola	1.0	2.0	3.0	4.0	0.98
Corn	1.1	1.9	3.1	3.9	0.97
Hexanal					
Soybean	1.0	2.1	2.9	4.0	0.99
Sunflower	1.1	2.0	2.9	3.8	0.99
Canola	1.1	2.1	2.8	4.0	0.98
Corn	1.1	1.9	3.1	3.9	0.96

[a] Flavor score data from Tables 3–6; pentane and hexanal data from Figures 5 and 6.

[b] All correlation coefficients significant at $P \leq 0.05$.

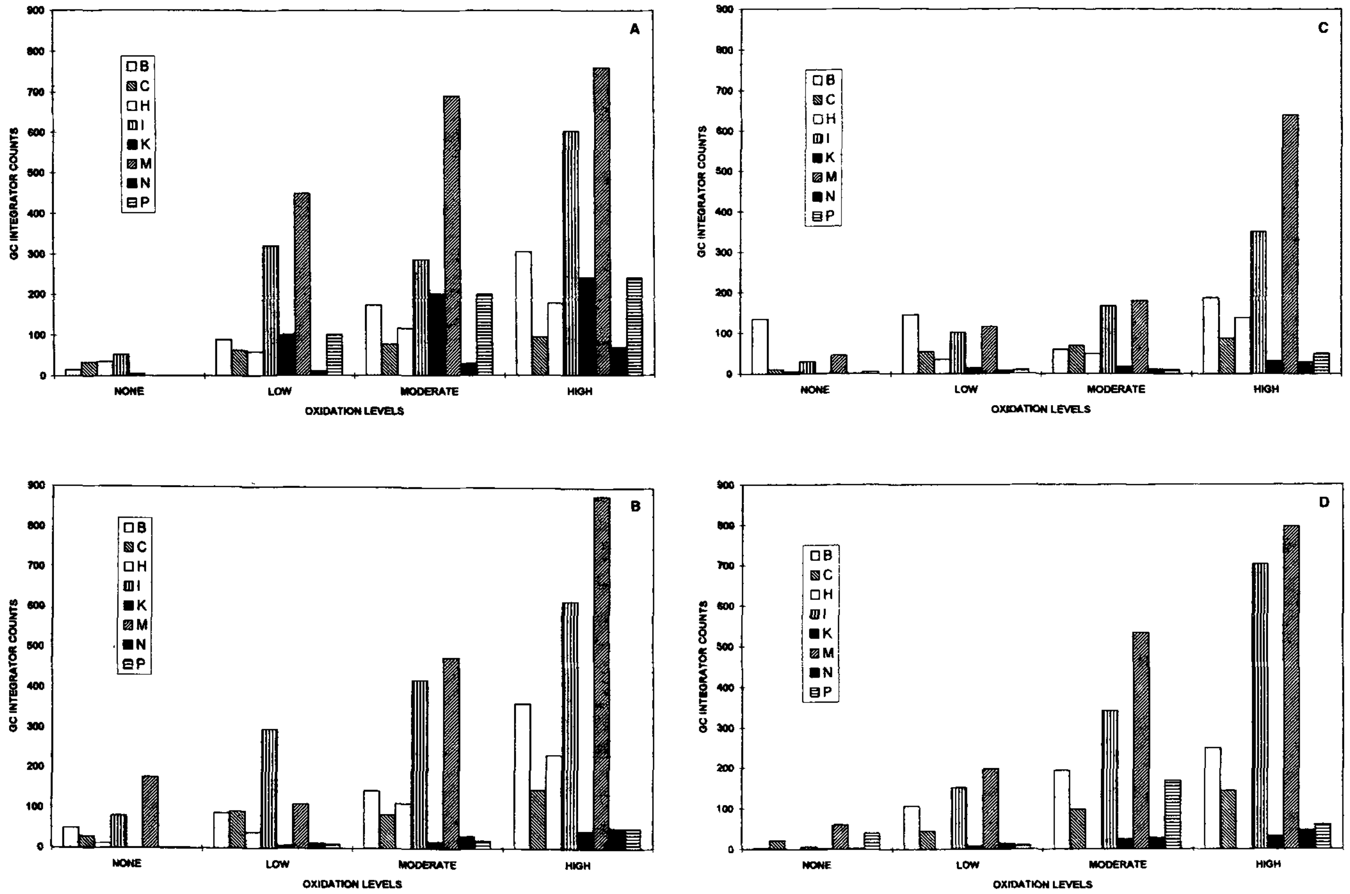

FIG. 6. Levels of hexanal in soybean (A), sunflower (B), canola (C), and corn (D) oils at four levels of oxidation: none, low, moderate, and high. Analyses by dynamic headspace GC: laboratories B, C, H, I, M, O; static headspace GC: laboratory N; direct injection GC: laboratories K and P. Laboratory H did not provide data for all oil samples. See Figure 5 for abbreviation.

canola oil. Corn oils were frequently misranked by both the GC and the sensory methods. Results of this collaborative study showed that these sensory methods—intensity and quality—for flavor analysis and GC methods (direct injection, static headspace and dynamic headspace) for volatile compounds were appropriate for determining levels of oxidation for soybean, sunflower, and canola oils. Oils with distinctive flavors, such as corn, may require modified methods to suitably evaluate oxidation.

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REFERENCES

1. Waltking, A.E., Progress Report of the AOCS Flavor Nomenclature and Standards Committee, *J. Am. Oil Chem. Soc. 59*:116A (1982).
2. Evans, C.D., G.R. List, R.L. Hoffmann, and H.A. Moser, Edible Oil Quality as Measured by the Thermal Release of Pentane, *Ibid. 44*:100A (1967).
3. Dupuy, H.P., S.P. Fore, and L.A. Goldblatt, Direct Gas Chromatographic Examination of Volatiles in Salad Oils and Shortenings, *Ibid. 50*:340 (1973).
4. Dupuy, H.P., E.T. Rayner, and J.I. Wadsworth, Correlation of Flavor Score with Volatiles of Vegetable Oils, *J. Nutr. 104*:628 (1976).
5. Snyder, J.M., E.N. Frankel, E. Selke, and K. Warner, Comparison of Gas Chromatographic Methods for Volatile Lipid Oxidation Compounds in Soybean Oil, *J. Am. Oil Chem. Soc. 65*:1617 (1988).
6. Warner, K., E.N. Frankel, and T.L. Mounts, Flavor and Oxidative Stability of Soybean, Sunflower, and Low Erucic Acid Rapeseed Oils, *Ibid. 66*:558 (1989).
7. Pongracz, G., Determination of Rancidity of Edible Fats by Headspace Gas Chromatographic Detection of Pentane, *Fette Seifen Anstrichm. 88*:383 (1986).

8. *Official Methods and Recommended Practices of the American Oil Chemists' Society,* 3rd edn., American Oil Chemists' Society, Champaign, 1985.
9. Walpole, R.E., and R.H. Myers, *Probability and Statistics for Engineers and Scientists,* 4th edn., Macmillan Publishing Co., New York, 1989.
10. Prell, P., Preparation of Reports and Manuscripts Which Include Sensory Evaluation Data, *Food Tech. 30*:40 (1976).
11. Przybylski, R., L.J. Malcolmson, N.A.M. Eskin, S. Durance-Tod, J. Mickle, and R. Carr, Stability of Low Linolenic Acid Canola Oil to Accelerated Storage at 60°C, *Lebensm. Wiss. U. Technol. 26*:205 (1993).
12. Malcolmson, L.J., M. Vaisey-Genser, R. Przybylski, and N.A.M. Eskin, Sensory Stability of Canola Oil: Present Status of Shelf Life Studies, *J. Am. Oil Chem. Soc. 71*:435 (1994).
13. Frankel, E.N., Volatile Lipid Oxidation Products, *Progress in Lipid Res. 22*:1 (1983).

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