Oxidative Stability of Soybean Oils with Altered Fatty Acid Compositions

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The oxidative stabilities of one canola oil and six soybean oils of various fatty acid compositions were compared in terms of peroxide values, conjugated dienoic acid values and sensory evaluations. Two of the soybean oils (Hardin and BSR 101) were from common commercial varieties. The other four soybean oils were from experimental lines developed in a mutation breeding program at Iowa State University that included A17 with 1.5% linolenate and 15.2% palmitate; A16 with 2% linolenate and 10.8% palmitate; A87-191039 with 2% linolenate and 29.6% oleate; and A6 with 27.5% stearate. Seed from the soybean genotypes was cold pressed. Crude canola oil was obtained without additives. All oils were refined, bleached and deodorized under laboratory conditions with no additives and stored at 60°C for 15 days. The A17, A16, A87-191039 and A6 oils were generally more stable to oxidation than the commercial soybean varieties and canola oil as evaluated by chemical and sensory tests. Canola oil was much less stable than Hardin and BSR 101 oils by both chemical and sensory tests. The peroxide values and flavor scores of oils were highly correlated with the initial amounts of linolenate ($r = 0.95$, $P = 0.001$). Flavor quality and flavor intensity had negative correlations with linolenate, ($r = -0.89$, $P = 0.007$) and ($r = -0.86$, $P = 0.013$), respectively.

KEY WORDS: Altered fatty acid patterns, fatty acid composition, modified fatty acid patterns, oxidation, soybean oils, stability.

Soybean oil is relatively unstable to oxidation. The off-flavors that develop are caused by volatile compounds released during the breakdown of hydroperoxides (1), which are flavorless but unstable compounds formed during the oxidation of unsaturated fats. The hydroperoxides are transformed to secondary products such as aldehydes, alcohols, ketones, acids, hydrocarbons, esters and lactones (1-3).

The degree of unsaturation of a fatty acid has a significant effect on the oxidation rate. The relative reaction rate with oxygen and the hydroperoxide decomposition rate of linolenate (18:3) are much faster than those of linoleate (18:2) and oleate (18:1) (2,4). Because 18:3 oxidizes much easier than the other fatty acids, it has been considered an important cause of off-flavor development in soybean oil, although it accounts for only 7 to 9% of the total fatty acids in soybean oil.

In 1936, Durkee (5) first suggested that 18:3 is an important precursor of the off-flavor compounds. Schwab *et al.* (6), Dutton *et al.* (7) and Evans *et al.* (8) further confirmed Durkee's theory in various ways. Frankel (9) summarized support for this theory, reporting that compounds identified in oxidized soybean oils were, in part, from the oxidation of 18:3. These compounds include acetaldehyde, propanal, 2-pentenal, 3-hexenal, 2,4-heptadienal,2,4,7-decatrienal and 2-pentenyl furan (9).

Partly hydrogenated soybean oils, whose 18:3 content was reduced to below 3%, were reported to have improved flavor and oxidative stabilities compared with unhydrogenated oils (8). Therefore, until recently, most oils sold commercially had been partly hydrogenated to reduce the 18:3 content. Consumer interest in "all natural" products has reduced this practice. An alternative method for reducing the 18:3 content is through breeding new soybean varieties with altered fatty acid patterns (10-13).

In the current study, four newly developed soybean lines, from the breeding program at Iowa State University, were examined to determine whether the altered fatty acid compositions of the oils could reduce the rate of oxidation and the development of off-flavors compared with traditional soybean oils. The experimental lines were A17 with 1.5% 18:3 and 15.2% palmitate (16:0); A16 with 2% 18:3 and 10.8% 16:0; A87 with 2% 18:3 and 29.6% oleate (18:1); and A6 with 27.5% stearate (18:0). Canola oil and two traditional soybean oils (Hardin and BSR 101) were used for comparison.

EXPERIMENTAL PROCEDURES

Extraction, refining and deodorization. Soybean seed of six genotypes [Hardin, BSR 101, A17, A16, A87-191039 (A87) and A6] was produced in 1989 near Ames, Iowa by W.R. Fehr, Iowa State University. The oil was removed from 30 kg of seed of each genotype by cold pressing with a Hander Screw Press (Model H54, Osaka, Japan). The crude oil yields (in percentage of seed weight) were 13.4% for Hardin; 12.1% for BSR 101; 5.7% for A17; 6.7% for A16; 6.6% for A87; and 4.1% for A6. Crude canola oil was provided by CSP Foods, Ltd. (Saskatoon, Saskatchewan, Canada) without additives.

The free fatty acid (FFA) contents of the crude oils were determined by American Oil Chemists' Society's (AOCS) official method Ca-5a-40 (14), and then were removed by alkali-refining according to AOCS official method Ca-9d-52 (14). A hot plate and a large magnetic stirrer set at slow speed were used to simulate the hot water bath and the paddle described in the method. At the end of the alkali-refining procedure, the soapstock was separated from the oil by a 20-min centrifugation at $g = 10{,}000$.

The alkali-refined oil was bleached according to AOCS official method Cc-8b-52 (14) with the official natural bleaching earth (for soybean oils) or with the official activated bleaching earth (for canola oil). Both bleaching earths were purchased from the Office of the Secretary of AOCS (Champaign, IL). The bleached oil was steam deodorized by a high-vacuum (0.1 Torr or better), high-temperature (230 to 240°C for 2 h) steam distillation procedure reported by Stone and Hammond (15).

Because of the dark green color and strong fish-like flavor of the refined canola oil, it was bleached and deodorized twice, whereas refined soybean oils were bleached and deodorized only once. Immediately after deodorization, all oils were tested for peroxide value (PV), stored under nitrogen and held at −10°C until storage tests began. No additives or citric acid were included in any oils. Duplicate sets of each oil were refined, bleached and deodorized separately.

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Storage tests. Each oil (250 mL) was stored in a 500-mL beaker loosely covered and held in a 60°C oven for 15 days. Aliquots were taken and analyzed at regular intervals. Duplicate sets of storage tests were conducted on each oil.

Chemical analyses. In a review article, Frankel (16) suggested that minor soybean oil constituents such as sterols contribute to off-flavor development, depending on the relative concentrations present. Tocopherols in soybean oil also contribute to off-flavor development by increasing the oil's oxidative stability (1), but the effects are dependent upon concentration (17). Neumann *et al.* (18) compared the susceptibilities of soybean, sunflower and peanut oils to singlet molecular oxygen photooxidation and concluded that the enhanced instability of soybean oil was because of its pronounced unsaturation, as well as its lack of protective components such as tocopherols, as compared with peanut and sunflower oils.

The tocopherol and sterol contents of refined, bleached and deodorized (RBD) oils were determined by gas-liquid chromatography of the saponified and extracted compounds on a 30 m × 0.32 mm i.d. capillary column (with a 0.5-μm film of cross-linked 5% Phenylsilicone and 95% Methylsilicone; Supelco, Bellefonte, PA). A Hewlett-Packard Model 5890A gas-liquid chromatograph (GLC) (Palo Alto, CA) equipped with a split-splitless injector and a flame ionization detector was used. Peak areas were measured with a Hewlett-Packard 3390A reporting integrator. Samples were saponified with potassium hydroxide and extracted with ether in a 30-min distillation. The solvents were removed by evaporation under nitrogen. The saponified tocopherols and sterols were dissolved in cholesteryl isovalerate pyridine/butyric anhydride solution and measured by GLC. The tocopherol and sterol contents of the finished oils were not significantly different among the oils.

The peroxide values (PV) of the RBD oils were determined by the Stamm Test (19), and conjugated dienoic acid (CDA) values were measured according to AOCS official method Ti-la-64 (14). Fatty acid methyl esters (FAME), prepared according to the method of Metcalfe *et al.* (20), were determined by gas-liquid chromatography (Varian Aerograph series 3700 GLC, equipped with a flame ionization detector, Varian Associates, Palo Alto, CA) of the methyl esters on a 6.0 ft × 0.085 in stainless-steel packed column (100/120 Gas Chrom Q II with 10% Silar 10C coating; Alltech Associates, Deerfield, IL). Peak areas were measured with a Hewlett-Packard 3390A reporting integrator. All test results are the average of duplicate samples.

Sensory evaluation. Triangle tests (21) were used to determine the panelists' abilities to recognize oxidized flavors. Five additional training sessions were conducted to develop agreement on oxidized flavor and sample scores. Twelve trained panelists evaluated the oils in isolation booths. The oils were tasted at ambient temperature and scored according to the AOCS Flavor Quality Scale and AOCS Flavor Intensity Scale (22). On the Flavor Quality Scale, 8 to 10 is acceptable and 1 to 3 is poor and repulsive. On the Flavor Intensity Scale, 10 is bland and 1 is an extremely intense flavor. The samples were presented in random order, and the panelists were instructed to smell the samples first and then to taste them in approximate order of increasing odor intensity. This procedure reduced the possibility of a strongly flavored sample overwhelming a panelists' ability to judge less flavorful samples. Samples were held in the mouth for 10 to 30 s and then expectorated. Panelists rinsed their mouths with distilled water between samples. In addition, unsalted crackers were used to remove the oxidized flavor and oil residue from their mouths. A fresh, bland soybean oil was provided as a reference standard to aid in judging samples.

Statistical analysis. The data were analyzed by means of Analysis of Variance (ANOVA), Duncan's Multiple Range Test (Duncan's Test) and linear regression (23). Pearson correlation coefficients were determined from mean values of the two replicates. Statistical significance was accepted at a level of $P < 0.05$.

RESULTS

Chemical analyses. The fatty acid compositions of the seven oils are presented in Table 1. The 18:3 contents of all soybean oils were lower than expected, likely because of the hot, dry conditions during the 1989 growing year, which favors saturated fatty acid development in the seed (24,25). For example, when grown during a normal Iowa summer, Hardin and BSR 101 oils will have typical 18:3 contents of 7.5 and 8.0%, respectively, compared with the 5.7 and 6.5% levels observed (26,27).

The relative amounts of 18:3 among the seven oils on day 0 increased in the following order: A17 < A16 = A87 < A6 < Hardin < BSR 101 < canola. The A17 oil contained more 16:0 than did the other six oils. The A16 and A17 oils contained more 18:1 and less linoleate (18:2) than did the commercial varieties. The A87 oil contained more 18:1 than did oils from Hardin and BSR 101. When compared with all soybean oils, canola oil was lower in 16:0 and 18:2 and higher in 18:1 and 18:3.

Oxidation causes a decrease in the relative percentages of the unsaturated fatty acids and an increase in the relative percentages of the saturated fatty acids (26,28). In general, the end values for all seven oils reflected this change in 18:2, 18:3 and 16:0, but the differences between the beginning and ending values were not great. White and Miller (27) and Moser *et al.* (28) also found small differences between beginning and ending fatty acid values when oils were stored at 60°C. In comparing the percentages of polyunsaturated fatty acids (PUFA), all oils, except A16, showed an expected drop in the percentages of polyunsaturation during oxidation.

The PVs of oils are shown in Table 2. After 2 and 7 days of storage, there was no significant difference in PVs of the oils, except for canola oil on day 2. By day 15, however, PVs of oils from A17, A16, A87 and A6 were significantly lower than those for Hardin, BSR 101 and canola oils. At day 15, the PV for canola oil was significantly higher than those for all other oils. The faster oxidation of the canola oil was likely due to its high 18:3 content. Hardin and BSR 101 oils, which were the second highest in 18:3, were the next highest in PVs by day 15; whereas A17, A16 and A87 had low 18:3 contents and low PVs by day 15. Oil from A6 had a low PV at day 15, but an intermediate amount (4%) of 18:3; however, the oil also had a low content of 18:2 and, thus, a low total PUFA content.

The amount of CDAs, which are formed during oxidation of polyunsaturated fatty acids, can be measured by the absorbance in the UV spectrum at 233 nm. Table 3

TABLE 1

Fatty Acid Composition (relative area %) of Oils Before and After 60°C Storage for 15 Days

Oil type	Relative fatty acid composition by GLC, %[a]					
	16:0	18:0	18:1	18:2	18:3	18:2 + 18:3
Hardin						
Beginning values	10.7	3.7	25.7	54.5	5.7	60.2
Ending values	10.9	3.8	26.4	53.6	5.1	58.7
BSR 101						
Beginning values	10.4	4.1	23.3	56.5	6.5	63.0
Ending values	10.6	4.1	24.0	55.7	5.7	61.4
A17						
Beginning values	15.2	5.4	30.0	48.7	1.5	50.2
Ending values	15.5	5.6	30.2	47.4	1.2	48.6
A16						
Beginning values	10.8	5.8	32.5	48.7	2.0	50.7
Ending values	11.0	5.8	32.6	49.9	1.0	50.9
A87						
Beginning values	10.3	4.3	29.6	54.2	2.0	56.2
Ending values	10.8	4.7	30.3	53.0	1.4	54.4
A6						
Beginning values	8.4	27.5	21.5	39.5	4.0	43.5
Ending values	8.6	27.1	22.0	38.9	3.5	42.4
Canola						
Beginning values	3.8	1.7	63.1	21.2	10.1	31.3
Ending values	3.8	1.5	64.8	20.0	9.1	29.1

[a]Values represent the average of duplicate runs of two replicates.

TABLE 2

Peroxide Values[a] of Oils During 60°C Storage for 15 Days

Oil type	Day 0	Day 2	Day 7	Day 15
Hardin	0.1[b]	1.2[b]	13.5[b]	45.2[c]
BSR 101	0.2[b]	1.8[b]	18.5[b]	51.8[c]
A17	0.1[b]	0.5[b]	16.8[b]	27.4[b]
A16	0.2[b]	1.1[b]	15.0[b]	25.8[b]
A87	0.1[b]	1.0[b]	14.2[b]	24.7[b]
A6	0.4[c]	1.1[b]	12.0[b]	25.0[b]
Canola	0.1[b]	7.4[c]	17.4[b]	64.5[d]

[a]Values represent the average of duplicate analyses of two replicates except for A17, where only one replicate was available.

[b-d]Values in the same column with different superscript letters are significantly different ($P < 0.05$) as measured by Duncan's Test.

TABLE 3

Conjugated Dienoic Acid Values[a] of Oils During 60°C Storage for 15 Days

Oil type	Day 0	Day 2	Day 7	Day 15
Hardin	0.13[b]	0.22[b]	0.50[b]	1.34[b]
BSR 101	0.19[c]	0.27[b]	0.52[b]	1.60[b]
A17	0.16[c]	0.22[b]	0.50[b]	1.10[b]
A16	0.15[b]	0.22[b]	0.48[b]	1.30[b]
A87	0.11[b]	0.29[b]	0.46[b]	1.46[b]
A6	0.29[d]	0.32[b]	0.53[b]	1.27[b]
Canola	0.74[e]	0.81[c]	1.07[c]	1.50[b]

[a]As in Table 2.

[b-e]As in Table 2.

shows CDA values for the oils during storage. After day 0, only canola oil differed significantly ($P < 0.05$) from the other oils in CDA values at days 2 and 7. This difference is likely due to canola oil's high 18:3 content, which is oxidized more quickly than are 18:2 and 18:1 (4). By day 15, even canola oil was not significantly different from the other oils. Perhaps, after the initial rapid formation of CDA from 18:3 was exhausted, the relatively low 18:2 content of canola oil did not contribute as much CDA as did the 18:2 from the other oils. Thus, the CDA values for all oils evened out. At day 15, the relative CDA values of the six soybean oils generally followed the same order as the PUFA contents; the higher the PUFA, the higher the CDA value. The significant differences among CDA values on day 0 were probably due to the close agreement of duplicate batches of the oils. After oxidizing, duplicates stored at the same conditions begin to differ somewhat. St. Angelo *et al.* (29) and White and Miller (27) reported that CDA did not develop quickly at room or accelerated room temperature storage conditions.

Sensory evaluation. The flavor quality scores of the oils (Table 4) on day 0 were significantly higher (better) ($P < 0.05$) for Hardin, BSR 101, A16 and A87 than for A17, A6 and canola oils. The A6 oil was graded significantly lower ($P < 0.05$) than Hardin and BSR 101 oils on days 0, 2 and 7, but there was no difference by day 15. Throughout storage, canola oil was generally significantly lower ($P < 0.05$) than most other oils.

As storage progressed, the flavor quality scores for Hardin, BSR 101, A6 and canola oils dropped more rapidly than did those for A17, A16 and A87. Although the differences were not significant, on day 15, oils from A17 and A87 tended to have higher scores than did Hardin, BSR 101 and A6. On day 15, A16 oil had a significantly higher ($P < 0.05$) flavor quality score and canola oil had a significantly lower ($P < 0.05$) score than did all other oils. The flavor quality scores of the oils on day 15 generally followed the order of the beginning values of 18:3 content found in the oils. The higher the beginning 18:3 content, the lower (worse) the flavor quality score of the oil.

TABLE 4

Flavor Quality Scores[a] of Oils During 60°C Storage for 15 Days

Oil type	Day 0	Day 2	Day 7	Day 15
Hardin	9.3[b]	8.5[c]	7.3[b]	5.5[c]
BSR 101	9.5[b]	8.4[c]	7.3[b]	5.8[c]
A17	8.6[c]	8.5[c]	7.7[b]	6.2[c]
A16	9.2[b]	8.9[c]	7.7[b]	7.0[b]
A87	9.3[b]	9.2[b]	7.5[b]	6.2[c]
A6	8.2[c]	5.9[d]	6.0[c]	5.6[c]
Canola	8.0[c]	6.5[d]	5.3[c]	4.2[d]

[a]Values represent the average of duplicate analyses of two replicates.
[b-d]As in Table 2.

TABLE 5

Flavor Intensity Scores[a] of Oils During 60°C Storage for 15 Days

Oil type	Day 0	Day 2	Day 7	Day 15
Hardin	9.4[b]	8.4[b]	7.1[c]	5.6[c]
BSR 101	9.6[b]	8.4[b]	6.3[c]	5.9[c]
A17	8.6[c]	8.5[b]	7.2[c]	6.2[c]
A16	9.0[b]	8.9[b]	7.5[b]	6.8[b]
A87	9.3[b]	9.1[b]	7.6[b]	6.5[b]
A6	8.3[c]	6.1[c]	6.8[c]	5.3[c]
Canola	8.2[c]	6.5[c]	5.2[d]	4.5[d]

[a]Values represent the average of duplicate analyses of two replicates.
[b-d]As in Table 2.

The flavor intensity scores (Table 5) of Hardin, BSR 101, A16 and A87 oils were significantly higher (better) ($P < 0.05$) than A17,A6 and canola oils on day 0. On day 2, the A6 and canola oils were graded significantly lower (worse) ($P < 0.05$) than Hardin, BSR 101, A17, A16 and A87 oils. Canola oil had significantly lower ($P < 0.05$) flavor intensity scores throughout the storage test than did most other oils. On day 15, the flavor intensities of the oils also tended to follow the order of the beginning values of 18:3 contents in the oils. The higher the beginning 18:3 content, the lower (worse) the flavor intensity score of the oil.

DISCUSSION

During storage the A17, A16, A87 and A6 oils were more stable to peroxide development than the other oils, but there were few significant differences among the oils in CDA development. The A17, A16 and A87 oils had more acceptable flavor quality scores than did the other oils, although only the A16 oil had a significantly better ($P < 0.05$) score. The flavor intensities of the A17, A16 and A87 oils tended to be more bland than those of the other oils, but only the values of A16 and A87 oils were significantly higher ($P < 0.05$).

The A6 oil was semisolid at room temperature and was therefore different from other oils in texture. Its mouth-coating property may have hindered washing its off-flavor from the tongue and thus enhanced its flavors. Through mutation breeding, other flavor compounds also may have been changed. Therefore, the panelists may have had difficulty in accurately judging the extent of oxidation of the A6 oil and tended to rank it lower than the other soybean oils. The A6 oil was more stable than Hardin, BSR 101 and canola oils in PV tests. White and Miller (27) also found similar results when comparing the room temperature flavor stability of A6 oil with other soybean oils.

TABLE 6

Correlation Coefficients and Probability Levels Among Selected Test Results

Test measurements	Correlation coefficients	Probability level
18:3[a] *vs.* PV[b]	0.95	0.001
18:3 *vs.* flavor quality	−0.89	0.007
18:3 *vs.* flavor intensity	−0.86	0.013
PV *vs.* flavor quality	−0.80	0.032
PV *vs.* flavor intensity	−0.72	0.071

[a]18:3 = initial values.
[b]PV, flavor quality and flavor intensity scores from oils after storage at 60°C for 15 days.

The correlations and probability levels between the initial 18:3 contents of oils and PVs, flavor quality scores and flavor intensity scores of oils after storage at 60°C for 15 days are shown in Table 6. All correlations were high and were highly significant. The CDA values did not correlate well with the 18:3 contents of the oils ($r = 0.63$, $P = 0.13$) nor with the total PUFA values ($r = 0.13$, $P = 0.79$). In fact, the PUFA values did not correlate well with any other test results. The flavor quality and the flavor intensity scores of oils were highly correlated with each other ($r = 0.97$, $P = 0.0004$). The poorer the flavor quality of the oil, the stronger the oxidized flavor intensity. As shown from these correlation coefficients, the initial 18:3 contents of oils can closely predict their oxidative and flavor stabilities.

The correlations between the PVs of oils and flavor quality and flavor intensity scores after storage at 60°C for 15 days also are shown in Table 6. The correlation coefficient (r) for PV *vs.* flavor quality was significant at $P = 0.032$, and for PV *vs.* flavor intensity the values approached significance at $P = 0.071$.

In general, the oils from the experimental soybean lines (A17, A16, A87-191039 and A6) were more stable to oxidation than the commercial varieties and canola oil, as evaluated by chemical and sensory tests. The PV and flavor scores after 15 days of storage at 60°C were highly correlated with initial 18:3 contents of the oils, and PV was fairly well correlated with both flavor scores.

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REFERENCES

1. Frankel, E.N., *J. Sci. Food Agric. 54*:495 (1991).
2. Frankel, E.N., in *Flavor Chemistry of Fats and Oils*, edited by D.B. Min, and T.H. Smouse, American Oil Chemists' Society, Champaign, IL, 1985, p. 1.

3. Grosch, W., in *Autoxidation of Unsaturated Lipids,* edited by H.W.S. Chan, Academic Press, London, 1987, p. 95.
4. Gunstone, F.D., and T.P. Hilditch, *J. Chem. Soc. 1945*:836 (1945).
5. Durkee, M.M., *Ind. Eng. Chem. 28*:898 (1936).
6. Schwab, A.W., H.A. Moser, P.A. Cooney and C.D. Evans, *J. Am. Oil Chem. Soc. 27*:314 (1950).
7. Dutton, H.J., C.R. Lancaster, C.D. Evans and J.C. Cowan, *Ibid. 28*:115 (1951).
8. Evans, C.D., R.E. Beal, D.G. McConnell, L.T. Black and J.C. Cowan, *Ibid. 41*:60 (1964).
9. Frankel, E.N., *Prog. Lipid Res. 22*:1 (1982).
10. Dutton, H.J., *J. Am. Oil Chem. Soc. 58*:234 (1981).
11. Graef, G.L., W.R. Fehr, L.A. Miller, E.G. Hammond and S.R. Cianzio, *Crop Sci. 28*:55 (1988).
12. Hammond, E.G., and W.R. Fehr, in *Biotechnology for the Oils and Fats Industry,* edited by J.B.M. Rattray, and C. Ratledge, American Oil Chemists' Society, Champaign, 1984, p. 89.
13. Wilcox, J.R., and J.F. Cavins, *Theor. Appl. Genet. 71*:74 (1985).
14. *Official and Tentative Methods of the American Oil Chemists' Society,* 3rd edn., edited by R.C. Walker, American Oil Chemists' Society, Champaign, 1983.
15. Stone, R.R., and E.G. Hammond, *J. Am. Oil Chem. Soc. 60*:1277 (1983).
16. Frankel, E.N., *Prog. Lipid Res. 19*:1 (1980).
17. Min, D.B., and M.Y. Jung, in *Flavor Chemistry of Lipid Foods,* edited by D.B. Min, and T.H. Smouse, American Oil Chemists' Society, Champaign, 1989, p. 242.
18. Neumann, M.M., S.N. Fusero and N.A. Garcia, *J. Am. Oil Chem. Soc. 68*:662 (1991).
19. Hamm, D.L., E.G. Hammond, V. Parvanah and H.E. Snyder, *Ibid. 42*:920 (1965).
20. Metcalfe, L.D., A.A. Schmitz and J.R. Pelka, *Anal. Chem. 28*:514 (1966).
21. *Manual on Sensory Testing Methods,* American Society for Testing and Materials, Philadelphia, 1968, STP 434.
22. Warner, K., in *Flavor Chemistry of Fats and Oils,* edited by D.B. Min, and T.H. Smouse, American Oil Chemists' Society, Champaign, 1985, p. 207.
23. Montgomery, D.C., in *Design and Analysis of Experiments,* 2nd edn., edited by D.C. Montgomery, John Wiley and Sons, Inc., New York, 1984, p. 43.
24. Howell, R.W., and F.I. Collins, *Agron. J. 49*:593 (1957).
25. Chu, W.S., and V.L. Sheldon, *J. Am. Oil Chem. Soc. 56*:71 (1979).
26. Miller, L.A., and P.J. White, *Ibid. 65*:1324 (1988).
27. White, P.J., and L.A. Miller, *Ibid. 65*:1334 (1988).
28. Moser, H.A., C.D. Evans, G. Mustakas and J.C. Cowan, *Ibid. 42*:811 (1965).
29. St. Angelo, A.J., R.L. Ory and L.E. Brown, *Ibid. 52*:34 (1975).

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