The Impact of Furanoid Fatty Acids and 3-Methylnonane-2,4-dione on the Flavor of Oxidized Soybean Oil[1]

Jian-Wen Kao, Xiangdong Wu, Earl G. Hammond, and Pamela J. White*

Department of Food Science and Human Nutrition, Iowa State University, Ames, Iowa 50011

ABSTRACT: Oils from soybeans with high or low contents of furanoid fatty acids were evaluated during storage for flavor intensity of soybean oil (SBO) off-flavor, but no significant differences were found. In addition, the compound 3-methylnonane-2,4-dione (MND), a breakdown product of furanoid fatty acids suggested by other researchers to contribute to reversion flavor of SBO, was evaluated for its contribution to off-flavor. The compound was synthesized in the laboratory and purified by gas chromatography (GC) on a Silar 10 C column. GC analysis of the purified MND on a nonpolar SPB-1 column showed two well-separated main peaks that have been suggested to represent keto and enol forms. Between these two peaks, a bridge of poorly resolved compounds may have represented various possible enol forms or an equilibration among these forms during the GC separation. MND had an intense straw-like and fruity odor when evaluated at the outlet of a gas chromatograph. Sensory evaluation of MND in a mineral oil/water emulsion system showed that its flavor intensity increased almost imperceptibly with increased concentration (from 0.09 to 2.56 ppm). An explanation for this unusual flavor response may be that, when molecularly dispersed in air, MND has an intense odor, but when placed in a mineral oil or soybean oil emulsion, MND may exist in a form with relatively low flavor intensity, or it may be bound by the media. The concentrations of MND in SBO at various peroxide values were measured at 0 to 0.804 ppb, which were far less than concentrations tested in mineral oil/water emulsions during sensory evaluation and below published odor threshold values for MND in oil. Therefore, these results do not support the theory that furanoid fatty acids or MND contribute strongly to the reversion flavor of SBO.
JAOCS 75, 831–835 (1998).

KEY WORDS: Flavor threshold, gas chromatography, 3-methylnonane-2,4-dione, peroxide value, reversion flavor, sensory evaluation, soybean oil.

3-Methylnonane-2,4-dione (MND) has been found in reverted soybean oil (SBO), and it has been suggested that MND contributes significantly to reversion flavor (1,2). MND may be derived from light-induced oxidation of the furanoid fatty acid (F acid) 10,13-epoxy-11,12-dimethyloctadeca-10,12-dienoic acid, which was one of three F acids detected in SBO by Guth and Grosch (3). Two other F acids reported in SBO are 10,13-epoxy-11-methyloctadeca-10,12-dienoic acid and 12,15-epoxy-13,14-dimethyleicosa-12,14-dienoic acid. The total F acid content of SBO is comparatively low (0.016 to 0.049%) (4). The F acids are found in both plants and animals; however, they may differ in chainlength, placement, and methylation of the furan ring.

The odor threshold of MND was reported by Guth and Grosch (1) to be low (0.007 to 0.014 ng/L) in air, but the odor threshold increased greatly when it was dissolved in refined SBO (15 to 30 μg/kg); however, the taste threshold of MND in an oil was not determined. When MND was analyzed on capillary columns of low or intermediate polarity, it separated into two peaks, believed to be keto and enol tautomers; however, only one peak was observed on a polar column.

The objectives of the current work were to evaluate (i) the impact of the amount of F acids in SBO off-flavor development, and (ii) the flavor intensity and chemical characteristics of synthetic MND and its flavor impact during oxidation of SBO under fluorescent lights at 35°C.

EXPERIMENTAL PROCEDURES

Soybeans. To study the effect of F acid content on oxidized flavor development of SBO, six soybean varieties that represented a wide range in F acid content were planted in Puerto Rico in January 1996 and harvested in April 1996. Varieties PI 398672, PI 398955, and FA4 had the lowest F acid content, and PI 165929, PI 171444 and PI 323276 had the highest F acid content. Only soybean variety PI 323276 produced enough seeds for sufficient oil to do sensory evaluations. When combined, seed from varieties PI 398672 and PI 398955 provided an adequate oil sample.

Fatty acid methyl ester (FAME) analysis. Oil from soybeans was extracted as previously described (5) and converted to FAME before injection into a Hewlett-Packard Model 5890 gas chromatograph (Avondale, PA). The F acids were measured as described by Wu *et al.* (6).

Oxidative stability of SBO with high and low furanoid acid contents. SBO extraction. The soybean seeds with high and low F acid content were ground in a Wiley Mill (Arthur H. Thomas Co. Scientific Apparatus, Philadelphia, PA), which

[1]This is Journal Paper J.17472 of the Iowa Agriculture and Home Economics Experiment Station, Ames, Iowa. Project No. 3396.

*To whom correspondence should be addressed at Department of Food Science and Human Nutrition, 2312 Food Sciences Bldg., Iowa State University, Ames, IA 50011. E-mail: pjwhite@iastate.edu

was fitted with a 2-mesh delivery unit. The resulting soybean powder was extracted in a Soxhlet extractor. Sixty grams of soybean powder was put into a cotton-covered thimble, and 250 mL hexane was added to the flask and refluxed for 4 h. Both varieties required about 20 such extractions to obtain sufficient quantities of oil for further analyses. The extracts were pooled into two duplicate samples for each level of F acid content. The hexane was evaporated in a rotary evaporator under a water pump vacuum at 50˚C. The crude oils were stored at –20˚C under nitrogen until needed.

SBO processing. Crude oils from each replicate of the two varieties were refined, bleached, and deodorized as follows before being used in storage stability tests. The free fatty acid (FFA) content of the crude oils was determined by modified AOCS official method Ca-5a-40 (7). An amount of NaOH (14˚Bé) was added to each oil according to AOCS Official Method Ca 9b-52 (7). The refinings were made by using 87.5% of the maximum of 14˚Bé sodium hydroxide solution. The alkali-refined oils were bleached with Official bleaching earth (3%) by using the AOCS official method Cc 8b-52 (7). The bleached oils were steam-deodorized at high vacuum (<0.005 mm Hg) and high temperature (230 to 240˚C for 2 h) as described (8) and modified according to Moulton (9). After deodorization, the oil was cooled below 35˚C before releasing the vacuum. The oil was then stored under nitrogen at –20˚C until needed.

Oxidative stability tests. Two replicates of both SBO (65 g each) with high and low F acid were stored in 500-mL beakers, to obtain a large surface area for exposure to air, and then covered with plastic wrap at 35˚C under 1937 lux of fluorescent light until their peroxide values (PV) reached 35 meq/kg. This PV was selected so that the oil would be well oxidized, for easier detection of potential differences in the oil types. Two PV measurements were determined on each replicate oil sample by using the Stamm method as modified by Hamm *et al.* (10).

Sensory evaluation of the fresh and oxidized oil samples. Sensory evaluations were performed on the freshly deodorized oils and at PV of approximately 10 and 35 meq/kg. The oils were presented in emulsion form at room temperature as described by Dixon and Hammond (11). The 24 samples (4 emulsion samples × 3 PV × 2 replications) were randomly presented to the panelists, four samples at a time, during six sessions. Panelists were trained during two practice sessions with both fresh and oxidized SBO to familiarize them with samples similar to those they would be evaluating and with the evaluation methods. A 15-cm line scale was used, with zero representing no perceptible off-flavor and 15 representing extreme off-flavor. Panelists used the line scale by placing a perpendicular mark on the scale according to their perceptions of the intensities of off-flavor in each sample. The marks from the line scales were converted to numbers by measuring the distance of each mark from the left end of the scale. The panelists' data per treatment replicate were averaged, and the data were analyzed by using analysis of variance (ANOVA) and least significant difference (LSD), with statistical significance accepted at a level of $P \leq 0.05$ (12).

Synthesis and isolation of MND. The synthesis of MND was carried out by procedures reported elsewhere (13). Because the purity of synthesized MND was only 40%, it was isolated by preparative gas chromatography (GC) using a Varian 3700 Series Instrument (Palo Alto, CA). The effluent from a packed Alltech Silar 10 C column (1.83 m, 3.2 mm o.d., 2.2 mm i.d.) was split in a ratio of 1:8, with the lesser amount going to a flame-ionization detector (FID) and the greater amount going to a cold trap, which consisted of glass capillary tubing surrounded by dry ice. A 4% solution of MND in hexane was injected in 2-µL portions. The temperature was held 1 min at 80°C, programmed at 6°C/min to 144°C, and held at 144°C for 2 min. MND eluted at an oven temperature of about 144°C, at which time the cold trap was connected to collect the sample. At the end of each run, the oven temperature was set at 250°C for 20 min to volatilize any impurities that remained in the column.

When the desired amount of sample was collected from the gas chromatograph, the concentrated sample was further analyzed by GC (Hewlett-Packard 5890 series II)–mass spectrometry (MS; Hewlett-Packard 5970 series) on a Supelco SPB-1 fused-silica capillary column (Bellefonte, PA). The conditions were the same as just described for purification of MND.

Sensory evaluation of MND. Room temperature. The flavor intensity of MND was evaluated at room temperature in an oil/ water emulsion system by comparing its flavor with a blank and a series of 2-heptanone emulsion standards (11,14). Nine panelists were trained during three separate sessions, with blank emulsions and emulsions with different amounts of 2-heptanone and MND. After each training evaluation, the panelists were informed of the identity and concentration of each sample, and panelists were allowed to reevaluate the samples to improve their accuracy. During each actual sensory session, MND was prepared in mineral oil at concentrations of 28.4, 82.5, and 256 ppm by volume. Each determination of the flavor intensity of MND was the average of two replications and nine measurements from panelists and was calculated relative to the concentration (intensity) of 2-heptanone. The data were analyzed by standard ANOVA. Differences in mean values among data were determined by LSD at $P \leq 0.05$ (12).

After heating. During the course of the study, one theory we considered to explain the behavior of MND was that MND may be easily polymerized. The reasoning for this theory is explained in the Results and Discussion section. Therefore, to see whether MND could be depolymerized by heating, mineral oil with 9 ppm MND was divided into two parts. One part was heated in a boiling water bath for 5 min, and another part was not heated. Both mineral oil treatments were then diluted 100-fold with water and prepared into emulsions with 0.09 ppm MND by the procedures described above. The flavor intensity of the MND emulsions with and without heat treatment was evaluated by the sensory panel as just described.

Detection of MND during storage of commercial SBO. Commercial SBO (Wesson and Hy-Vee brands, 800 mL) were

deodorized at 220°C for 30 min under vacuum (0.005 mm Hg). Oil samples (27 g in 50-mL beaker) were stored at 35°C under 1937 lux of fluorescent light. Two beakers that contained replicate oil samples were taken from the oven every 2 to 4 d, and PV were measured twice per beaker by using the Stamm test as modified by Hamm *et al.* (10).

The detection of MND and other volatiles was conducted by headspace concentration, followed by GC–MS analysis (14). The amount of MND in SBO was calculated from the peak area of the gas chromatogram by the method of Lee *et al.* (14).

TABLE 2
Peroxide Values (meq/kg) of Soybean Oils with Low and High F Acid Content, Held at 35°C Under Fluorescent Light[a]

	PV (meq/kg)	
Storage (d)	Oil with low F acid content	Oil with high F acid content
0	0.1[a]	0.2[a]
2	1.4[a]	3.0[a]
3	2.6[a]	6.0[a]
4	4.9[a]	9.6[b]
5	10.1[a]	21.5[b]
6	19.2[a]	35.0[b]
7	37.8	—

[a]Values in a row with different roman superscripts were significantly different at $P \leq 0.05$; $n = 2$. For abbreviation see Table 1.

RESULTS AND DISCUSSION

Oxidative stability of SBO with high or low furanoid acid content. To have enough oil for the stability tests, the seeds from PI 398672 and PI 398955, both containing low amounts of the F acids, were mixed in approximately equal amounts before oil extraction, as noted earlier. The FAME and F acid contents of the oils with low and high F acid contents are shown in Table 1. Furanoid acid **I**, 10,13-epoxy-11-methyloctadeca-10,12-dienoic acid, identified in minute quantities by Guth and Grosch (3), was negligible so is not reported. F acid **II** was 10,13-epoxy-11,12-dimethyloctadeca-10,12-dienoic acid, and F acid **III** was 12,15-epoxy-13,14-dimethyleicosa-12,14-dienoic acid.

Table 2 shows the changes in PV with time as the oils were stored under fluorescent light at 35°C. An ANOVA showed that the PV of oil with high F acid content were significantly greater than those of the oil with low F acid content, and an LSD test showed that this difference occurred after 3 d of storage. Possibly, the greater linoleate, linolenate, and F acid content of the oil with high F acid content contributed to the increased PV.

There were no statistical interactions between varieties and PV, varieties and replicates, or replicates and PV, so the average value was used to calculate significant differences for the oils (Table 3). There were no significant differences in the flavor intensity between the oils with high and low F acid content. The oils with PV ~10 and ~35 meq/kg had significantly stronger off-flavors than the fresh oil, but there was no significant difference in the flavor intensity between the oils at PV values ~10 and ~35 meq/kg. These results do not support the belief that F acid oxidation is a major factor in the off-flavor development of SBO.

GC of MND. GC of synthetic MND on a SBP-1 (low polarity capillary column) resulted in two distinct and well-separated peaks joined by a bridge of poorly resolved components. Figure 1 shows examples of these peaks formed at several GC temperatures. The mass spectra of these two peaks and the intervening bridge were identical and similar to those reported for MND by Guth and Grosch (1), who also obtained a similar picture of poorly resolved peaks for MND when they chromatographed it on SE-30, SE-54, and OV-1701. Our MND preparation was estimated to be 40% pure, based on GC peak areas, and was purified further by GC on a packed Silar 10 C (polar) column. On this column, MND emerged as one peak, in agreement with the report of Guth and Grosch (1). A grassy, sweet aroma was noted as MND emerged. After purification on the Silar 10 C column, MND was estimated to be about 99% pure by peak area when rechromatographed on an SPB-1 column.

Sensory evaluation of MND. Aqueous mineral oil emulsions with 0.28, 0.85, and 2.56 ppm of MND had flavor intensities that were similar to each other and were rated by the panel as equal to those of 0.389, 0.323, and 0.379 ppm of 2-heptanone, respectively, in similar emulsions (Table 4). Dixon and Hammond (11) reported that emulsions of 0.25 ppm of 2-heptanone were near the flavor threshold, so intensities equal to 0.389 ppm are slightly above threshold.

In considering the GC evidence that MND exists in two or more forms and the intense odors obtained when MND was subjected to GC separations, it seemed possible that the flavor intensity of solutions of MND in nonpolar solvents, such as mineral oil, might become more intense when heated.

TABLE 1
Fatty Acid Methyl Ester and Furanoid Acid Contents

	Fatty acid composition by GLC (rel. %)					F acid content (mg/g FAME)		
Soybean oil	16:0	18:0	18:1	18:2	18:3	**II**[c]	**III**[d]	**II** + **III**
Low F acid[a]	11.6	3.7	21.9	54.1	8.6	0.154	0.104	0.258
High F acid[b]	11.4	4.1	15.8	57.9	10.7	0.370	0.281	0.651

[a]Soybean PI 398672 and PI 398955, ~1:1 mixture of seeds.
[b]Soybean PI 323276.
[c]F **II** = 10,13-epoxy-11,12-dimethyloctadeca-10,12-dienoic acid.
[d]F **III** = 12,5-epoxy-13,15-dimethyleicosa-12,14-dienoic acid. Abbreviations: GLC, gas–liquid chromatography; rel. %, relative percentage; F, furanoid fatty acid; FAME, fatty acid methyl ester.

TABLE 3
Sensory Evaluation Scores[a] of Soybean Oils with Low and High F Acid Content, Held at 35°C Under Fluorescent Light

	Low F acid content oil[b]	High F acid content oil[b]	Average[c]
Fresh, PV = 0.1[d]	4.7[a]	4.5[a]	4.6[x]
PV = 10	7.4[a]	6.8[a]	7.1[y]
PV = 35	8.1[a]	7.6[a]	7.8[y]
Average	6.7[a]	6.3[a]	

[a]1 = no perceptible off-flavor, 15 = extreme off-flavor.
[b]Values in a row with different roman superscripts were significantly different at $P \leq 0.05$; $n = 2$.
[c]Values in a column with different roman superscripts were significantly different at $P \leq 0.05$.
[d]PV, peroxide value (mg/kg). For other abbreviation see Table 1.

Therefore, a 9-ppm solution of MND in mineral oil was heated to 100°C before emulsification, dilution to 0.09 ppm with water, and evaluation at room temperature. The emulsions made from unheated and heated MND were rated as equivalent to 0.317 and 0.280 ppm emulsions of 2-heptanone, respectively (Table 4). Thus, heating to 100°C did not increase the flavor intensity perceptibly when emulsions were tasted at room temperature.

Statistical evaluation of the sensory results indicated that there was no significant difference in flavor between emulsions that contained 0.09 and 2.56 ppm MND at the probability level of $P \leq 0.05$ (Table 4). Based on these results, MND seemed to have a fairly high flavor threshold, and its flavor intensity increased almost imperceptibly with increased concentration. This lack of increase in flavor intensity with concentration is unique among the flavor compounds we have investigated by this technique. If 0.09 ppm is considered the threshold, this amount is comparable to that of 2-octenal and several similar aldehydes (11). But because the flavor intensity of MND remained about the same over a 28-fold dilution, it is not clear how much more it might have to be diluted before a true threshold was reached. Panelists indicated that, during sensory evaluation of the emulsion, they could smell the samples before tasting them, but that when tasted, the flavor intensity seemed less strong than expected. In general, these results do not support the belief that MND is an important flavor compound in SBO at room temperature.

Relation of flavor response to the properties of MND. An explanation for the unusual flavor response of MND may be that, when molecularly dispersed in air, MND has an intense odor, but when dispersed in an emulsion of SBO or mineral oil, MND may exist in a form with relatively low flavor intensity or be bound by the medium. Possibly, the sensory-potent form, favored by gaseous dispersions, is in equilibrium with the sensory-impotent form found in the emulsion so that, when the concentration of MND in the emulsion is increased, the equilibrium shifts to maintain a fairly constant concentration of the sensory-potent form. In this way, the flavor intensity of oil emulsions does not increase much with MND concentration.

It is not clear what relation may exist between the sensory-potent and -impotent forms of MND and the various peaks observed during GC on nonpolar columns. Guth and Grosch (1) believed, on the basis of nuclear magnetic resonance data, that the keto form of MND is favored but that, when dissolved in a nonpolar solvent or when the temperature was raised, the equilibrium shifts more to an enol form. The keto and enol forms are presumed to represent the two major peaks observed in the GC traces. The bridge between the two peaks may represent materials that have been in equilibrium between the two forms during the separation process and have migrated part of the time in each form.

Figure 1 indicates that the situation may be even more complex than Guth and Grosch suspected (1). Low-temperature chromatograms in Figure 1 show shoulders on the two main peaks that suggest that there may be a number of components in equilibrium with each other. These may represent the enol form located on both the 2- and 4-positions and various hydrogen-bonding associations.

Grosch *et al.* (1,2,15,16) have advocated identifying important flavor compounds by dilution to aroma threshold. In this method, a flavor concentrate is injected into a gas chro-

TABLE 4
Flavor Intensities of Different Concentrations of 3-Methylnonane-2,4-dione (MND) Emulsions Represented by the Intensities of 2-Heptanone[a]

Concentration of MND (ppm)	2.56	0.85	0.28	0.09	0.09H[b]
Intensity of MND represented by the concentration of 2-heptanone (ppm)[c]	0.379	0.323	0.389	0.317	0.280

[a]Means of two replications and nine panelists.
[b]H = heated in boiling water bath for 5 min.
[c]Values within row 2 were not significantly different ($P \leq 0.05$). Intensity scale is described in References 11 and 14 and in the text.

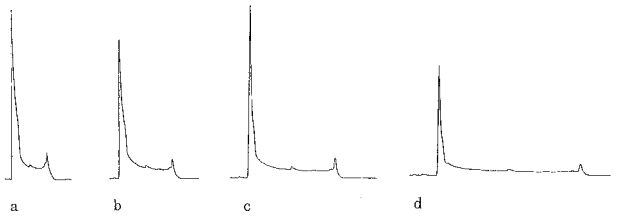

FIG. 1. The gas chromatograms of 3-methylnonane-2,4-dione (MND) on a Supelco SPB-1 capillary column at various isothermal conditions: a: 110°C; b: 100°C; c: 90°C; d: 80°C. The two major peaks likely represent the keto and enol forms, and the bridge between the two peaks may represent materials that have been in equilibrium between the two forms during separation.

TABLE 5
Concentration of MND and PV in Soybean Oils During Storage at 35°C Under Fluorescent Light

Brand name	Day								
	0	2	4	6	10	14	20	24	28
Wesson									
PV[a] (meq/kg)	0.1 ± 0	1.2 ± 0.1	3.4 ± 0.3	14.7 ± 0.1	40.9 ± 3.8	78.9 ± 2.3	—[b]	—	—
MND[c] (ppb)	0	0.038 ± 0.076	0.095 ± 0.09	0.097 ± 0.082	0.406 ± 0.268	0.253 ± 0.109	—	—	—
Hy-Vee									
PV[a] (meq/kg)	0.1 ± 0	1.0 ± 0.1	2.4 ± 0.4	2.9 ± 0.5	3.3 ± 0.4	4.7 ± 0.6	12.2 ± 1.4	14.6 ± 1.1	34.8 ± 5.3
MND[c] (ppb)	0	0	0	trace	trace	trace	0.044 ± 0.089	0.316 ± 0.234	0.804 ± 0.248

[a]PV ± standard deviation; $n = 2$.
[b]— : not measured.
[c]MND concentration ± standard deviation. For abbreviations see Tables 3 and 4.

matograph, and peaks with odors are noted. More and more dilute solutions are injected until the odors, one by one, disappear. The last ones to disappear are regarded as the most important. This method was used to demonstrate the importance of MND. Our results indicate that this technique should be applied with care because MND does not seem to be an important flavor compound in oil at room temperature despite its odor intensity when smelled at gas chromatograph outlets.

Detection of MND and PV during storage of commercial SBO. To relate the flavor intensity of MND to realistic amounts of MND formed in oxidizing oils, two commercial oils were oxidized and evaluated. The PV and the amounts of MND of the commercial SBO stored at 35°C under light are shown in Table 5. PV development over time followed a typical lipid oxidation curve. There was a small peak at the same retention time as the standard MND, but this identification could not be confirmed by MS because of its small quantity when volatiles were collected at 50°C for 30 min. But when the volatiles were collected in a 75°C water bath for 60 min, the peak with the same retention time as MND showed the same MS pattern as MND. The concentrations of MND in SBO generally increased as the PV increased, although there was some variability, especially in Wesson brand SBO stored for 14 d. The MND concentrations in SBO at various PV were between 0 and 0.804 ppb, which were far below those tested in mineral oil emulsions during sensory evaluation, and below published odor threshold levels in SBO (1). Thus, MND could not strongly contribute to the reversion flavor of SBO.

ACKNOWLEDGMENT

We gratefully acknowledge the Iowa Soybean Promotion Board for partial financial support.

REFERENCES

1. Guth, H., and W. Grosch, 3-Methylnonane-2,4-dione. An Intense Odour Compound Formed During Flavour Reversion of Soya-bean Oil, *Fat Sci. Technol. 91*:225–230 (1989).
2. Guth, H., and W. Grosch, Comparison of Stored Soya-Bean and Rapeseed Oils by Aroma Extract Dilution Analysis, *Lebensm. Wiss. Technol. 23*:59–65 (1990).
3. Guth, H., and W. Grosch, Detection of Furanoid Fatty Acids in Soya-Bean Oil—Cause for the Light-Induced Off-Flavor, *Fat Sci. Technol. 93*:249–255 (1991).
4. Hannemann, K., V. Puchta, E. Simon, H. Ziegler, G. Ziegler, and G. Spitell, The Common Occurrence of Furan Fatty Acids in Plants, *Lipids 24*:296–298 (1989).
5. Hammond, E.G., in *Modern Methods of Plant Analysis*, New Series, edited by H.F. Linskins and J.F. Jackson, Springer-Verlag, Berlin, 1991, Vol. 12, p. 321.
6. Wu, X., E.G. Hammond, P.J. White, and W. Fehr, Analysis of Furanoid Esters in Soybean Oil and the Effect of Variety and Environment on Furanoid Ester Content, *J. Am. Oil Chem. Soc. 74*:1099–1103 (1997).
7. *Official and Recommended Practices of the American Oil Chemists' Society*, 4th edn., edited by D. Firestone, American Oil Chemists' Society, Champaign, 1989.
8. Stone, R.R., and E.G. Hammond, An Emulsion Method for the Sensory Evaluation of Edible Oils, *J. Am. Oil Chem. Soc. 60*:1277–1281 (1983).
9. Moulton, K.J., Laboratory Deodorization of Vegetable Oil, *Ibid. 66*:302–308 (1989).
10. Hamm, D.L., E.G. Hammond, V. Parvanah, and H.E. Snyder, The Determination of Peroxides by the Stamm Method, *Ibid. 42*:920–922 (1965).
11. Dixon, M.D., and E.G. Hammond, The Flavor Intensity of Some Carbonyl Compounds Important in Oxidized Fats, *Ibid. 61*: 1452–1456 (1984).
12. *SAS® User's Guide: Statistic*, SAS Institute Inc., Cary, 1989.
13. Becker, H., *et al., Organicum: Practical Handbook of Organic Chemistry*, edited by P.A. Ongley, Addison-Wesley, 1973, pp. 490–499.
14. Lee, I., S.H. Fatemi, E.G. Hammond, and P.J. White, Quantitation of Flavor Volatiles in Oxidized Soybean Oil by Dynamic Headspace Analysis, *J. Am. Oil Chem. Soc. 72*:539–546 (1995).
15. Ullrich, F., and W. Grosch, Identification of the Most Intense Volatile Flavour Compounds Formed During Autoxidation of Linoleic Acid, *Z. Lebensm.-Unters. U.-Forsch. 184*:277–282 (1987).
16. Ullrich, F., and W. Grosch, Flavor Deterioration of Soya-bean Oil: Identification of Intense Odour Compounds Formed During Flavour Reversion, *Fat Sci. Technol. 90*:332–336 (1988).

[Received June 19, 1997; accepted February 9, 1998]