Eur Food Res Technol (2004) 219:190–198
DOI 10.1007/s00217-004-0933-2

ORIGINAL PAPER

A. Kanavouras · P. Hernandez-Münoz ·
F. A. Coutelieris

# Shelf life predictions for packaged olive oil using flavor compounds as markers

Received: 28 January 2004 / Revised: 7 April 2004 / Published online: 18 May 2004


**Abstract** The experimentally-derived amounts of five selected flavor compounds, namely hexenal, 2-pentyl furan, (*E*)-2-heptenal, nonanal, and (*E*)-2-decenal, all produced during the oxidation of extra virgin olive oil packaged in various storage conditions (glass/PET/PVC bottles; 15/30/40 °C temperature; light or dark conditions) for one year, were used in a mathematical model for calculating the probability that the olive oil would not have reached the end of its shelf life ($P_{safe}$) after a certain storage period time. The storage times corresponding to probabilities of 70%, 50% and 30% were also calculated. On the basis of these results, an optimal group of flavor compounds were selected that were highly correlated to the degradation factors (storage conditions), and therefore the $P_{safe}$, of the oil. These flavor compounds could then be used as markers to identify the cause of the oxidative degradation (the "storage history") of the olive oil.

**Keywords** Oxidation · Olive oil · Packaging · Storage conditions · Shelf life prediction · Modeling · Off-flavors

## Introduction

Proper packaging of olive oil is necessary to ensure an adequate shelf life for distribution and sales. Physical characteristics of the packaging material may significantly affect the final quality of the oil, depending on the extent of the deteriorative interactions [1]. Oil placed in bottles with high air permeability should be sold within a few months to avoid appreciable quality loss, while the use of transparent materials for bottling olive oil leads to photo-oxidation of the product and to a significant reduction in its shelf life [2, 3, 4, 5].

The flavor profile of the oil and its sensory notes have been broadly related to the quality of the product. Changes in the olive oil's unique flavor during oxidation can be directly related to the decomposition of the hydroperoxides formed and the consequent formation of novel volatile compounds [6]; this may aid our understanding of the oxidative alterations of the lipids [7]. The volatile aldehydes and vinyl ketones are mainly responsible for potent off-flavors, because their odor threshold levels are very low. Other volatile oxidation products such as furan derivatives, vinyl alcohols, ketones, alcohols, alkynes, and short-chain fatty acids also contribute to the formation of undesirable flavor notes to a varying extent [8]. Kanavouras et al [9] separated and identified flavor compounds from differently preserved olive oil samples. Based on the abundance and evolution of individual flavor compounds, it was suggested that hexanal, 2-pentyl furan, (*E*)-2-heptenal, nonanal, and (*E*)-2-decenal were the compounds that most clearly described the oxidation. The researchers also concluded that it was mainly the presence of fluorescent light, followed by elevated temperature, that stimulated the oxidative alterations in the oil.

In a search for quick, easy to use, and accurate methods requiring minimal amounts of experimental work, a generalized approach to describing oxidation of olive oil packaged in bottles of various shapes and materials was presented by Del Nobile et al [10, 11]. These works did not include any refinement in terms of storage conditions (temperature and light) while they were also limited to hydroperoxides evolution without any reference to the flavor compounds evolved. Dekker et al [12] presented a model to predict the development of hydroperoxides as a function of both time and location in the package, based

A. Kanavouras (✉)
Spreads and Cooking Products Category,
Unilever-Bestfoods Europe,
Nassaukade 3, 3071 JL Rotterdam, The Netherlands
e-mail: antonis.kanavouras@unilever.com
Tel.: +31-1043-93919
Fax: +31-1043-94658

P. Hernandez-Münoz
Departamento de Tecnología de Alimentos,
Universidad Politecnica de Valencia,
Camino de Vera s/n, Valencia, Spain

F. A. Coutelieris
Unilever-Bestfoods Research and Development,
Oliver van Noortlaan, 3130 AT Vlaardingen, The Netherlands

on the reaction kinetics of the food and its active ingredients, the film permeability, and the mass transfer rate within the product in the presence or absence of oxygen absorbers. A shelf life estimation study by Kanavouras et al [9] for olive oil packaged in different packaging materials and stored under different combinations of temperatures and availability of light, resulted in the estimation of the production rate for various oxidation derived off-flavor compounds, as well as the equilibrium oxidation reaction constants. However, the mass transport of the compounds due to diffusion was not incorporated into the model presented in that study. Consequently, Coutelieris and Kanavouras [13] introduced a predictive mathematical model to describe the mass transport to and from the oil phase through various packaging materials for several temperature and light availability storage conditions. Based on validated simulations, in a following work [14], the researchers summarized the qualitative changes of packaged olive oil stored at various conditions for prolonged periods of time.

The goal of this study was to select a minimum number of flavor compounds whose evolution could be closely related to the factors that deteriorate olive oil during storage, namely light, temperature and oxygen. To achieve this, the present study applied the model previously developed by Coutelieris and Kanavouras [13] for the selected characteristic flavor compounds evolved in extra virgin olive oil when packaged in various packaging materials and stored under a wide range of storage conditions for a one year period. The probability that the oil would not reach the end of its shelf life during the storage period was also used as a shelf life prediction tool.

## Materials and methods

Portuguese organic extra virgin olive oil was packed under nitrogen gas, without headspace, in cleaned and dried 500 mL PET drinking water bottles, in 500 mL PVC bottles (Novapack, Co. Paris, IL, USA), and in 500 mL glass bottles (Fisher Scientific Co., New Jersey, USA). The bottles were tightly sealed with standard polypropylene threaded caps. Half of the bottles were covered with aluminum foil and placed inside fiberboard boxes while the other half were exposed to fluorescent light. Filled bottles were stored in controlled environment chambers at 15, 30 or 40 °C and 60% RH. During the experiment, four 40 W fluorescent light bulbs were placed 30 cm above the bottles. Weekly rearrangement of the bottles was performed to ensure uniform exposure to light. Two bottles per treatment were analyzed monthly in triplicate up to 12 months. An automatically operating stripping apparatus (Dynatherm 1000, Dynatherm Analytical Instruments Inc., Kelton, PA) was used to strip volatile compounds out of the oil, kept at 37 °C, into a Tenax-TA trap (Supelco, Bellefonte, PA). Compounds were desorbed using a desorption unit (Model 890 from Dynatherm Analytical Instruments Inc. Kelton, PA) connected to a gas chromatography apparatus (Hewlett Packard 5890 Series II, Hewlett Packard, Philadelphia, PA) with a 30 m×0.32 mm ID×0.25 $\mu$m film thickness, fused silica capillary column (SPB-5, Supelco, Bellefonte, PA). The temperature program was: initial temperature, 35 °C for 5 min, increased to 80 °C at a rate of 3 °C/min, held for 1 min, then increased to 180 °C at 10 °C/min, held for 1 min, and finally increased to 220 °C at 4 °C/min where it was held for 10 min. The carrier gas was maintained at a flow rate of 1.75 mL/min at 40 °C. Identification of compounds was performed with a

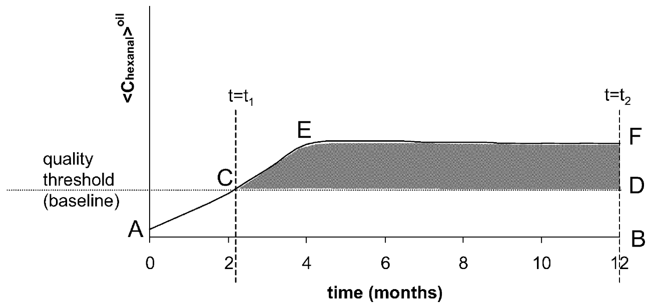


**Fig. 1** Graphical representation of probability $P_{safe}$

Varian 2000 mass spectrometer (Varian, TX, USA) interfaced with the Dynatherm desorption unit. The tuning value for the ITMS was 100, using cedrol as the tuning standard. Other parameters were: tune sensitivity, 9000; acquisition parameters: full scan, scan range: 41–300 amu, scan time: 1.0 s, threshold: 1 count, multiplier from 1500–2300 V depending on multiplier conditions; transfer line temperature, 240 °C; exit nozzle 240 °C; manifold 240 °C. In addition, the standard compounds of hexanal, 2-pentyl furan, (*E*)-2-heptenal, nonanal and (*E*)-2-decenal (Sigma-Aldrich, St. Louis, MO, USA) were injected in the GC for a further verification of the identified volatiles.

Based on the experimentally measured amounts of each compound, the probability of the olive oil reaching the end of its shelf life by a particular elapsed time is analogous to the ratio of the areas below and above an arbitrarily defined quality threshold. According to the graphical representation of this concept (see Fig. 1), the probability of the oil reaching the end of its shelf-life during the time period $[t_1,t_2]$ is analogous to the ratio of the surfaces defined by the curves CDFEC and ABFEA. The upper limit for the quality acceptance (the quality threshold) is defined as a particular concentration of one of the oxidation-related flavor compounds, C. If we take C to be hexanal, then since the above-mentioned areas can be expressed as integrals of the spatially averaged hexanal concentration, we can now define the probability, $P_{\text{safe}}$, of the oil not reaching the end of its shelf life during the time period $[t_1,t_2]$, as:

$$P_{\text{safe}} = 1 - \frac{\int_{t_1}^{t_2} \langle C_{\text{hexanal}} \rangle (t) dt}{\int_{0}^{t_2} \langle C_{\text{hexanal}} \rangle (t) dt} \tag{1}$$

where the brackets denote spatial averaging, $t_1$ is the time when $C$ (hexanal in this case) reaches the critical concentration level, and the upper edge of the integrals, $t_2$, could be any elapsed time period. In this study $t_2$=12 months. In general, $P_{\text{safe}}$ is an easily estimated quality indicator, depending on the evolution history of the compound in question through a single value that allows an extensive analysis of experimental data and easy-to-make comparisons. It will be employed later in this study to analyse the results.

## Results and discussion

The initial statistical analysis, performed using the amounts of flavor compounds isolated from the olive oil samples, showed that the individual components with the most significant changes were: hexanal, 2-pentyl furan, (*E*)-2-heptenal, nonanal, and (*E*)-2-decenal. Their concentrations at each sampling time were used to derive the $P_{\text{safe}}$ of the packaged olive oil, for each specific set of storage conditions – combinations of three packaging

**Table 1** Relative area response, obtained by gas chromatography, for the selected flavor compounds in extra virgin olive oil at the time of bottling and after 12 months of storage

| Packaging | Flavor compound | Initial amount | Amount after 12 months | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | 15 °C, light | 15 °C, dark | 30 °C, light | 30 °C, dark | 40 °C, light | 40 °C, dark |
| Glass | Hexanal | 1102970 | 3916998 | 1161028 | 4719663 | 2091647 | 5637828 | 4630000 |
| | 2-Pentyl furan | 27088 | 50968 | 22465 | 59003 | 36236 | 103875 | 52000 |
| | (*E*)-2-Heptenal | 18746 | 29850 | 24800 | 29541 | 24421 | 27853 | 17744 |
| | Nonanal | 52492 | 185822 | 52349 | 194433 | 73043 | 177820 | 110000 |
| | (*E*)-2-Decenal | 8003 | 21983 | 21435 | 28300 | 27163 | 28449 | 30403 |
| PET | Hexanal | 1102970 | 2703657 | 982667 | 3764985 | 1656607 | 6608820 | 3040000 |
| | 2-Pentyl furan | 27088 | 31134 | 21506 | 80469 | 31298 | 86866 | 61091 |
| | (*E*)-2-Heptenal | 18746 | 25468 | 21506 | 27315 | 31500 | 86866 | 21139 |
| | Nonanal | 52492 | 169000 | 49408 | 226000 | 60125 | 200942 | 85225 |
| | (*E*)-2-Decenal | 8003 | 17400 | 21897 | 13286 | 20147 | 38100 | 18500 |
| PVC | Hexanal | 1102970 | 3233641 | 956888 | 3639919 | 2088757 | 4975994 | 3004558 |
| | 2-Pentyl furan | 27088 | 76594 | 21181 | 21014 | 54547 | 89634 | 72064 |
| | (*E*)-2-Heptenal | 18746 | 25510 | 21077 | 65938 | 26996 | 59459 | 23005 |
| | Nonanal | 52492 | 198824 | 49901 | 260000 | 97137 | 226000 | 99401 |
| | (*E*)-2-Decenal | 8003 | 23340 | 19716 | 14600 | 35527 | 17100 | 21806 |

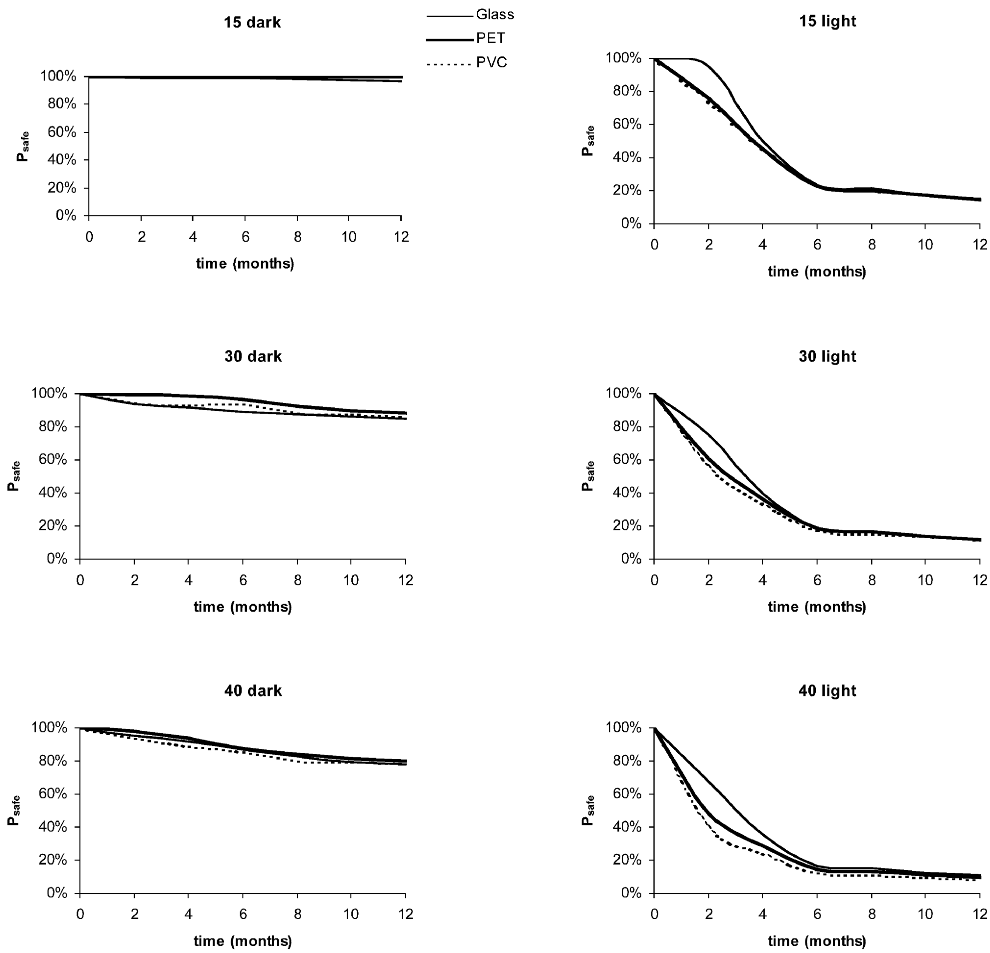


**Fig. 2** Relationship of $P_{safe}$ to storage time for olive oil packaged in various materials and storage conditions. Calculation of $P_{safe}$ is based on hexanal content

materials (glass, PET and PVC), three storage temperatures (15, 30 and 40 °C), and the light availability (light, dark). Table 1 shows the amount of each of the aforementioned flavor compounds (in GC area response) in extra virgin olive oil at the time of bottling and at the end of the 12 month storage period, in each of the packaging materials and for each of the storage conditions. At the time of bottling, the flavor compounds inside the olive oil are indicative of the initial olive oil quality. Overall, the concentrations of these flavor compounds were increasing during storage, so the values recorded after 12 months of storage can be considered as indicative of the previous increment. Nevertheless, for the following

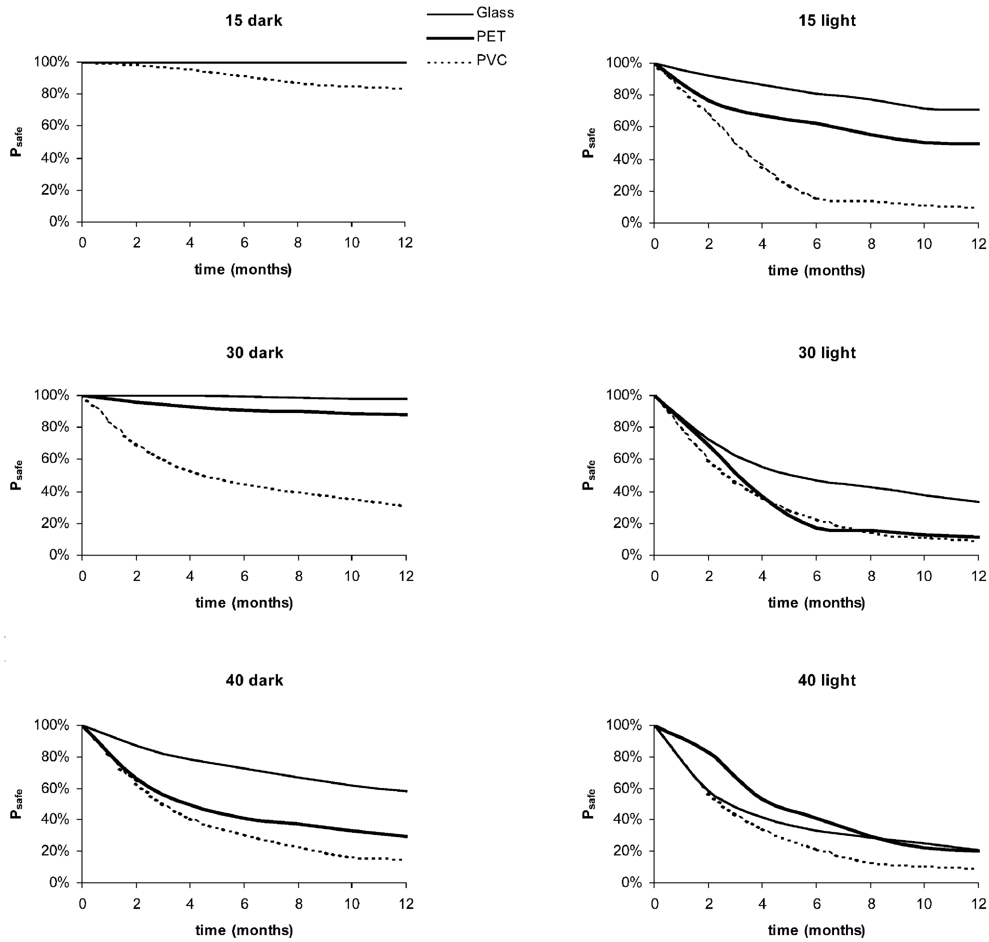


**Fig. 3** Relationship of $P_{safe}$ to storage time for olive oil packaged in various materials and storage conditions. Calculation of $P_{safe}$ is based on 2-pentyl furan content

calculations of the $P_{\mathrm{safe}}$ values, all of the recorded amounts data points were used.

Figures 2, 3, 4, 5, and 6 present $P_{\mathrm{safe}}$ as a function of time for hexanal, (Fig. 2), 2-pentyl furan (Fig. 3), (*E*)-2-heptenal (Fig. 4), nonanal (Fig. 5) and (*E*)-2-decenal (Fig. 6). The time evolution of $P_{\mathrm{safe}}$ for the selected compounds could be used to further demonstrate the influence of the various storage conditions and packaging materials on the shelf life of the packaged olive oil.

The values of $P_{\mathrm{safe}}$ for hexanal, nonanal, (*E*)-2-heptenal, and (*E*)-2-decenal showed a rather sharp decrease when the oil was stored in the light for 12 months, indicating the strong effect of light on the oil. Under dark conditions hexanal and nonanal showed similar $P_{\mathrm{safe}}$ values at low temperatures, namely 15 and 30 °C. At 40 °C, nonanal was influenced by the type of packaging material, indicating a strong oxygen synergistic effect. The further decrease in the $P_{\mathrm{safe}}$ values based on the nonanal concentrations at high temperatures may be related to the additional oleic acid auto-oxidation that takes place in the presence of oxygen, contributing to an additional 23% increase in the amount of the compound [8]. Morales et al [15, 16] showed that at elevated temperatures the ratio of hexanal/nonanal might be indicative of the oxidation level of the olive oil, which may also correlate with the differences in the two compounds shown for the olive oil stored at 40 °C.

The $P_{\mathrm{safe}}$ for olive oil stored in glass and placed in the dark for 12 months, as calculated from the evolution of (*E*)-2-heptenal, did not seem to differ between any of the storage temperatures. On the other hand, a synergistic effect of temperature and light was rather obvious, as the $P_{\mathrm{safe}}$ was clearly lower for olive oil stored in the light. In the presence of oxygen permeating the polymeric packaging materials, the $P_{\mathrm{safe}}$ was always lower than for glass and reduced values were recorded when olive oil was stored in the light. Temperature had a great effect on the reduction of $P_{\mathrm{safe}}$, especially when light was involved.

The $P_{\mathrm{safe}}$ for olive oil based on the amounts of 2-pentyl furan during storage indicated a similarly high correlation of the level of the compound in the olive oil to the availability of light. When olive oil was stored in the dark and at high temperatures, the $P_{\mathrm{safe}}$ values were significantly lower for olive oil packed in PET, while for olive oil in PVC they were not that different between 30 and 40 °C. For olive oil packaged in material impermeable to oxygen, in this case glass, the $P_{\mathrm{safe}}$ values for olive oil, as calculated based on the evolution of 2-pentyl furan, were clearly higher than those for the polymeric – oxygen permeable – packaging materials at 15 and 30 °C and in the light, while when the oil was kept at 40 °C, the

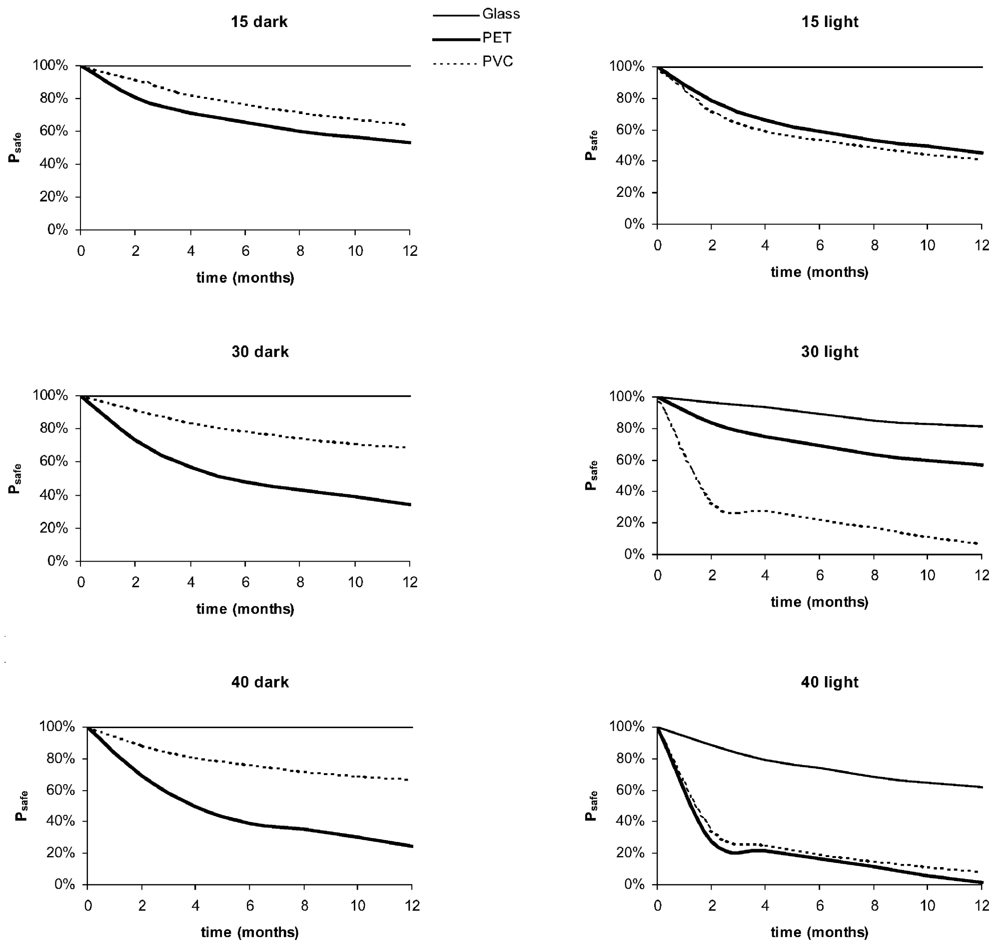


**Fig. 4** Relationship of $P_{safe}$ to storage time for olive oil packaged in various materials and storage conditions. Calculation of $P_{safe}$ is based on (*E*)-2-heptenal content

evolution of $P_{\mathrm{safe}}$ was similar for all of the packaging materials, indicating that temperature is a stronger factor than oxygen in the production of 2-pentyl furan. When olive oil was stored at dark and high temperatures (40 °C), obvious differences could also be reported between permeable and impermeable packaging materials, denoting the light-temperature synergism. The above results can be explained by the proposed 2-pentyl furan formation from 4-ketononanal, an oxidized linoleic acid 10-OOH derivative. Though not a typical hydroperoxide, 10-OOH can derive from singlet oxidation of linoleic acid. Another possible pathway may come from 9-OOH of linoleic acid in the presence of singlet oxygen combined with a liberation of formaldehyde, while its formation from linolenic acid has also been suggested [8]. Therefore, due to the demand for singlet oxygen, the presence of 2-pentyl furan should be more closely related to photo-oxidation. According to Chan et al [17], the presence of 2-pentyl furan deriving from oxidized linoleic acid can be related to the oxidation level of oils containing high amounts of linoleic acid. Frankel [18] proposed the formation of 2-pentyl furan through the degradation of 9-OOH to form a cyclic peroxide and further pentyl furaldehyde, which can be decomposed to 2-pentyl furan and formaldehyde. In general, the higher the temperature of storage, the lower the relative $P_{\mathrm{safe}}$ values recorded, independent of the other storage parameters.

Besides the slight distinction between packaging materials related to the storage temperature increase, the $P_{\mathrm{safe}}$ values from (*E*)-2-decenal did not provide any conclusive evidence about the effect of light on the production of that compound. Therefore, although there is an abundance of (*E*)-2-decenal in packaged olive oil, it probably cannot be considered to be a compound useful for differentiating storage conditions.

Differences in the $P_{\mathrm{safe}}$ values were also recorded for the two polymeric packaging materials. In particular, the $P_{\mathrm{safe}}$ values for the oil stored in PET and PVC, based on either hexanal and nonanal amounts at any storage conditions, did not show any substantial differences, while for (*E*)-2-heptenal and 2-pentyl furan the $P_{\mathrm{safe}}$ strongly depended on the specific packaging material, in addition to the high temperature and light conditions. The $P_{\mathrm{safe}}$ values from 2-pentyl furan were always lower for olive oil stored in PVC in either light or dark conditions. The $P_{\mathrm{safe}}$ values calculated from (*E*)-2-heptenal were almost the same for the olive oil stored in PVC and PET packages in the light, while PVC appears to retard the development of (*E*)-2-heptenal inside the olive oil stored in the dark.

Based on the results presented above, and in order to estimate the influence of each of these compounds on the

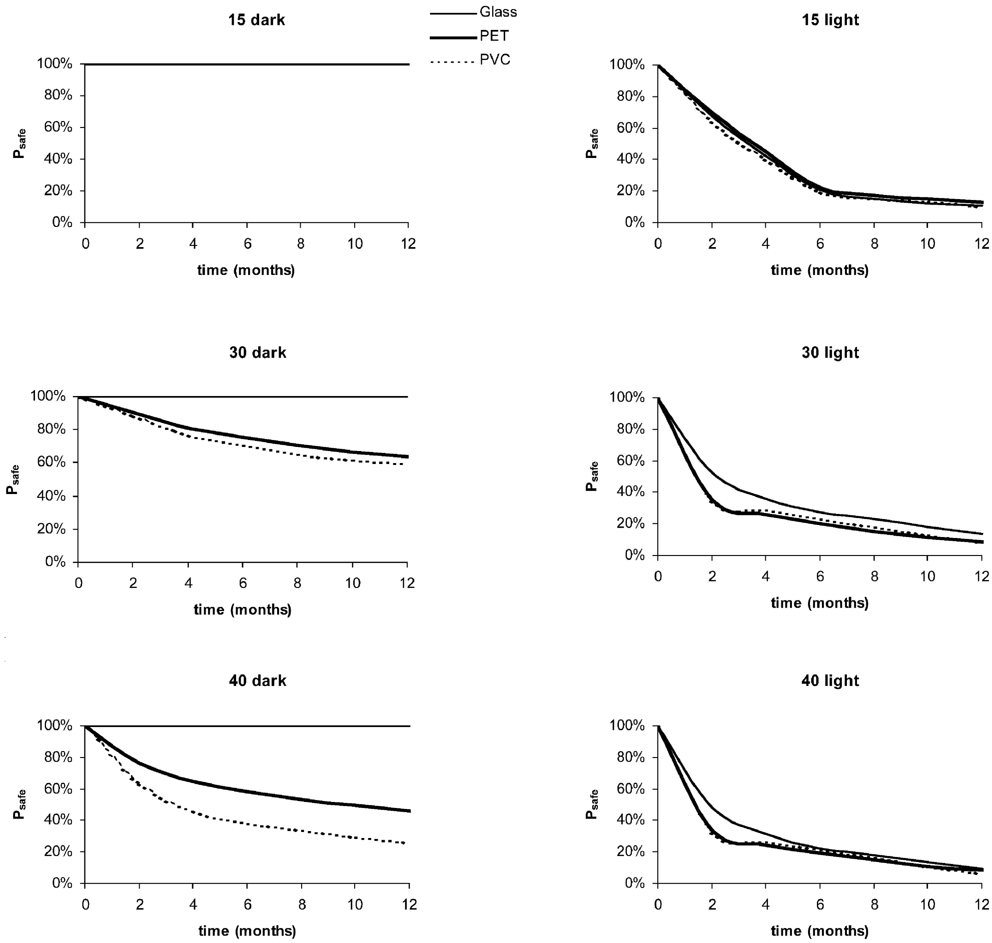


**Fig. 5** Relationship of $P_{safe}$ to storage time for olive oil packaged in various materials and storage conditions. Calculation of $P_{safe}$ is based on nonanal content

shelf life of packaged olive oil in each of the storage conditions, the critical times (in months) after which the oil has 30%, 50% and 70% of $P_{\text{safe}}$, are presented in Table 2. These $P_{\text{safe}}$ values were arbitrarily selected to demonstrate the expected shelf life. They were selected as representatives of three quite characteristic levels of quality loss tolerance for commercially available packaged olive oil, and they can be adjusted based on the quality standards for each individual product. The general trends for the shelf life presented in Table 2, show a reduction in the critical time as the temperature of storage and the amount of light were increased, for all compounds and packaging materials.

In order to identify the compounds that could be employed as quality indicators for the packaged olive oil, a process for identifying simple selection criteria was investigated, taking into consideration the previously presented analysis for the $P_{\text{safe}}$ values. Table 3 shows $P_{\text{safe}}$ at 12 months only, since the $P_{\text{safe}}$ value at the end of the 12 months of storage was the minimum achieved for each storage condition based on the evolution of each individual flavor compound, and so it is representative of the $P_{\text{safe}}$ evolution during the storage period.

The criteria employed for the selection of the quality indicators were:

- The level of $P_{\text{safe}}$ at 12 months: the lower the $P_{\text{safe}}$ value became, the higher the variation of this compound in the oil over time, so the more significant the effect of storage conditions on the oil. Such a compound can be used as an oxidation-describing agent.
- The relative variations in levels of the compounds within the same storage conditions: highly significant differences recorded for a particular compound merited its selection, while a group of compounds were selected as markers when such differences were not present.

The storage conditions selected in this study were based on three major contributors to the oxidative degradations inside the packaged olive oil: temperature, availability of light, and presence of oxygen. These factors were further classified into groups to allow for an additional investigation of the effect of each one on the oil. Therefore, two main groups based on the oxygen availability (oxygen/no oxygen) were formed, each one including all of the possible combinations of low or high temperature and light or dark conditions. A summary of the compounds for each group of conditions is presented in Table 4. The information in Table 4 can be employed when flavor analysis is used during a quality evaluation of packaged olive oil. The application of the $P_{\text{safe}}$ concept

**Table 2** Storage time (in months) corresponding to 30%, 50% and 70% of $P_{safe}$ for packaged olive oil, derived for specific flavor compounds

| Flavor compounds | Material, T °C | $P_{safe}$, dark | | | $P_{safe}$, light | | |
|---|---|---|---|---|---|---|---|
| | | 30% | 50% | 70% | 30% | 50% | 70% |
| Hexanal | Glass, 15 | >12 | >12 | >12 | 4–6 | 2–4 | 2–4 |
| | PET, 15 | >12 | >12 | >12 | 4–6 | 2–4 | 0–2 |
| | PVC, 15 | >12 | >12 | >12 | 4–6 | 2–4 | 2–4 |
| | Glass, 30 | >12 | >12 | >12 | 4–6 | 2–4 | 2–4 |
| | PET, 30 | >12 | >12 | >12 | 4–6 | 2–4 | 0–2 |
| | PVC, 30 | >12 | >12 | >12 | 4–6 | 2–4 | 0–2 |
| | Glass, 40 | >12 | >12 | >12 | 4–6 | 2–4 | 0–2 |
| | PET, 40 | >12 | >12 | >12 | 2–4 | 0–2 | 0–2 |
| | PVC, 40 | >12 | >12 | >12 | 2–4 | 0–2 | 0–2 |
| 2-Pentyl furan | Glass, 15 | N.A. | N.A | N.A. | >12 | >12 | 10–12 |
| | PET, 15 | N.A. | N.A. | N.A. | >12 | 0–2 | 2–4 |
| | PVC, 15 | >12 | >12 | >12 | 4–6 | 2–4 | 0–2 |
| | Glass, 30 | >12 | >12 | >12 | >12 | 6–8 | 2–4 |
| | PET, 30 | >12 | >12 | >12 | 4–6 | 2–4 | 0–2 |
| | PVC, 30 | 10–12 | 4–6 | 2–4 | 4–6 | 2–4 | 0–2 |
| | Glass, 40 | >12 | >12 | 6–8 | 6–8 | 2–4 | 0–2 |
| | PET, 40 | 10–12 | 4–6 | 0–2 | 6–8 | 4–6 | 2–4 |
| | PVC, 40 | 6–8 | 2–4 | 0–2 | 4–6 | 2–4 | 0–2 |
| (*E*)-2-Heptenal | Glass, 15 | N.A. | N.A. | N.A. | N.A | N.A | N.A |
| | PET, 15 | >12 | >12 | 4–6 | >12 | 8–10 | 2–4 |
| | PVC, 15 | >12 | >12 | 8–10 | >12 | 8–10 | 0–2 |
| | Glass, 30 | N.A. | N.A. | N.A. | >12 | >12 | >12 |
| | PET, 30 | >12 | 4–6 | 2–4 | 5–6 | >12 | >12 |
| | PVC, 30 | >12 | >12 | 10–12 | 0–2 | 0–2 | 2–4 |
| | Glass, 40 | N.A. | N.A. | N.A. | N.A | N.A | N.A |
| | PET, 40 | >12 | N.A. | N.A | 0–2 | 0–2 | 0–2 |
| | PVC, 40 | >12 | >12 | 8–10 | 2–4 | 0–2 | 0–2 |
| Nonanal | Glass, 15 | >12 | >12 | >12 | 4–6 | 2–4 | 0–2 |
| | PET, 15 | N.A | N.A | N.A | 4–6 | 2–4 | 0–2 |
| | PVC, 15 | N.A | N.A | N.A | 4–6 | 2–4 | 0–2 |
| | Glass, 30 | N.A | N.A | N.A | 4–6 | 2–4 | 0–2 |
| | PET, 30 | >12 | >12 | 6–8 | 2–4 | 0–2 | 0–2 |
| | PVC, 30 | >12 | >12 | 6–8 | 2–4 | 0–2 | 0–2 |
| | Glass, 40 | N.A | N.A | N.A | 4–6 | 0–2 | 0–2 |
| | PET, 40 | >12 | 8–10 | 2–4 | 2–4 | 0–2 | 0–2 |
| | PVC, 40 | 0–2 | 2–4 | 0–2 | 2–4 | 0–2 | 0–2 |
| (*E*)-2-Decenal | Glass, 15 | 0–2 | 0–2 | 0–2 | 2–4 | 0–2 | 0–2 |
| | PET, 15 | 2–4 | 0–2 | 0–2 | 2–4 | 0–2 | 0–2 |
| | PVC, 15 | 4–6 | 2–4 | 0–2 | 4–6 | 0–2 | 0–2 |
| | Glass, 30 | 4–6 | 2–4 | 2–4 | 2–4 | 0–2 | 0–2 |
| | PET, 30 | 0–2 | 0–2 | 0–2 | 6–8 | 2–4 | 0–2 |
| | PVC, 30 | 4–6 | 2–4 | 0–2 | 6–8 | 2–4 | 0–2 |
| | Glass, 40 | 2–4 | 0–2 | 0–2 | 8–10 | 4–6 | 0–2 |
| | PET, 40 | 4–6 | 2–4 | 0–2 | 0–2 | 0–2 | 0–2 |
| | PVC, 40 | 8–10 | 4–6 | 0–2 | 6–8 | 2–4 | 0–2 |

N.A. indicates that $P_{safe}$ could not be calculated because the concentration of the compound exhibited non-monotonic behavior at the respective conditions

with the marker compounds suggested may provide a rough estimation of the "storage history" of the product.

## Conclusions

The flavor compounds produced during the oxidation of packaged olive oil were experimentally evaluated in this study, resulting in an extensive data-set of the evolution of flavor profiles. The characteristically different responses of selected compounds under different storage conditions contributed to the suggestion that they may be used to find the probability that the oil will not reach the end of its shelf-life ($P_{safe}$). The introduction of this probability allowed us to create a set of selection criteria that could be used to relate the levels of particular flavor compounds to storage factors that influence the deterioration of the oil. A suggested correlation outline was presented to describe the "storage history" of the product and identify the oxidation-promoting factor, using an optimal number of flavor compounds. Therefore, an accurate and fast evaluation of the quality level of stored olive oil, and a subsequent estimation of its shelf life, could be achieved. This quick evaluation of the oxidation level of packaged olive oil has potential applications to other oxidation sensitive packaged food components.

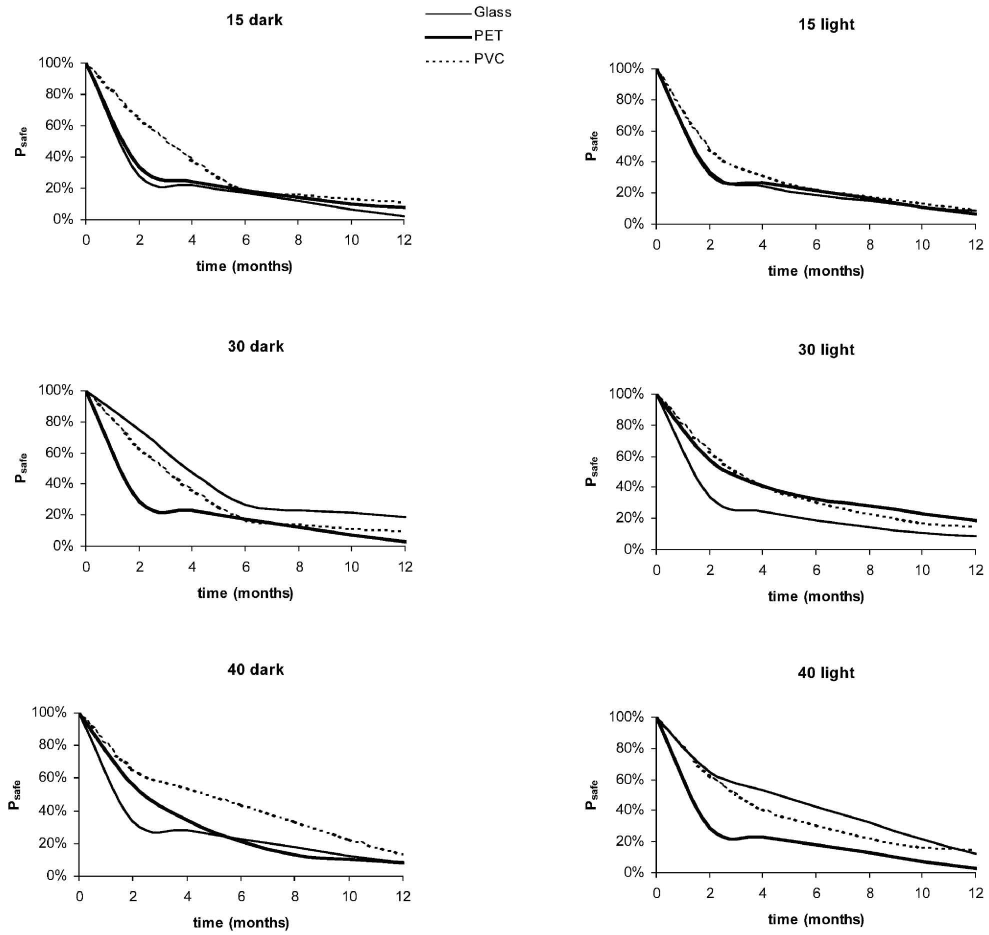


**Fig. 6** Relationship of $P_{safe}$ to storage time for olive oil packaged in various materials and storage conditions. Calculation of $P_{safe}$ is based on (*E*)-2-decenal content

**Table 3** $P_{\text{safe}}$ after 12 months of storage for packaged olive oil, derived using specific flavor compounds

| Storage conditions (packaging / temp °C / light or dark) | Hexanal | 2-Pentyl furan | (*E*)-2-Heptenal | Nonanal | (*E*)-2-Decenal |
|---|---|---|---|---|---|
| Glass/15/light | 15.22% | 70.78% | 99.99% | 11.00% | 8.54% |
| PET/15/light | 14.69% | 49.52% | 45.54% | 12.97% | 6.43% |
| PVC/15/light | 15.28% | 9.98% | 41.93% | 10.41% | 9.11% |
| Glass/15/dark | 96.68% | 99.99% | 99.99% | 99.99% | 2.25% |
| PET/15/dark | 99.99% | 99.99% | 53.28% | 99.99% | 7.96% |
| PVC/15/dark | 99.99% | 83.53% | 64.36% | 99.99% | 11.47% |
| Glass/30/light | 12.03% | 33.27% | 81.07% | 13.87% | 8.30% |
| PET/30/light | 11.80% | 11.24% | 56.58% | 8.86% | 18.73% |
| PVC/30/light | 11.52% | 9.43% | 7.47% | 8.38% | 15.42% |
| Glass/30/dark | 84.63% | 97.73% | 99.99% | 99.99% | 19.01% |
| PET/30/dark | 88.11% | 87.60% | 34.22% | 63.45% | 2.61% |
| PVC/30/dark | 86.21% | 30.88% | 69.28% | 59.88% | 9.84% |
| Glass/40/light | 10.76% | 21.14% | 61.53% | 9.07% | 12.23% |
| PET/40/light | 9.36% | 20.17% | 1.52% | 8.61% | 3.15% |
| PVC/40/light | 8.26% | 9.01% | 8.90% | 6.32% | 15.31% |
| Glass/40/dark | 77.84% | 58.08% | 99.99% | 99.99% | 7.91% |
| PET/40/dark | 80.10% | 29.25% | 24.69% | 46.09% | 8.52% |
| PVC/40/dark | 78.37% | 15.40% | 66.59% | 25.63% | 13.43% |

**Table 4** Summary of the flavor compounds selected as markers for various storage conditions

| Storage conditions | | Compound(s) |
|---|---|---|
| No oxygen | Low temperature and light | nonanal + hexanal |
| | High temperature and light | hexanal + nonanal |
| | Low temperature and dark | hexanal |
| | High temperature and dark | 2-pentylfuran + hexanal |
| Oxygen | Low temperature and light | hexanal + nonanal + (*E*)-2-heptenal |
| | High temperature and light | 2-pentylfuran + nonanal + (*E*)-2-heptenal |
| | Low temperature and dark | (*E*)-2-heptenal |
| | High temperature and dark | nonanal |

**Acknowledgements** Dr. Coutelieris has a Marie Curie Fellowship with the Unilever Research and Development Center in Vlaardingen, NL.

## References

1. Kiritsakis AK, Dugan LR (1985) J Am Oil Chem Soc 62:892–896
2. Mastrobattista G (1990) Ital J Food Sci 3:191–195
3. Gutierrez GQ (1975) Bottling and canning. In: Moreno JMM (ed) Olive oil technology. FAO, Rome
4. Gutierrez FR, Herrera CG, Gutierez GQ (1988) Grasas Aceites 39:245–253
5. Min BD (1998) Lipid oxidation of edible oils. In: Akoh CC, Min BD (ed) Food lipids: chemistry, nutrition, and biotechnology. Marcel Dekker, New York, pp 283–296
6. Fedeli E (1977) Lipids of olives. In: Holman RT (ed) Progress in the chemistry of fats and other lipids (Vol 15). Academic, Oxford, UK, pp 57–76
7. Frankel EN (1993) Trends Food Sci Tech 4:220–225
8. Kochar SP (1993) Oxidative pathways to the formation of off-flavors. In: Saxby MJ (ed) Food taints and off-flavors. Chapman and Hall, New York, pp 150–201
9. Kanavouras A, Hernandez-Münoz P, Coutelieris FA, Selke S (2004) J Am Oil Chem Soc 81:251–257
10. Del Nobile MA, Ambrosino ML, Sacchi R, Masi P (2002) J Food Sci 68:170–175
11. Del Nobile MA, Bove S, La Notte E, Sacchi R (2003) J Food Eng 57:189–197
12. Dekker M, Kramer M, van Beest M, Luning P (2002) Modeling oxidative quality changes in several packaging concepts. In: Proc 13th IAPRI Conf Packaging. CRC, Boca Raton, FL, pp 297–303
13. Coutelieris FA, Kanavouras A (2004) J Food Eng (accepted)
14. Kanavouras A, Coutelieris FA (2004) Food Chem (accepted)
15. Morales MT, Aparicio R, Rios JJ (1994) J Chromatogr A 668:455–462
16. Morales MT, Aparicio R, Rios JJ (1997) J Agr Food Chem 45:2666–2673
17. Chan HWS, Coxon DT, Peers KE, Price KR (1982) Food Chem 9:21–34
18. Frankel EN (1982) Prog Lipid Res 22:1–33