# Triacylglycerol and Fatty Acid Compositions of French Virgin Olive Oils. Characterization by Chemometrics

**ARTICLE** *in* JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY · OCTOBER 2003

Impact Factor: 2.91 · DOI: 10.1021/jf034365p · Source: PubMed



**5 AUTHORS**, INCLUDING:

Denis Ollivier
Service Commun des Laboratoires, Ministère…
**20** PUBLICATIONS **398** CITATIONS

SEE PROFILE

Jacques Artaud
Aix-Marseille Université
**68** PUBLICATIONS **875** CITATIONS

SEE PROFILE

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

*J. Agric. Food Chem.* **2003,** *51,* 5723−5731 **5723**

# Triacylglycerol and Fatty Acid Compositions of French Virgin Olive Oils. Characterization by Chemometrics

DENIS OLLIVIER,*,† JACQUES ARTAUD,‡ CHRISTIAN PINATEL,§ JEAN PIERRE DURBEC,|| AND MICHEL GUÉRÈRE†

Laboratoire Interrégional de la Direction Générale de la Concurrence et de la Répression des Fraudes, 146 traverse Charles-Susini, 13388 Marseille Cedex 13, France, Laboratoire de Chimie Analytique de l'Environnement, UMR CNRS 6171, IFR 112, Europôle de l'Arbois, 13545 Aix-en-Provence Cedex 4, France, Association Française Interprofessionnelle de l'Olive (AFIDOL), Maison des Agriculteurs, 22 avenue Henri-Pontier, 13626 Aix-en-Provence Cedex 1, France, and Centre d'Océanologie de Marseille, Université de la Méditerranée, Campus de Luminy Case 901, 13288 Marseille Cedex 09, France

There is no data concerning the fatty acid and triacylglycerol composition of French virgin olive oil. Thus, these compositions were determined using 564 samples coming from four olive harvests (1998−1999 to 2000−2001). Among these 564 samples, 372 came from the four main French cultivars (Aglandau, Cailletier, Picholine, and Salonenque) and from both of the oldest French protected designations of origin: "Nyons" (cv. Tanche) and "Vallée des Baux". The fatty acid compositions took the different isomeric monounsaturated fatty acids (C16:1 and C18:1) into account. The eicosenoic acid is gondoic acid (20:1n-9) and was determined by dimethyl disulfide adduct using GC/MS. The use of propionitrile as a mobile phase for the HPLC analysis of the triacylglycerols led to better resolutions between triacylglycerols than those resolutions obtained with the mix of solvents recommended by the normalized method (acetone/acetonitrile). Of the samples, 88 had a 9-heptadecenoic acid level (17:1n-8) higher than 0.3% and 33 had a linolenic acid level higher than 0.9%, which are maximal values accepted by the International Olive Oil Council and the European Union. A linear discriminant analysis was carried out on 372 samples with the SAS system and particularly with STEPISC and CANDISC procedures. Variables ($n = 37$) representing the different fatty acids, the sum of saturated, monounsaturated, and polyunsaturated fatty acids, squalene, and triacylglycerols were used, thus allowing us to classify samples into six groups defined with 100% of well classified samples. These results constitute an original data bank that can be used to identify the origin of virgin olive oils.

**KEYWORDS: French virgin olive oil; fatty acids; triacylglycerols; monocultivars; PDO; chemometrics**

## INTRODUCTION

The annual world olive oil production amounts to 2.6 million tons for the 2000/2001 harvest, and should be about 3 million tons for the 2001/2002 harvest (*1*). The European Union (EU) is the most important olive oil producer in the world, with 75% of the total production. Within the EU, Spain (43%), Italy (32%), and Greece (22%) provide 97% of olive oil production. France holds a special position with a very modest production from 2500 to 3500 tons, which is approximately 0.15% of world production. Consumers' interest for the Mediterranean diet and the extensive media coverage for the beneficial effects of virgin olive oil on health has created a revival in French olive-tree farming. This revival has resulted in new plantations of different varieties, including some that were not farmed much, in renovated mills using recent technology, in special oil vintages or monocultivar oils that correspond to a recent demand from informed consumers. In twenty years, French consumption, made up of 98% of extra virgin olive oil, has increased from 20 000 tons to 80 000 tons. The particularity of French olive oils lies in the diversity of their organoleptic characteristics representative of different varieties and olive growing areas. The pursuit of authenticity and increased quality over the past few years has led to the recognition of five French protected designations of origin ("AOC" in French), including two that received a European protected denomination of origin (PDO).

Faced with the difficult nature of the problems to be solved to characterize an original variety of virgin olive oil, different ways were explored: stable carbon isotope ratios ($^{13}C/^{12}C$) (*2*),

* To whom correspondence should be addressed. Tel.: 33-491618201. Fax: 33-491618219. E-mail: denis.ollivier@dgccrf.finances.gouv.fr.
† Laboratoire Interrégional de la Direction Générale de la Concurrence et de la Répression des Fraudes.
‡ Laboratoire de Chimie Analytique de l'Environnement.
§ Association Française Interprofessionnelle de l'Olive (AFIDOL).
|| Centre d'Océanologie de Marseille.

10.1021/jf034365p CCC: $25.00 
Published on Web 08/09/2003


**Figure 1.** Geographical areas of French continental olive growing.

$^{13}C$ nuclear magnetic resonance (NMR) (*3*), and physicochemical characteristics (*4*, *5*).

There are a great deal of data on fatty acid and triacylglycerol compositions in various original olive oils but paradoxically, there is no general data on French virgin olive oils and more particularly on the main varieties farmed.

The aim of the present work was to fill this gap by studying fatty acid and triacylglycerol compositions in virgin olive oils produced in France during the four crops of years 1997−1998 to 2000−2001. These data have been used to research discriminant criteria between main cultivars and two PDOs farmed in France.

## MATERIALS AND METHODS

**Samples.** French virgin oil samples ($n = 564$) were obtained from the French Inter-professional Olive Association (AFIDOL), Aix en Provence, France. Industrial samples were carried out during the 1997 (81 samples), 1998 (88 samples), 1999 (128 samples), and 2000 (267 samples) crops. Among 564 samples, 372 stem from the four main French cultivars, Aglandau (65), Cailletier (96), Picholine (33), Salonenque (19), and from both of the oldest French PDOs, PDO "Nyons" (cv. Tanche) (95) and PDO "Vallée des Baux" (64). Oils of the PDO Vallée des Baux came from a blend of four main sorts (Aglandau, Salonenque, Grossane, Verdale des Bouches du Rhône). The other samples came from oils of which the variety origin was unknown or which were not numerous enough to constitute a group. The harvest time lasted three months (November to January).

**Figure 1** represents the French continental olive grove areas (11 departments). Aglandau is the first French variety for oil with approximately 20% of produced oil on a national scale. Its crop area spreads mainly over three departments, "Alpes de Haute Provence", "Bouches du Rhône", "Vaucluse", and as a secondary variety in some areas in the "Var".

Cailletier is better known under the name of "Olive de Nice". Its crop area spreads essentially over the department of "Alpes-Maritime".

Picholine is an original variety of the Gard department whose crop has developed considerably in the French olive farming basin. It is rather a rustic variety whose production can be used as green table-olives as well as olives for oil.

Salonenque is a variety essentially grown in the "Bouches du Rhône"; it represents more than half of the variety population. This variety is also used as green table-olives.

Tanche is a variety essentially localized in the region of Nyons (Drome department), mainly used as "black table-olives". Generally, normal fruits are used for the table and small fruits for oil.

**Analysis of Fatty Acid Composition by Gas Chromatography.** Olive oil in *n*-heptane (0.1 g /2 mL) was transmethylated with a cold solution of KOH (2 M) according to the NF EN ISO 5509 Norm (*6*). Fatty acid methyl esters (FAME) were analyzed according to the NF EN ISO 5508 Norm (*7*). Analyses were performed on a Perkin-Elmer Autosystem 9000 gas chromatograph equipped with a split/split-less injector ($t = 250$ °C) and flame ionization detector (FID) ($t = 250$ °C). A silica capillary column (25-m × 0.25-mm i.d., 0.25-$\mu$m film thickness) coated with BP 20 (poly(ethylene glycol), SGE) was used. The inlet pressure of the hydrogen as carrier gas was 83 kPa. The oven temperature program was as follows: 1 min at 180 °C, from 180 to 220 °C at 3 °C/min, 8 min at 220 °C.

**Analysis of Dimethyl Disulfide Adducts by Gas Chromatography/Mass Spectrometry.** FAME samples (1−2 mg) were treated in 200 $\mu$L of *n*-heptane by the addition of 100 $\mu$L of dimethyl disulfide (DMDS) and 50 $\mu$L of iodine solution containing 1−2 mg of iodine in 20 mL of diethyl ether (*8*). The reaction was carried out in a 2-mL closed vial fitted with a Teflon-lined screw-cap lid for 48 h at 50 °C in an oven. Thereafter, 200 $\mu$L of *n*-heptane were added to the reaction mixture and the excess iodine was reduced by treatment with 200 $\mu$L of an aqueous solution of 5% $Na_2S_2O_3$. After recovery of the organic phase, the aqueous phase was extracted for a second time with 200 $\mu$L of *n*-heptane. The combined extracts were concentrated under a stream of nitrogen prior to subsequent GC/MS analysis.

DMDS adducts were identified on a Hewlett-Packard 5890 series II gas chromatograph equipped with split-less injector and a DB-5MS fused silica capillary column (60-m × 0.25-mm i.d., film thickness 0.25 $\mu$m) (J&W Scientific) coupled to a Hewlett-Packard 5898 A MS Engine mass spectrometer. The transfer line was held at 295 °C and the source at 240 °C. Electron impact mass spectra were acquired at 70 eV from 40 to 600 Da. Helium was used as carrier gas at a constant flow rate of 1 mL/min. The oven temperature program was as follows: 1 min at 30 °C, from 30 to 70 °C at 50 °C/min, from 70 °C at 120 °C at 10 °C/min, from 120 °C to 290 °C at 2 °C/min, and finally held for 20 min.

**Analysis of Triacylglycerol Composition by Liquid Chromatography.** Triacylglycerols were analyzed by a HPLC system composed of a Merck liquid chromatograph model LaChrom equipped with a Merck RP-18 Supersphere 100 column (244 × 4 mm i.d., 4 $\mu$m, temperature 24 °C) coupled with a Merck refractometric L-7490 detector. A sample loop of 100-$\mu$L capacity was used. Propionitrile (*9*, *10*) (Carlo Erba, Milan) was the mobile phase with a flow rate linear gradient (0.5 to 0.8 mL/min) for 55 min. Triacylglycerols in olive oils were separated according to equivalent carbon number (ECN), often defined as CN-2*n*, where CN is the carbon number and *n* is the number of double bonds.

**Chemometric Methods.** To investigate the relationships between an oil sample origin and its triacylglycerol and fatty acid profiles, a linear discriminant analysis (*11*) was performed on 372 oil samples divided into six origin groups (Aglandau, Cailletier, Picholine, Salonenque, Tanche, and PDO Vallée des Baux). The original data were column centered before analysis. The multivariate distributions of the profiles in the six groups were assumed normal with equal variance−covariance matrixes. Indeed, considering that these matrixes were nonequal did not improve the quality of the results. In this case, the discriminant functions are linear combinations of initial variables. A stepwise method was used for the estimation of the discriminant functions. A leaving-one-out cross validation procedure was performed for assessing the performance of the classification rule. In this last step, the sample data minus one observation was used for calculating discriminant functions, then the omitted observation was classified from them. The procedure was repeated 372 times. Consequently, each sample was classified from discriminant functions which were estimated without its contribution (*12*). The calculations were carried out with the SAS system and particularly with the STEPISC and CANDISC procedures. The oil samples were plotted on the canonical axes (discriminant coordinates). These axes were determined in such a way that the ratio of the variability inter-groups at the variability intra-groups was maximized. These axes are orthogonal for the scalar product defined by the intra-group covariance matrix (*12*).

**Nomenclature.** *Fatty Acids*. 14:0, myristic acid (tetradecanoic acid); 16:0, palmitic acid (hexadecanoic acid); 16:1n-9, 7-hexadecenoic acid; 16:1n-7, palmitoleic acid (9-hexadecenoic acid); 17:0, margaric acid (heptadecanoic acid); 17:1n-8, 9-heptadecenoic acid; 18:1n-9, oleic acid (9-octadecenoic acid); 18:1n-7, cis vaccenic acid (11-octadecenoic acid); 18:2n-6, linoleic acid (9,12-octadecadienoic acid); 18:3n-3, linolenic acid (9,12,15-octadecatrienoic acid); 20:0, arachidic acid (eicosanoic acid); 20:1n-9, gondoic acid (11-eicosenoic acid); 22:0, behenic acid (docosanoic acid); 24:0, lignoceric acid (tetracosanoic acid).

**Table 1.** Characteristics of Olive Fruits

| cultivar | mean weight of olive fruit[a] (g) | fat value[b] (H) (%) | moisture[c] (%) | non fatty dry matter[d] (MSD) (%) | biological yield[d] (H/MSD) |
|---|---|---|---|---|---|
| Aglandau | 2.08 | 23.9 | 45.3 | 30.8 | 0.77 |
| Cailletier | 1.36 | 22.0 | 42.5 | 35.4 | 0.62 |
| Picholine | 2.98 | 19.0 | 57.2 | 23.8 | 0.80 |
| Salonenque | 3.40 | 22.1 | 54.0 | 24.0 | 0.92 |
| Tanche | 3.12 | 25.0 | 43.1 | 31.9 | 0.79 |

[a] Average on 100 olives. [b] Extraction with tetrachloroethylene and result obtained by density. [c] In oven at 104 °C during 24 h. [d] By calculation.

**Table 2.** Ion Fragments of DMDS Adducts of FAMEs Olive Oil

| fatty acid | name | mol ions | carboxylic ions Δ | carboxylic ions - MeOH | aliphatic ions *n* |
|---|---|---|---|---|---|
| | | *m/z* | *m/z* | *m/z* | *m/z* |
| C16:1n-9 | 7-hexadecenoic acid | 362 | 189 | 157 | 173 |
| C16:1n-7 | 9-hexadecenoic acid | 362 | 217 | 185 | 145 |
| C17:1n-8 | 9-heptadecenoic acid | 376 | 217 | 185 | 159 |
| C18:1n-9 | 9-octadecenoic acid | 390 | 217 | 185 | 173 |
| C18:1n-7 | 11-octadecenoic acid | 390 | 245 | 213 | 145 |
| C20:1n-9 | 11-eicosenoic acid | 418 | 245 | 213 | 173 |

*Triacylglycerols.* The triacylglycerols (TAG) are designated by letters corresponding to abbreviated names of fatty acid carbon chains that are fixed on the glycerol. The abbreviations of fatty acids names are P, palmitoyl; Po, palmitoleyl; S, stearoyl; O, oleoyl; L, linoleoyl; and Ln, linoleolenyl.

## RESULTS AND DISCUSSION

The biological characteristics of the five main French olive fruits are presented in **Table 1**. The biological yield was used rather than fatty matter content, because the aim was not to give the exhaustive composition of the fruits, but instead to give the ability of trees to make fats. The biological yield is a rather constant parameter for a same variety. For French cultivars, the lowest values were about 0.36, the best values were about 1.20, and the medium value was about 0.70.

Salonenque (0.92) and Picholine (0.80) had the most important biological yields, Aglandau (0.77) and Tanche (0.79) had a biological yield slightly higher at the mean, and Cailletier (0.62) had the lowest.

**Fatty Acid and Triacylglycerol Compositions in All Analyzed Samples.** The fatty acid composition in 564 French virgin olive oil samples was determined by gas chromatography of their fatty acid methyl esters (*6*) on a polar column (poly(ethylene glycol)) (*7*). The identification of fatty acids was carried out with standards, GC/MS, and European Community regulation (EC) (*13*). Furthermore, the position of double bonds on monounsaturated fatty acids was determined by GC/MS of the DMDS formed from fatty acid methyl esters (*8*).

**Table 2** gives the molecular ions of DMDS adducts of FAMEs olive oil and two specifical ion fragments (carboxylic Δ and aliphatic *n*) from the cleavage of the adduct between the two $S-CH_3$ groups and ion fragments corresponding to carboxylic ion minus the loss of MeOH.

The results obtained confirmed the identification of fatty acids usually admitted and showed that the eicosenoic acid was the 11-eicosenoic acid (20:1n-9) (gondoic acid).

The isomers of 16:1 and 18:1 acids were globally evaluated on one hand according to the International Olive Oil Council (IOOC) (*14*), the EC regulation (*15*), the Codex Alimentarius (*16*), and most of the numerous works found in the literature, and on the other hand separately, because the use of columns with high resolutions allows it. This more rigorous approach notably allowed us to differentiate the oleic acid (18:1n-9) from cis-vaccenic acid (18:1n-7) and the palmitoleic acid (16:1n-7) from the 7-hexadecenoic acid (16:1n-9). Fatty acid traces (<0.1%) were not taken into account.

**Table 3** gives variation ranges for each of the virgin olive oil fatty acids studied for the entire four years' harvest during the 1997/1998−2000/2001 crops. These values are compared with those of the IOOC (*14*), with those of EC regulation (*15*) and with those of the Codex Alimentarius (*16*).

The results obtained confirmed the great variability of the fatty acid composition of olive oil. This variability is well known and was attributed to numerous factors, cultivar nature, soil characteristics, climatic conditions, olive maturity, etc.

**Table 3.** Fatty Acid Composition (%) of 564 French Virgin Olive Oil Samples[a]

| origin | this work | | | | IOOC (*14*) | | EC (*15*) | codex alimentarius (*16*) | |
|---|---|---|---|---|---|---|---|---|---|
| sample no. or source | 564[a] | | | | regulation | | regulation | regulation | |
| | | range | | | range | | | range | |
| fatty acids | mean | min | max | %RSD[b] | min | max | max | min | max |
| C14:0 | | | tr | | | ≤0.05 | ≤0.05 | | <0.1 |
| C16:0 | 11.3 | 7.5 | 15.6 | 0.13 | 7.5 | 20.0 | | 7.5 | 20.0 |
| C16:1n-9+ C16:1n-7 | 0.89 | 0.4 | 2.0 | 5.44 | 0.3 | 3.5 | | 0.3 | 3.5 |
| C16:1n-9 | 0.14 | 0.1 | 0.2 | 0.66 | | | | | |
| C16:1n-7 | 0.76 | 0.3 | 1.9 | | | | | | |
| C17:0 | 0.1 | tr | 0.3 | 2.70 | | ≤0.3 | | | <0.5 |
| C17:1n-8 | 0.2 | tr | 0.5 | 2.40 | | ≤0.3 | | | <0.6 |
| C18:0 | 2.4 | 1.4 | 3.4 | 0.57 | 0.5 | 5.0 | | 0.5 | 5.0 |
| C18:1n-9+ C18:1n-7 | 75.2 | 63.1 | 83.3 | | 55.0 | 83.0 | | 55.0 | 83.0 |
| C18:1n-9 | 73.1 | 60.9 | 82.1 | 0.07 | | | | | |
| C18:1n-7 | 2.1 | 0.7 | 3.6 | 2.38 | | | | | |
| C18:2n-6 | 8.3 | 4.5 | 16.1 | 0.19 | 3.5 | 21.0 | | 3.5 | 21.0 |
| C18:3n-3 | 0.7 | 0.4 | 1.2 | 0.37 | | ≤1.0 | ≤0.9 | | <1.5 |
| C20:0 | 0.4 | 0.3 | 0.5 | 1.72 | | ≤0.6 | ≤0.6 | | <0.8 |
| C20:1n-9 | 0.3 | 0.2 | 0.5 | 2.54 | | ≤0.4 | ≤0.4 | | |
| C22:0 | 0.1 | tr | 0.2 | 3.48 | | ≤0.2 | ≤0.2 | | <0.2 |
| C24:0 | 0.1 | tr | 0.1 | 5.94 | | ≤0.2 | ≤0.2 | | <1.0 |

[a] Crops 1997/1998, 1998/1999, 1999/2000, and 2000/2001. [b] Relative standard deviation ($n = 13$); tr = trace.

**Table 4.** Triacylglycerol Composition (%) of 564 French Virgin Olive Oil Samples[a]

| origin | this work | | | | Spain | Italy Greece | Portugal | | Tunisia |
|---|---|---|---|---|---|---|---|---|---|
| sample no. or source | 564[a] | | | | (*23*) | (*23*) | (18) | | (*24*) |
| | | range | | | | | range | | |
| triacylglycerol | mean | max | min | RSD%[c] | mean | mean | max | min | mean |
| LLL | 0.13 | 0.90 | 0.01 | 1.01 | | 0.09 | 0.06 | 0.02 | 0.8 |
| OLnL + PoLL[b] | 0.24 | 0.85 | 0.02 | 1.72 | 0.9 | 0.26 | 0.6 | 0.1 | 0.6 |
| PLnL | 0.06 | 0.29 | tr | 9.42 | | | 0.3 | 0.01 | |
| LOL | 1.90 | 6.20 | 0.13 | 0.42 | 0.3 | | 5.1 | 0.0 | 5.8 |
| OLnO + PoOL[b] | 1.36 | 2.46 | 0.52 | 0.96 | 1.0 | 1.04 | 3.2 | 2.1 | 1.5 |
| PLL + PoPoO[b] | 0.55 | 2.17 | 0.05 | 0.95 | 0.5 | 0.27 | 1.2 | 0.4 | 2.8 |
| PLnO + PPoL[b] + PPoPo* | 0.64 | 1.35 | 0.25 | 1.73 | 0.3 | | 1.4 | 0.1 | 1.1 |
| LOO + LnPP[b] | 13.93 | 23.27 | 7.48 | 0.36 | 10.4 | 16.48 | 16.7 | 4.2 | 18.2 |
| PoOO | 1.10 | 3.21 | 0.14 | 0.72 | 1.1 | | 4.0 | 1.1 | |
| PLO + SLL[b] | 5.57 | 11.71 | 2.16 | 0.17 | 4.5 | 4.41 | 11.8 | 2.3 | 12.3 |
| PoOP + SPoL[b] + SOLn[b] + SPoPo[b] | 0.66 | 1.63 | 0.17 | 0.80 | 0.4 | | 2.2 | 0.1 | 1.2 |
| PLP | 0.46 | 1.53 | tr | 0.44 | 0.7 | 0.12 | 1.1 | 0.2 | 2.1 |
| OOO + PoPP[b] | 44.69 | 58.76 | 27.32 | 0.20 | 43.1 | 44.05 | 57.2 | 25.6 | 21.8 |
| SLO | 0.52 | 1.77 | tr | 0.73 | | 1.12 | | | |
| POO | 20.03 | 27.65 | 14.69 | 0.20 | 23.1 | 17.68 | 19.1 | 32.2 | 20.0 |
| POP | 3.08 | 5.38 | 0.45 | 0.55 | 2.9 | 1.77 | 5.4 | 2.1 | |
| SOO | 3.72 | 7.22 | 0.49 | 0.85 | 3.6 | 4.47 | 8.0 | 2.6 | 3.7 |
| POS + SLS[b] | 0.85 | 3.47 | 0.37 | 2.30 | 0.4 | 0.9 | 2.7 | 0.3 | 1.2 |
| PPS | 0.52 | 1.03 | 0.23 | 9.06 | 0.6 | | 1.9 | 0.4 | 0.5 |

[a] Crops: 1997/1998, 1998/1999, 1999/2000, and 2000/2001. [b] Low-level triacylglycerol. [c] Relative standard deviation ($n = 13$).

The values obtained and compared with those of the Codex Alimentarius (*16*) indicated that all samples had fatty acid levels replying to current norms except one, whose 18:1 level (18:1n-9 and 1n-7) was greater than 83%. If one compares results of the 564 samples with values retained by the IOOC (*14*) and by EC (*15*) and excluding the sample with a C18:1 level higher than 83%, there were 88 samples with a 9-heptadecenoic acid (17:1n-8) level higher than 0.3%, while 33 samples had a linolenic acid (18:3n-3) level higher than 0.9%, including 13 higher than 1.0%. The existence of French virgin olive oil samples whose linolenic acid (18:3n-3) level is above the maximum value fixed by the IOOC (1.0%) and by EU (0.9%) is in agreement with previous results obtained in various producing countries of the Mediterranean basin (*17*−*19*).

It appears therefore that values fixed by the IOOC and by EC are too low and should be modified to take into account the existence of a large variation range for linolenic acid (18:3n-3) and 9-heptadecenoic (17:1n-8).

The differentiation of the structural (position) isomers of fatty acids in C16 and C18, compared with the previous data that considers them as a whole, brings a better knowledge of the chemical composition of olive oil and can be of great interest in studying their nutritional impact. Considering both of the octadecenoic acid (18:1n-9 and 1n-7) isomers as oleic acid (18:1n-9) is indeed an unsatisfactory way, from both chemical and nutritional points of view.

The triacylglycerol composition of 564 French virgin olive oil samples was determined by liquid chromatography on a reverse phase RP 18 column using propionitrile (*9*) as mobile phase. This solvent allowed a better resolution between some triacylglycerol (*20*, *21*) that were not or badly separated with the solvent (acetone/acetonitrile) proposed by the normalized method (*22*). Nevertheless, some triacylglycerol are not yet separated.

Triacylglycerol identification was carried out with the help of standards and by comparison with data from literature (*21*).

**Table 4** gives variation ranges and the average value for each triacylglycerol from those olive oils studied for the entire four years' crops. The composition in triacylglycerol presents a great variability as previously described for fatty acids. For example, triolein (OOO) varies between 27.32 and 58.76%, 1,2-dioleoyl-3-palmitoyl-glycerol (POO) between 14.69 and 27.65%, 1,3-dioleoyl-2-linoleoyl-glycerol (LOO) between 7.48 and 23.27%, and 1-palmitoleoyl-2-oleoyl-3-linoleoyl-glycerol (PLO) between 2.16 and 11.71%.

The presence of a high triolein (OOO) level in an olive oil, in inverse proportion to trilinolein (LLL), constitutes a favorable authenticity indicator for the most common European oils.

The values obtained were compared with literature values (*18*, *23*, *24*). Although analysis conditions were different (mobile phase) the main triacylglycerol values (OOO, POO, LOO, PLO) are comparable to those of the most common Spanish, Italian, Greek, and Portuguese oils but have significant differences compared with Tunisian oils.

**Composition in FAME and TAG for the Four Main French Olive Oil Cultivars and Two French PDOs.** About 150 different cultivars were counted in France (*25*). Among these cultivars, only a few of them have significant importance for olive oil production. Their particularity is being often linked to a precise southern region of France, which often allows them to be associated with a region, a cultivar, and therefore, a monocultivar oil.

Compositions in fatty acids and triacylglycerols of 372 samples coming from the four main cultivars and from two French PDOs were determined. The results obtained are presented in **Tables 5** and **6**.

Aglandau oil was characterized by the highest 17 carbon fatty acid (17:0 and 17:1n-8) levels among all those analyzed samples. 9-Heptadecenoic acid (17:1n-8) had an average value of 0.38% including 54 samples with a level higher than 0.3% (IOOC limit).

The linolenic acid (18:3n-3) level was higher than 0.9% for 10 Picholine oil samples. The average value for this acid is high (0.85%) contrary to the other cultivars (0.63−0.66%).

Tanche oils had the highest average rate in oleic acid (79.21%) and trioleine (54.71%), while the palmitic acid rate

**Table 5.** Fatty Acid Composition (%) of 372 French Virgin Olive Oil Samples[a] Resulting from the Four Main French Cultivars and Both of the Two Most Ancient French PDOs, Nyons and Vallée des Baux

| cultivars | Aglandau | | | Cailletier | | | Picholine | | | PDO Nyons (cv. Tanche) | | | Salonenque | | | PDO Vallée des Baux | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| sample no. | 65 | | | 98 | | | 33 | | | 95 | | | 19 | | | 62 | | |
| | range | | | range | | | range | | | range | | | range | | | range | | |
| fatty acid | mean % | min | max | mean % | min | max | mean % | min | max | mean % | min | max | mean % | min | max | mean % | min | max |
| C16:0 | 11.94 | 10.85 | 13.97 | 10.42 | 8.76 | 12.34 | 10.74 | 9.19 | 11.99 | 8.63 | 7.47 | 10.21 | 14.55 | 13.02 | 15.59 | 13.51 | 11.28 | 14.78 |
| C16:1n-9 | 0.14 | 0.11 | 0.16 | 0.11 | 0.06 | 0.19 | 0.13 | 0.08 | 0.17 | 0.15 | 0.11 | 0.19 | 0.11 | 0.08 | 0.12 | 0.13 | 0.10 | 0.15 |
| C16:1n-7 | 0.87 | 0.71 | 1.22 | 0.55 | 0.33 | 0.92 | 0.60 | 0.41 | 0.80 | 0.41 | 0.30 | 0.54 | 1.11 | 0.94 | 1.29 | 1.06 | 0.73 | 1.63 |
| C17:0 | 0.19 | 0.11 | 0.26 | 0.05 | 0.03 | 0.09 | 0.06 | 0.03 | 0.08 | 0.05 | 0.03 | 0.06 | 0.08 | 0.06 | 0.12 | 0.08 | 0.04 | 0.18 |
| C17:1n-8 | 0.38 | 0.23 | 0.53 | 0.11 | 0.08 | 0.18 | 0.10 | 0.08 | 0.14 | 0.08 | 0.06 | 0.10 | 0.15 | 0.10 | 0.22 | 0.15 | 0.09 | 0.32 |
| C18:0 | 2.43 | 2.22 | 2.82 | 2.10 | 1.78 | 2.92 | 2.31 | 2.03 | 2.82 | 2.69 | 1.44 | 3.04 | 2.55 | 2.33 | 2.84 | 2.59 | 2.28 | 3.01 |
| C18:1n-9 | 73.24 | 68.48 | 75.93 | 76.46 | 71.26 | 80.39 | 73.22 | 68.37 | 76.90 | 79.21 | 76.16 | 82.12 | 64.76 | 61.11 | 68.54 | 66.73 | 62.74 | 73.18 |
| C18:1n-7 | 2.26 | 1.94 | 2.71 | 1.94 | 0.72 | 2.71 | 1.74 | 1.29 | 2.08 | 1.47 | 1.16 | 1.76 | 2.55 | 2.17 | 2.77 | 2.45 | 2.10 | 3.09 |
| C18:2n-6 | 7.06 | 5.72 | 9.36 | 6.75 | 5.57 | 9.80 | 9.31 | 6.16 | 12.78 | 5.84 | 5.00 | 6.80 | 12.59 | 10.19 | 15.12 | 11.75 | 7.79 | 14.52 |
| C18:3n-3 | 0.63 | 0.55 | 0.78 | 0.64 | 0.52 | 0.96 | 0.85 | 0.59 | 1.09 | 0.64 | 0.55 | 0.76 | 0.65 | 0.55 | 0.75 | 0.66 | 0.56 | 0.77 |
| C20:0 | 0.40 | 0.35 | 0.47 | 0.38 | 0.29 | 0.44 | 0.36 | 0.30 | 0.41 | 0.38 | 0.34 | 0.43 | 0.44 | 0.40 | 0.49 | 0.44 | 0.38 | 0.48 |
| C20:1n-9 | 0.28 | 0.24 | 0.30 | 0.33 | 0.27 | 0.40 | 0.33 | 0.25 | 0.39 | 0.31 | 0.27 | 0.36 | 0.27 | 0.25 | 0.29 | 0.26 | 0.23 | 0.31 |
| C22:0 | 0.12 | 0.09 | 0.14 | 0.12 | 0.03 | 0.16 | 0.09 | 0.04 | 0.11 | 0.10 | 0.03 | 0.12 | 0.13 | 0.10 | 0.17 | 0.13 | 0.11 | 0.15 |
| C24:0 | 0.05 | 0.02 | 0.07 | 0.05 | 0.02 | 0.06 | 0.05 | 0.03 | 0.07 | 0.04 | 0.02 | 0.05 | 0.06 | 0.02 | 0.08 | 0.06 | 0.05 | 0.07 |
| squalene[b] | 0.82 | 0.60 | 1.00 | 0.46 | 0.30 | 0.62 | 0.61 | 0.38 | 0.78 | 0.83 | 0.38 | 1.08 | 0.76 | 0.57 | 0.93 | 0.73 | 0.57 | 0.84 |
| Σ saturated fa[c] | 15.13 | 13.99 | 17.20 | 13.11 | 11.51 | 15.36 | 13.61 | 12.24 | 14.87 | 11.89 | 10.13 | 13.44 | 17.81 | 16.25 | 19.25 | 16.80 | 14.31 | 18.31 |
| Σ monoenic fa | 77.17 | 73.08 | 79.35 | 79.52 | 75.05 | 83.01 | 76.13 | 71.59 | 79.82 | 81.63 | 79.01 | 84.28 | 68.94 | 65.46 | 72.70 | 70.85 | 66.72 | 77.19 |
| Σ polyenic fa | 7.69 | 6.29 | 10.02 | 7.39 | 6.18 | 10.38 | 10.15 | 6.87 | 13.72 | 6.47 | 5.58 | 7.50 | 13.24 | 10.81 | 15.75 | 12.36 | 8.50 | 15.12 |

[a] Crops: 1997/1998, 1998/1999, 1999/2000, and 2000/2001. Values were calculated as the % of the total fatty acids. [b] Value was determined as the % of the total fatty acids and squalene sum. [c] fa = fatty acids.

**Table 6.** Triacylglycerol Composition (%) of 372 French Virgin Olive Oil Samples[a] Resulting from the Four Main French Cultivars and Both of the Two Most Ancient French PDOs, Nyons and Vallée des Baux

| cultivars | Aglandau | | | Cailletier | | | Picholine | | | PDO Nyons (cv. Tanche) | | | Salonenque | | | PDO Vallée des Baux | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| sample no. | 65 | | | 98 | | | 33 | | | 95 | | | 19 | | | 62 | | |
| | range | | | range | | | range | | | range | | | range | | | range | | |
| triacylglycerol | mean % | min | max | mean % | min | max | mean % | min | max | mean % | min | max | mean % | min | max | mean % | min | max |
| LLL | 0.08 | 0.03 | 0.15 | 0.05 | 0.01 | 0.15 | 0.17 | 0.02 | 0.28 | 0.06 | 0.02 | 0.13 | 0.25 | 0.11 | 0.41 | 0.27 | 0.11 | 0.44 |
| OLnL + PoLL[b] | 0.17 | 0.11 | 0.25 | 0.19 | 0.13 | 0.35 | 0.38 | 0.23 | 0.51 | 0.16 | 0.10 | 0.21 | 0.26 | 0.18 | 0.33 | 0.36 | 0.24 | 0.52 |
| PLnL | 0.05 | 0.02 | 0.08 | 0.05 | 0.02 | 0.07 | 0.08 | 0.04 | 0.11 | 0.02 | tr[c] | 0.05 | 0.11 | 0.06 | 0.16 | 0.11 | 0.06 | 0.15 |
| LOL | 1.22 | 0.61 | 1.87 | 1.20 | 0.57 | 2.38 | 2.41 | 1.27 | 3.82 | 1.07 | 0.56 | 1.70 | 3.25 | 2.21 | 4.52 | 3.73 | 1.99 | 5.01 |
| OLnO + PoOL[b] | 1.28 | 0.74 | 1.95 | 1.34 | 0.64 | 2.26 | 1.45 | 0.74 | 2.45 | 1.45 | 0.83 | 2.03 | 0.82 | 0.62 | 1.05 | 1.83 | 1.56 | 1.99 |
| PLL + PoPoO[b] | 0.37 | 0.11 | 0.67 | 0.31 | 0.10 | 0.80 | 0.47 | 0.19 | 0.75 | 0.16 | 0.05 | 0.37 | 1.20 | 0.69 | 1.91 | 1.46 | 0.60 | 2.17 |
| PLnO + PPoL[b] | 0.72 | 0.51 | 0.95 | 0.57 | 0.32 | 1.00 | 0.66 | 0.38 | 0.99 | 0.45 | 0.25 | 0.72 | 0.58 | 0.45 | 0.69 | 0.91 | 0.63 | 1.11 |
| LOO + LnPP[b] | 12.14 | 10.22 | 14.54 | 12.78 | 10.39 | 16.68 | 16.39 | 13.26 | 19.84 | 11.95 | 10.29 | 13.87 | 16.76 | 15.03 | 18.47 | 16.62 | 13.02 | 18.70 |
| PoOO | 1.30 | 0.70 | 2.10 | 0.89 | 0.20 | 2.01 | 0.68 | 0.14 | 1.57 | 0.71 | 0.27 | 1.27 | 0.88 | 0.58 | 1.50 | 2.13 | 1.68 | 3.21 |
| PLO + SLL[b] | 4.98 | 3.51 | 6.75 | 4.47 | 3.30 | 6.97 | 5.51 | 4.10 | 7.48 | 3.09 | 2.16 | 4.10 | 9.10 | 7.06 | 11.43 | 9.08 | 5.72 | 11.11 |
| PoOP + SPoL[b] + SOLn[b] + SPoPo[b] | 1.05 | 0.79 | 1.30 | 0.41 | 0.24 | 0.83 | 0.39 | 0.18 | 0.62 | 0.27 | 0.17 | 0.39 | 0.72 | 0.38 | 1.20 | 1.07 | 0.77 | 1.63 |
| PLP | 0.34 | 0.17 | 0.66 | 0.25 | tr | 0.69 | 0.40 | 0.10 | 0.55 | 0.16 | tr | 0.26 | 0.99 | 0.62 | 1.42 | 1.09 | 0.59 | 1.49 |
| OOO + PoPP[b] | 45.07 | 37.65 | 51.94 | 49.90 | 40.55 | 56.02 | 43.83 | 37.91 | 49.70 | 54.71 | 50.95 | 58.76 | 33.48 | 28.05 | 39.20 | 32.82 | 27.32 | 42.68 |
| SLO | 0.44 | 0.07 | 0.99 | 0.37 | 0.04 | 0.75 | 0.44 | 0.07 | 1.02 | 0.56 | 0.10 | 1.06 | 0.41 | 0.16 | 0.61 | 1.21 | 0.80 | 1.61 |
| POO | 21.97 | 18.95 | 24.92 | 19.98 | 17.56 | 22.22 | 18.82 | 16.29 | 21.07 | 17.37 | 14.69 | 18.78 | 21.79 | 20.70 | 23.08 | 19.23 | 17.03 | 23.47 |
| POP | 3.53 | 2.53 | 4.68 | 2.67 | 1.99 | 3.95 | 3.02 | 2.07 | 3.77 | 2.06 | 1.53 | 2.65 | 4.31 | 3.87 | 4.94 | 3.41 | 2.89 | 4.06 |
| SOO | 3.85 | 2.98 | 5.15 | 3.36 | 2.43 | 5.12 | 3.62 | 2.58 | 5.13 | 4.57 | 3.75 | 5.60 | 3.49 | 3.00 | 4.37 | 3.24 | 2.94 | 3.69 |
| POS + SLS[b] | 0.93 | 0.65 | 1.26 | 0.66 | 0.43 | 1.19 | 0.80 | 0.52 | 1.08 | 0.72 | 0.38 | 1.05 | 1.11 | 0.61 | 1.39 | 0.95 | 0.76 | 1.34 |
| PPS | 0.52 | 0.36 | 0.72 | 0.53 | 0.41 | 0.98 | 0.46 | 0.33 | 0.59 | 0.55 | 0.39 | 0.74 | 0.50 | 0.43 | 0.67 | 0.49 | 0.35 | 0.80 |

[a] Crops: 1997/1998, 1998/1999, 1999/2000, and 2000/2001. Values were calculated as the % of the total fatty acids. [b] Low-level triacylglycerol. [c] tr = trace.

(8.63%) and linoleic acid rate (5.84%) were the weakest of the studied cultivars. On the other hand, Salonenque oils possessed the weakest average level in oleic acid (64.76%) and triolein (33.48%) within studied groups. On the other hand, they had the highest average level in palmitic acid (14.55%) and linoleic acid (12.59%).

Variability in fatty acid and triacylglycerol concentrations between oil samples led to perform multivariate statistical methods to describe the characteristics of oils coming from the different cultivars and to assess their differences.

Several approaches were previously used as monovariate and multivariate analyses of variance or as principal component analysis and hierarchical and nonhierarchical classification methods (*26*−*28*). Neuronal classification methods were also used (*26*). In this work, stepwise linear discriminant analysis was used with fatty acid, triacylglycerol, and squalene concen-

**Table 7.** Generalized Distances—Mahalanobis Distances—between the Centers of Oil Groups

| group | Aglandau | Cailletier | Picholine | Salonenque | Tanche | Vallée des Baux |
|---|---|---|---|---|---|---|
| Aglandau | 0 | 137.75 | 177.25 | 89.69 | 146.90 | 121.51 |
| Cailletier | 137.75 | 0 | 89.75 | 88.32 | 80.20 | 129.62 |
| Picholine | 177.25 | 89.75 | 0 | 134.56 | 120.17 | 125.79 |
| Salonenque | 89.69 | 88.32 | 134.56 | 0 | 134.06 | 59.08 |
| Tanche | 146.90 | 80.20 | 120.17 | 134.06 | 0 | 158.11 |
| Vallée des Baux | 121.51 | 129.62 | 125.79 | 59.08 | 158.11 | 0 |

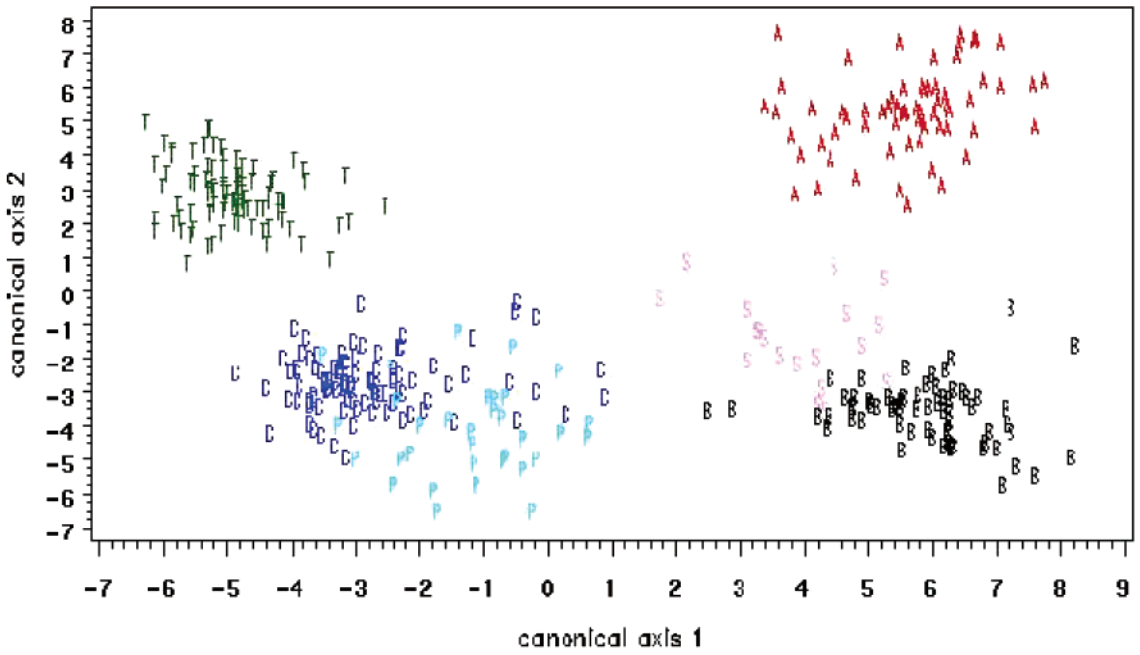


**Figure 2.** Plan of the two first canonical axes with cultivars Aglandau (A), Cailletier (C), PDO Vallée des Baux (B), Picholine (P), PDO Nyons as cv. Tanche (T), and Salonenque (S).

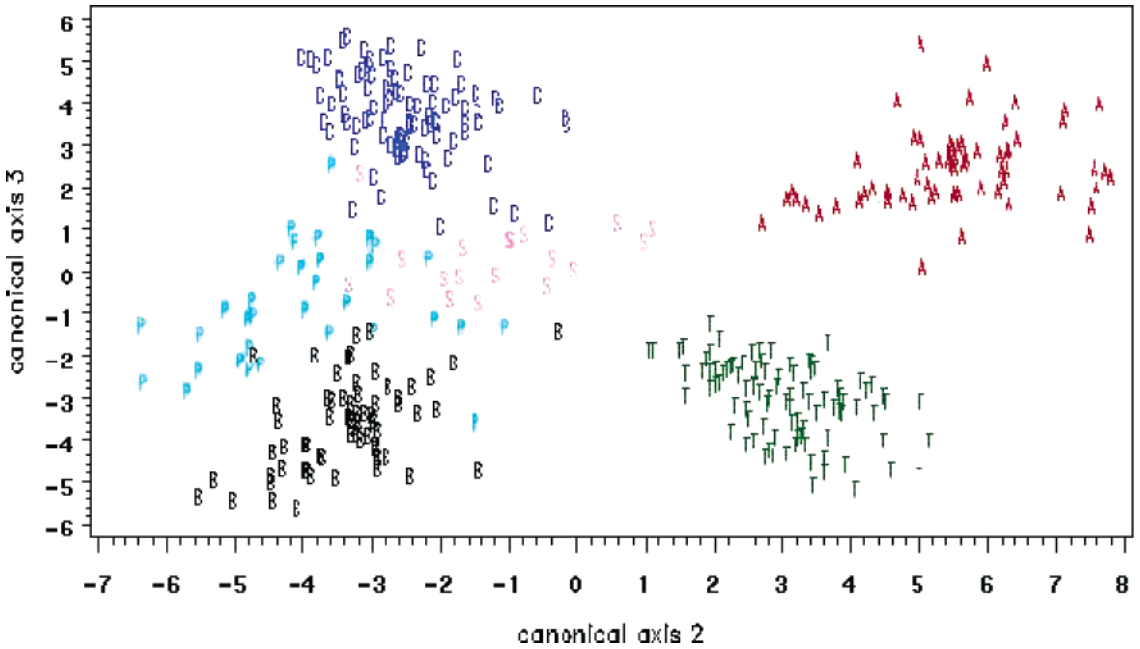


**Figure 3.** Plan of the canonical axes 2 and 3 with cultivars Aglandau (A), Cailletier (C), PDO Vallée des Baux (B), Picholine (P), PDO Nyons as cv. Tanche (T), and Salonenque (S).

trations. These parameters were the only ones retained, because they are the most stable and the most indicative (*29*−*31*) among commonly measured parameters (acidity, peroxide value, phenols, tocopherols, etc.).

Each oil sample was described by 37 relative percentages of individual, total saturated, total monounsaturated, total polyunsaturated fatty acids, triacylglycerols, and squalene variables.

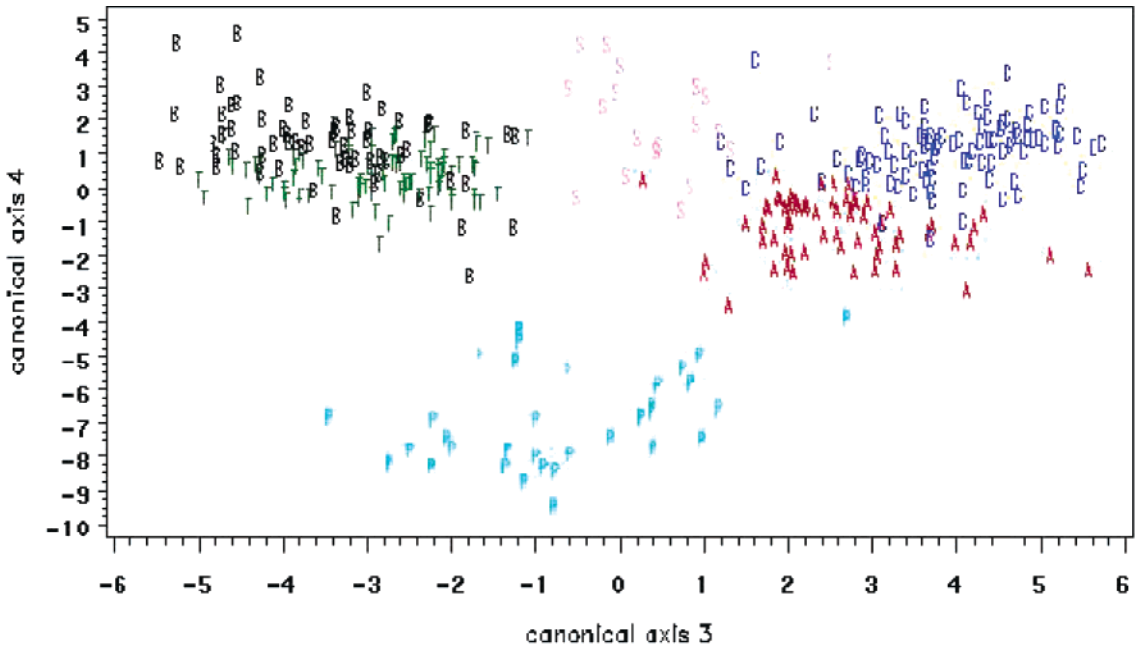


**Figure 4.** Plan of the canonical axes 3 and 4 with cultivars Aglandau (A), Cailletier (C), PDO Vallée des Baux (B), Picholine (P), PDO Nyons as cv. Tanche (T), and Salonenque (S).

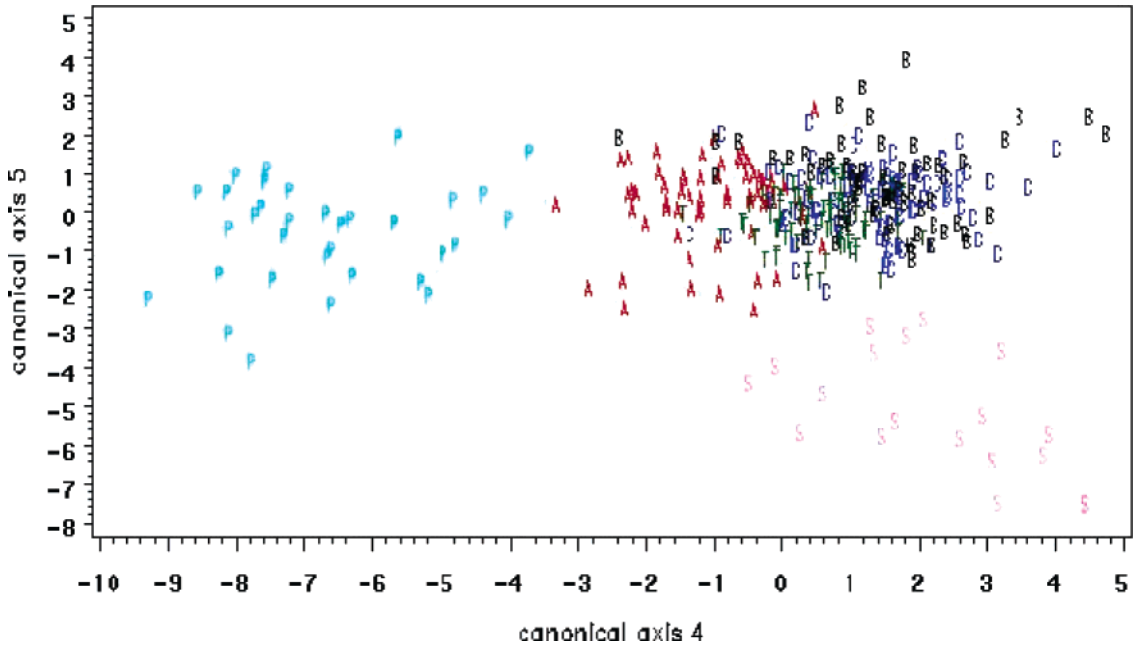


**Figure 5.** Plan of the canonical axes 4 and 5 with cultivars Aglandau (A), Cailletier (C), PDO Vallée des Baux (B), Picholine (P), PDO Nyons as cv. Tanche (T), and Salonenque (S).

The generalized distances between groups (Mahalanobis distances) are displayed in **Table 7**. The Vallée des Baux and Salonenque groups were the nearest (59.08) and presented the most similar mean profiles. This result was foreseeable because Vallée des Baux oils contain a large rate of Salonenque oil. On the other hand, the Aglandau and Picholine groups presented the most different mean profiles (177.25).

Stepwise procedure of discriminant analysis led us to select only twenty variables (OOO, 17:1n-8, 18:0, OlnL, PoOP, PLO, squalene, POO, 16:0, saturated fatty acids, 16:1n-9, 18:3n-3, LOO, 18:1n-7, LLL, OlnO, POP, 24:0, 17:0, 20:0) to determine discriminant functions.

The correct classification rate, given by the leaving-one-out method, was 100%. This perfect classification can be explained by absolutely certain identification of the cultivars used in the analysis.

The plots of samples on canonical planes clearly showed the differences between groups. The discrimination between, on one hand, Aglandau, Vallée des Baux and Salonenque groups, and on the other hand, Tanche, Cailletier and Picholine groups was clearly displayed along the first canonical axis (41.9% of total variability) (**Figure 2**). On the second canonical axis (25.1% of the total variability), the Tanche and Aglandau groups were distinctly separated of Vallée des Baux, Cailletier and Picholine groups.

The third canonical axis (19.1% of total variability) displayed differences between Vallée des Baux and Tanche groups on

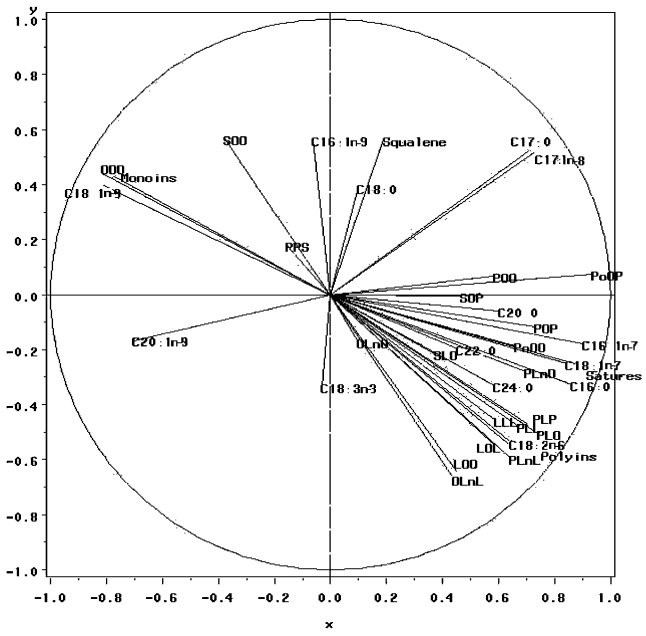


**Figure 6.** Correlation circle of the coefficients of correlation of all variables with the canonical variables 1 and 2.

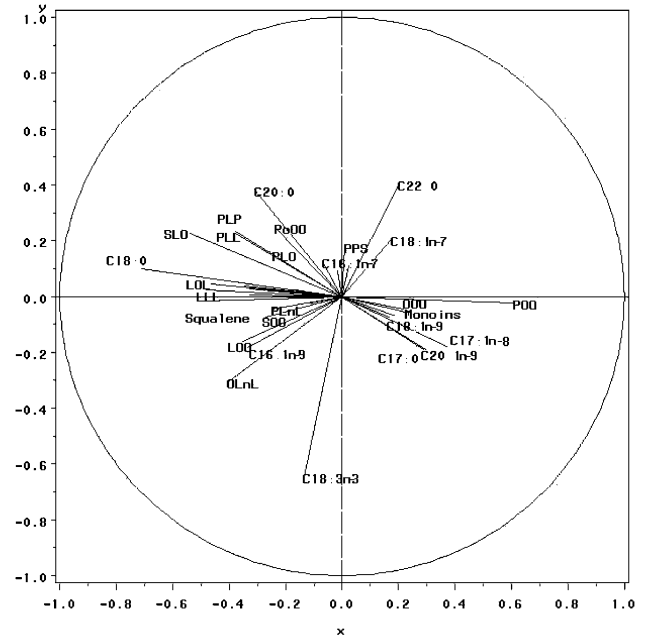


**Figure 7.** Correlation circle of the coefficients of correlation of all variables with the canonical variables 3 and 4.

the same side of the axis from Cailletier and Aglandau groups on the other side (**Figure 3**).

The fourth canonical axis (10.82% of total variability) enabled the Picholine group to be individualized (**Figure 4**), and the fifth canonical axis (3.07% of total variability) individualized the Salonenque group (**Figure 5**).

The coefficients of correlation of all variables with canonical variables are displayed on correlation circles (**Figures 6** and **7**). A vector, the components of which are the coefficients of correlation with two canonical variables, was associated with each variable. Only the variables with the highest correlation coefficients are displayed.

Most relative percentages were positively correlated with the first canonical variable (**Figure 6**). Only 18:1n-9, 20:1n-9, total monounsaturated fatty acids and OOO were negatively correlated with it.

Consequently, Tanche and Cailletier oil samples had higher contents in these fatty acids and triacylglycerols than Aglandau, Vallée des Baux, and Salonenque oil samples.

The second canonical variable (**Figure 6**) was negatively correlated with OLnL, LOO, LOL, total polyunsaturated fatty acids, PLO, and PLP. It was positively correlated with squalene, 16:1n-9, 17:0, 17:1n-8 and SOO, but correlation coefficients were relatively weak (0.4−0.6). The Picholine, Vallée des Baux, Cailletier and Salonenque oil samples had higher contents in OLnL, LOO, LOL, polyunsaturated fatty acids, PLO, and PLP than Aglandau and Tanche samples.

The correlation circles enabled us to interpret the canonical variables in terms of the relative percentages of variables.

The third canonical variable (**Figure 7**) displayed negative correlations with 18:0 and SLO and positive correlation with POO (other correlation coefficients were weak). This axis enabled us to discriminate between Aglandau and Cailletier groups on one hand and Tanche and Vallée de Baux groups on the other hand.

The fourth canonical variable (**Figure 7**) was essentially negatively correlated with C18:3n-3. Picholine oil samples had higher contents in this fatty acid than those from the other five groups.

Finally, the fifth canonical axis enabled us to individualize Salonenque oil samples as having higher contents in POP and total polyunsaturated acids than the other oil samples.

This work has enabled us to constitute a bank of original data concerning the fatty acid and triacylglycerol composition of French virgin olive oils. Some samples were characterized by higher levels of fatty acids (17:1n-8 and 18:3n-3) than those defined by the IOOC and EC. The existence of virgin olive oils presenting "out of norm" physicochemical characteristics requires the adjustment of commercial criteria and current regulations (EC).

The statistical processing of the different data obtained by commonly practiced physicochemical methods (fatty acids, triacylglycerols) allowed the characterization of the main cultivars (Aglandau, Cailletier, Picholine, Salonenque) and two PDOs (Nyons and Vallée des Baux) that split into distinct groups. These results should enable us to verify the authenticity of monocultivar and French PDO oils and also to trace them.

## ACKNOWLEDGMENT

We appreciate the technical assistance of Pierre Doumenq for GC-MS analysis and Véronique Ollivier, to whom this paper is dedicated. We also thank the reviewers for their constructive criticisms of the manuscript.

## LITERATURE CITED

(1) International Olive Oil Council. The world market for olive oils. *Olivae* **2002**, *92*, 22−24.

(2) Bianchi, G.; Angerosa, F.; Camera L.; Reiniero, F.; Anglani, C. Stable carbon isotope ratios ($^{13}C/^{12}C$) of olive oil components. *J. Agric. Food Chem.* **1993**, *41*, 1936−1940.

(3) Vlahov, G.; Shaw, A. D.; Kell, D. B. Use of $^{13}C$ nuclear magnetic resonance distortionless enhancement by polarization transfert pulse sequence and multivariable analysis to discriminate olive oil cultivars. *JAOCS* **1999**, *76*, 1223−1231.

(4) Alessandri, S. Agronomics tehniques and characteristics of olive oil. In *World Encyclopedia of Olive Tree. International Olive Oil Council.* Plazza & Janès: Barcelone, Spain, 1997, ch 5, pp 212−217.

(5) Dugo, G.; Lo Curto, S.; Alfa, M.; Di Bella, G.; Lo Turco, V.; Mavrogeni, E.; Saitta, M.; Salvo, F.; Pizzimenti, G.; Maisano, R. Caratterizzazione di oli di oliva vergini siciliani. *Riv. Ital. Sostanze Grasse* **2000**, *77*, *12*, 803−833.
(6) European Standard NF EN ISO 5509. Preparation of methyl esters of fatty acids. AFNOR: Paris, 2000.
(7) European Standard NF EN ISO 5508. Analysis by gas chromatography of methyl esters of fatty acids. AFNOR: Paris, 1995.
(8) Francis, G. W.; Veland, K. Alkylthiolation for the determination of double bond positions in linear alkenes. *J. Chromatogr*. **1981**, *219*, 379−384.
(9) Schulte Von, E. Trennung von triglyceriden nach kettenlänge und Sättigungsgrad durch HPLC. *Fette Seifen Anstrichmittel* **1981**, *83* (8), 289−291.
(10) Fiebig Von, H.-J. HPLC-Trennung von Triglyceriden. *Fette Seifen Anstrichmittel* **1985**, *87* (2), 53−57.
(11) Dillon, W.; Goldstein, M. *Multivariate Analysis. Methods and Applications*; John Wiley: New York, 1994.
(12) Rencher, A. C. *Methods of Mutivariate Analysis*; John Wiley: New York, 1995.
(13) European Community Regulation 796/2002, Annex X B. *Off. J. Eur. Communities* 2002, L128, 8−28.
(14) International Olive Oil Council. Trade standard applying to olive oil and olive-pomace oil. COI/T15/NC no 2/Rev. 10, 8 November 2001.
(15) European Community Regulation 2568/91. *Off. J. Eur. Communities* 1991, 34 (9/5/91).
(16) Codex Alimentarius: 1992. Food and Agriculture Organization of the United Nations. World Heath Organisation. Viale delle Terme di Caracalla 00100 Rome.
(17) Dettori, S.; Russo, G. Influence of the cultivar and the hydrology regime on the volume of the production and the quality of olive oil. *Olivae* **1993**, *49*, 36−43.
(18) Gouveia, J. M. B. Comparative study between olive oils of the cvs Cobrançosa, Blanqueta, Azeiteira, and Picual and that of the cv Galega vulgar, produced in the North of the Alentejo. I Principal chemical and sensory characteristics. *Olivae* **1997**, *66*, 34−45.
(19) El Antari, A.; Hilal, A.; Boulouha B.; El Moudni A. Study of the influence of the variety, the environment, and cultivatin techniques on characteristics of fruit and the chemical composition of the extra virgin olive oil to Morocco. *Olivae* **2000**, *80*, 29−36.
(20) Ollivier, D.; Bruckert, B.; Noyer, C.; Guérère, M.; Artaud J. Multicriteria analysis for the research of adulteration of virgin olive oil by hazelnut and almond oils. *Ann. Fals. Exp. Chim.* **1999**, *92*, (947), 161−176.
(21) Moreda, W.; Pérez-Camino, M. C.; Mateos, R.; Cert, A. Improved method for the determination of triacylglycerols in olive oils by HPLC. *Grasas Aceites* (Sevilla), **2003**, *54*, 115−119.
(22) IUPAC. *Determination of Triglycerides in Liquid Vegetable Oils in Terms of Their Equivalent Carbon Number by High-Performance Liquid Chromatography*, 7th ed., Blackwell Scientific Publications: Oxford, UK, 1987; 2324.
(23) Fedelli, E. Stockage and processing technologies. In *World Encyclopedia of Olive tree. International Olive Oil Council.* Plazza & Janès: Barcelona, Spain, 1997, ch 7, p 263.
(24) Amamou, T. Typology and variability of olive oils according to the fruit origin. *OCL* **1999**, *6* (1), 77−79.
(25) Ruby, J. Recherche morphologiques et biologiques sur l′olivier et sur ses variétés cultivées en France. Thèses, 1918, Facultés des Sciences de Paris.
(26) Bucci, R.; Magri, A. D.; Magri, A. L.; Marini, D.; Marini, F. Chemical authentification of extra virgin olive oil varieties by supervised chemometric procedures. *J. Agric. Food Chem.* **2002**, *50*, 413−418.
(27) Forina, M.; Tiscornia, E. Pattern recognition methods in the prediction of italian olive oil origin by their fatty acid content. *Ann. Chim.* **1982**, *72*, 143−155.
(28) Boggia, R.; Zunin, P.; Lanteri, S.; Rossi, N.; Evangelisti, F. Classification and class modeling of "Riviera Ligure" extra virgin olive oil using chemical-physical parameters. *J. Agric. Food Chem.* **2002**, *50*, 2444−2449.
(29) Ranalli, A.; Pollastri, L.; Contento, S.; Di Loreto, G.; Iannucci, E.; Lucera, L.; Russi, F. Acylglycerol and fatty acid components of pulp, seed, and whole olive fruit oils. Their use to characterize fruit variety by chemometrics. *J. Agric. Food Chem.* **2002**, *50*, 3775−3779.
(30) Tsimidou, M.; Macrae, R.; Wilson, I. Authentication of virgin olive oils using principal component analysis of triglyceride and fatty acid profiles. I Classification of Greek olive oils. *Food Chem.* **1987**, *25* (3), 227−239.
(31) Ollivier, D.; Soulliol, S.; Guérère, M.; Pinatel, C.; Durbec, J. P.; Artaud, J. Preliminary study with the aim of characterizing French Virgin Olive Oil. *Ann. Fals. Exp. Chim.* **2001**, *94* (956), 273−280.

**Received for review April 10, 2003. Revised manuscript received June 21, 2003. Accepted June 25, 2003.**

JF034365P