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Saturated hydrocarbon content in olive fruits and crude olive-pomace oils

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Abstract

Olive fruits contain an *n*-alkane series of saturated hydrocarbons mainly in the pulp. Lower amounts of a complex mixture of paraffins, unresolved by gas chromatography (UCM), have been found in cuticle, stone (woody shell and seed), olive leaves, and talc used as an aid to olive oil extraction. The amounts of both kinds of hydrocarbons are related to the olive cultivar and are transferred to oils in a proportion depending on the oil obtaining process (centrifugation or solvent extraction). In olive oil obtained by centrifugation only *n*-alkanes were detected. However in olive oil extracted by second centrifugation, small amounts of UCM paraffins were detected together with the *n*-alkanes. Olive-pomace oils showed a

very variable content of both types of hydrocarbons according to the different obtaining process, such as double centrifugation, solvent extraction, or centrifugation followed by solvent extraction. "White mineral oil" used in oil extraction machinery is the source of the high concentrations of UCM paraffins found in some olive and olive-pomace oils. In the case of second centrifugation olive oil a maximum limit of $50 \text{ mg}\cdot\text{kg}^{-1}$ of UCM is suggested, whereas in the case of crude olive-pomace oil it amounts to $250 \text{ mg}\cdot\text{kg}^{-1}$ plus an additional minimum of 1.0 for the n-alkanes/UCM ratio.

Keywords: *Alkanes, Mineral Oil, Hydrocarbons, Olive-Pomace Oil, Unresolved Complex Mixture (UCM)*

Abbreviations

A/U ratio	<i>n</i> -Alkanes/UCM paraffins ratio
EC	European Union
EFSA	European Food Safety Authority
FID	Flame Ionization Detector
GC	Gas chromatography
HRGC	High Resolution Gas Chromatography
ID	Internal diameter
IS	Internal Standard
LOQ	Limit of Quantitation
SCFAH	Standing Committee on the Food Chain and Animal Health
UCM	Unresolved Complex Mixture

Introduction

Normally mineral oils comprise a mixture of saturated hydrocarbons in the range of 10 to 50 C-atoms, being branched alkanes and saturated alkyl-substituted cycloalkanes the main components. Minor amounts of *n*-alkanes, unsaturated naphthenes (alkyl-substituted cycloalkenes) and aromatic (including polyaromatic) hydrocarbons can also be found.

Mineral oil hydrocarbons may be found in foods due to contamination of diverse origins (EFSA 2012): Atmospheric exposure of raw materials (Neukom *et al.*, 2002), treatment of raw materials with pesticides, contamination during food processing with hydraulic, lubricants or mineral oils (Grob *et al.*, 1991; Fiselier and Grob, 2009; Moret *et al.*, 2009), or migration from packaging materials (Biederman and Grob, 2012; Moret *et al.*, 2012). However intentional addition of mineral oil would not be legal (Biedermann and Grob, 2009).

Lubricating or hydraulic oils for use in the food industry are named “white mineral oil” and they comprise saturated hydrocarbons only (EFSA, 2012). In all kinds of mineral oils the saturated hydrocarbon fraction is a complex mixture –called unresolved complex mixture (UCM)- not resolved by HRGC, but that yield a profile with one or various broad humps (Grob *et al.*, 1991; Grob *et al.*, 2001). Consequently, humps in the HRGC profile of the saturated hydrocarbon fraction from olive oils and olive-pomace oils may be considered markers of contamination by mineral oils.

Hydrocarbons are also to be found in natural systems in amounts not higher than 0.2% of total lipids, with the only exception of fats and oils containing greater amounts of squalene (Gómez-Coca *et al.*, 2015). In vegetable oils, aliphatic hydrocarbons are present as a homologous series of linear compounds that are

mainly saturated chains of C8-C35 atoms, of which the C21-C35 alkanes are the most abundant ones, those with odd C-atom numbers predominating. Moreover, minor amounts of n-alkenes have been isolated of which squalene is the main hydrocarbon in shark liver and olive, pumpkin, and rice bran oils (Moreda *et al.*, 2001, Gómez-Coca *et al.*, 2015). Natural hydrocarbons, when isolated and analysed by HRGC, render chromatograms consisting on well-resolved peaks where individual compounds are easily quantifiable. Linear aliphatic hydrocarbons can be used as fingerprint of a given seed oil (McGill *et al.*, 1993) and for differentiation of olive oil varieties (Guinda *et al.*, 1996; Koprivnjak and Conte, 1996). In addition, the composition of this fraction (alkanes and alkenes) has been used for the chemical detection of irradiated foods, since irradiation induces the formation of unsaturated compounds (Lesgards *et al.*, 1993).

In refined fats and oils significant amounts of unsaturated steroidal hydrocarbons coming from the dehydration of sterols and triterpenic alcohols are found together with squalene derivatives formed by cyclization (Bastic *et al.*, 1978, Lanzón *et al.*, 1994). Both types of hydrocarbons are complex mixtures which some times cannot be resolved by GC, yielding humps in the HRGC profile of the unsaturated hydrocarbon fraction. Actually, in fats and vegetable oils UCM of saturated hydrocarbons at concentrations in the range of 1-260 mg·kg⁻¹ have been reported (Wagner *et al.*, 2001; Fiorini *et al.*, 2008; Fiselier and Grob, 2009; Moret *et al.*, 2009; Biedermann *et al.*, 2009; Tranchida *et al.*, 2011). However, a severe case of contamination (as high as 3100 mg·kg⁻¹) in Ukrainian sunflower oils was detected (Biedermann and Grob, 2009) so the SCFCAH of the European Commission was requested to assess the risk related to their occurrence. This Committee agreed an acceptable level of 50 mg·kg⁻¹ of non-natural paraffins taking into account

toxicological considerations (EFSA, 2012), and the natural values found in sunflower oils (Fiselier and Grob, 2009). This value was adopted by the European Commission as legal limit for sunflower oils from Ukraine (EC, 2009).

In grape seed oils high concentrations of UCM paraffins (40-250 mg·kg⁻¹) are usually found, which is mainly attributed to the peels (Fiorini *et al.*, 2008).

In the particular case of olive oil various average levels of UCM paraffins have been found depending on the oil category. Actually it is below 4 mg·kg⁻¹ in crude olive oils (extra, virgin, and lampante olive oils), and around 14 mg·kg⁻¹ in olive oil (blend of virgin and refined olive oils), whereas it reaches 140 mg·kg⁻¹ and 230 mg·kg⁻¹ in crude olive-pomace oils obtained from the olive-pomace by centrifugation, and crude olive-pomace oils extracted by solvents, respectively (Moret *et al.*, 2003; Moret *et al.*, 2009).

The level of UCM hydrocarbons found in olive-pomace oils obtained by centrifugation (16-125 mg·kg⁻¹) or solvent extraction (100-300 mg·kg⁻¹) is surprisingly high in comparison with that in virgin olive oils. Researchers suggest (Moret *et al.*, 2003) that fresh olives contain mineral paraffins hardly extracted with the oil, meaning that they remain in the pomace. Therefore a second extraction of ageing digested olive pomace would yield a small amount of oil in which paraffins would concentrate. Besides, hexane is more efficient as extraction solvent for paraffins than triglycerides (centrifugation). We may say that the presence of paraffins in olive oil might be mostly attributed to the exposure of the fruits to atmospheric contamination or to the exhaust gas of the machinery used during harvesting. In the case of olive pomace the sources could be atmospheric contamination during storage in the pools, exhaust gas from combustion engines during transport, processing aids, and leaks of hydraulic fluids and lubricants from

the machinery utilized in extraction the plants.

The objective of this work is to identify the origin of paraffins (branched saturated *aliphatic* hydrocarbons) in olive-pomace oils and verify whether they are mainly due to the olive itself, or to atmospheric contamination or incorrect industrial practices. We also want to set up the limits from where paraffins must be considered the result of bad working practices. In order to do so, studies on olive fruits and materials used in olive-mills and extraction plants have been carried out. The determination of mineral paraffins were performed isolating the saturated hydrocarbons by column chromatography on silvered silica gel and subsequent analysis by HRGC using short silica capillary columns and FID according to the published procedure (Gómez-Coca *et al.*, 2016), complying the requirements of the EU (SCFCAH 2008).

Materials and methods

Material and reagents

Special care was taken to avoid the contamination of glassware with mineral oil. All the reagents were of analytical grade unless otherwise specified, and were: distilled water, silver nitrate (Panreac, Castellar del Vallés, Spain), and *n*-heptane (Merck, Darmstadt, Germany). A 0.05 mg/mL stock solution of *n*-eicosane (Sigma-Aldrich, Co. LLC, St. Louis, Missouri, USA) was used as IS for HRGC analysis. *n*-Hexane (95 % SpS, Romil, Cambridge, GB) was used for glass column chromatography, and a mixture of alkanes with boiling point range of 65-70 °C (Scharlau, Sentmenat, Spain) for solvent extraction. Both solvents were distilled using a Vigreux column. We checked the absence of mineral oil concentrating 200 mL hexane (1 mL IS) in a rotary evaporator down to 0.5 mL and analysing the concentrate by GC. Silica gel 60 for column chromatography, 70-230 mesh, (Merck,

ref. 7734) was used directly from the container without any treatment. Silver nitrate solutions were prepared by dissolving 4.5 g silver nitrate in 6 mL distilled water.

Samples

Samples of talc used as aid to olive oil extraction, "white mineral oils" used as hydraulic fluids, and fresh olive pomace were obtained from the Instituto de la Grasa –CSIC- olive oil mill.

Olive fruits of the Aloreña, Arbequina, Ascolano, Blanqueta, Carola, Empeltre, Frantoio, Hojiblanca, Leccino, Lechín de Granada, Lechín de Sevilla, Manzanilla, Pajarera, and Uovo di Pichone varieties were harvested by hand from an only olive grove not subjected to pest treatments, placed in a medium polluted area of Seville (southern part of Spain) in October 2010 and October 2011.

A total of 118 samples of crude and refined olive-pomace oils from Greece, Morocco, Spain, and Tunisia were provided by a local oil refinery.

Laboratory procedure to obtain olive and olive-pomace oils from olives

Olive oil and olive-pomace oil from small lots of olives were extracted using the Abencor® laboratory extraction procedure (MC2 Ingeniería de Sistemas, Sevilla, Spain) described somewhere else (Gómez-Coca *et al.*, 2013). This system simulates a commercial three-phase oil extraction system. The olives were crushed with a hammer mill equipped with a 4-mm sieve and 700 g of olive paste were processed using the system's malaxer and centrifuge. Conditions: malaxation temperature, 35 °C; malaxation time, 30 min, plus 10 min after water addition (100 mL); addition of talc (if applied). The oil/water mixture was left to decant before proceeding to oil separation. Olive pomace was collected from the centrifuge walls

Solvent extraction of paraffins from olive pomace, olive leaves, and talc

The Soxhlet extractor was utilized to extract the lipids from the olive pomace and from other solid materials. In general vegetable samples were previously dried (105 °C, 24 hours) and crushed. Extractions were carried out with distilled analytical grade alkane mixture (150 mL each). The thimbles to contain the samples were made of thick filter paper and extracted beforehand in the Soxhlet assuring the cleanness of entire apparatus. The loaded thimbles were taken to the main compartment of the Soxhlet extractor, which was then equipped with the condenser and heated to reflux during 6-8 hours. After the extraction the solvent was removed by means of a rotary evaporator, yielding the extracted compound. Sometimes the solids remaining in the thimble were again chopped and re-extracted via Soxhlet.

Olive leaves were dried at 105 °C and extracted in Soxhlet just as they are. Talc was directly extracted.

Determination of paraffins on olive and stone surfaces

Paraffins were extracted from olive peels by rinsing the fruits with n-hexane in an ultrasonic bath similar to previous procedures (Rodríguez-Acuña *et al.*, 2008): 500 g olives were placed in a 500 mL beaker containing n-hexane. Ultrasound was applied during 10 min at maximum power and room temperature. The solvent was poured into a graduated cylinder and the olives washed again in the same way. The volume of the combined extracts was measured and 100-mL aliquots spiked with 1 mL internal standard solution were transferred to amber bottles stored in the dark at -20 °C. Each aliquot was concentrated until reduced volume (~1 mL) in a rotary evaporator under vacuum and at room temperature, and the solution analysed following the same procedure as in oil samples. The LOQ was 0.05 mg of UCM per kg of olive fruits.

A 100-mL beaker containing clean olive stones and hexane was placed in an ultrasonic bath during 10 min and the hexane concentrated until reduced volume in a rotary evaporator.

Paraffin determination in olive fruit: cuticle, pulp, stone, woody shell, and seed

Olives (350-400 g) were pitted with the aid of a pitting machine and the remaining flesh from the stones was removed with a knife yielding around 200 g of a mixture of skin and pulp. The clean stones were cut down the middle using a tube cutter and the seeds were taken out with the aid of tweezers. The cuticle+pulp portions, stones, stone wood shells, and seeds were separately dried (105 °C) and crushed, yielding the samples for Soxhlet extraction with hexane.

In the case of olives from the Uovo di Pichone cultivar, whose fruits are noticeable big, the skin was removed with the help of a peeler, then dried, and extracted with hexane.

Determination of paraffins

Saturated hydrocarbons were determined according to the method described somewhere else (Gómez-Coca *et al.*, 2016) that complies with the requirements of the European Communities for determining mineral oil in vegetable oils (SCFCAH 2008). The method consists of the isolation of the fraction of saturated hydrocarbons by silver-silica column chromatography followed by HRGC-FID analysis. Briefly, silica gel (45 g is enough for three columns) was introduced in a 500 mL round-bottomed flask and the silver nitrate solution was added drop-wise with a Pasteur pipette shaking continuously. The flask was attached to a rotary-equipment, left to rotate for 30 min without vacuum, and let to stand for 12 hours before use. A glass chromatography column (50 cm long x 1.5 cm ID) provided of Teflon stopcock was packed with slurry of silver-silica gel (18.5 g) in hexane; when

it was settled the excess of solvent was discarded and the column washed with 60 mL hexane. A mixture of oil (1 g) with 1 mL of a 0.05 mg/mL n-eicosane solution was poured into the column and eluted with 60 mL hexane with a cadence of 15 drops every 10 s. The fraction was evaporated in a rotary evaporator at room temperature under vacuum, and finally until dryness with a nitrogen stream. The residue was re-dissolved in 0.5 mL heptane.

GC analyses were carried out with an Agilent 6890N Gas Chromatograph (Agilent Technologies, Santa Clara, California) equipped with an Agilent 7683B Automatic Liquid Sampler and FID. Acquisition of data was done with the Agilent ChemStation for HRGC System program. The conditions for the HRGC assays were: silica capillary column (10 m length x 0.32 mm ID x 0.1 μ m film; Sugelabor, Spain), 2.0 μ L injection volume, hydrogen carrier gas at 3.7 mL/min and cool on-column injection. The oven temperature program was: 60 $^{\circ}$ C (1 min), 12 $^{\circ}$ C/min up to 350 $^{\circ}$ C (4 min). The detector temperature was 350 $^{\circ}$ C.

To quantify the UCM of branched aliphatic and alicyclic saturated hydrocarbons, a straight baseline was manually drawn under the broad hump and then, the total area of the hump and all sharp peaks appearing on top was determined (Figure 1). Next, a new valley-valley integration of sharp peaks corresponding to natural n-alkanes was done. The difference between both areas is attributed to branched and alicyclic saturated hydrocarbons and quantified respective to the internal standard n-eicosane. The LOQ was 15 mg UCM per kg oil.

The extracts obtained from olive pomace, olive leaves, micronized talc, pulp, pits and seeds were re-dissolved in the internal standard solution and analysed in the same way.

Results and discussion

State of art in 2008-2009 olive crop

Olive oil obtained by second centrifugation in the 2008-2009 crop year together with crude and refined olive-pomace oil showed n-alkane HRGC profiles which were typical of virgin olive oil. However, most of the second centrifugation oils and all olive-pomace oils showed humps corresponding to UCM paraffins.

The results of 20 samples of second centrifugation oils showed 3 samples with less than 15 mg·kg⁻¹ (LOQ), 4 samples in the 15-50 mg·kg⁻¹ range, 12 samples in the 50-159 mg·kg⁻¹ range (mean value 119 mg·kg⁻¹), and 1 sample with 476 mg·kg⁻¹ UCM paraffins.

At the same time, 77 samples of commercial crude olive-pomace oil from different origins (Greece, Morocco, Spain, and Tunisia) were analysed. In all the cases the presence of C18-C40 UCM paraffins was noticeable (from 62 up to 967 mg kg⁻¹) with no dependence on the origin, of which the majority of the samples ranged in concentration between 150 and 400 mg kg⁻¹ (Table I). Olive pomace is the solid by-product, with high water content, obtained in olive oil mills after oil extraction. It is usually stored for variable time (0-3 months) in big outdoor pools and then processed either in the same oil mill or in an extraction plant. Olive pomace is usually subjected to a new mixing and centrifugation procedure yielding crude olive-pomace oil (second centrifugation oil) and a humid solid residue that may be processed again in the same way. Finally, the wet solid residues can be dried, crushed, and extracted with hot hexane to obtain the residual oil. In some cases concentrations of n-alkanes were higher than those of UCM (Figure 1) but the opposite situation occurred too (Figure 2).

As far as n-alkanes are concerned, significant differences were found between

Spanish (200-300 mg kg⁻¹), and Greek or Tunisian samples (450-650 mg kg⁻¹), suggesting the influence of the olive variety and/or of the different procedures for oil extraction.

The narrower UCM range (257-433 mg kg⁻¹) found in 47 samples of refined olive-pomace oils in comparison with that of the raw material is attributed to the fact of having prepared batches from different origins before refining.

Talc

Talc is the only processing aid legally admitted in olive oil extraction. It is added to the olive paste during the mixing step, in a percentage of 1-3%, and separated from the oil in the decanter together with the olive pomace. Table II shows the result on the analysis of different talc branches commonly utilized in the olive industry. In all of them the presence of paraffins becomes evident although not in important quantities. Actually, the HRGC profiles of the n-alkane series did not show odd elements (Figure 3).

In order to check the effect of talc on the paraffin content, two identical batches of olives from the Lechín de Sevilla variety were processed with and without the addition of 2% talc "Abencor 09-11" (25 mg kg⁻¹ UCM). Thereafter, the pomace was also dried and Soxhlet extracted. The olive oils obtained by centrifugation showed similar n-alkanes contents and UCM below the LOQ (Table III). Talc addition improves the yield of olive oil extraction and therefore the oil remaining in the pomace diminishes. Comparing the presence of saturated hydrocarbons in both olive-pomace oils, it seems to be higher when talc had been added, which is due to the concentrating effect in a more degreased pomace. Finally, the higher concentration of UCM paraffins in olive-pomace oil coming from a process with talc addition may be also due to the paraffin transfer from the aid. In order to check this,

comparisons should be made on the bases of the initial weight of olive fruits since it is not affected by oil yield. Actually, it seems that the 0.32 mg kg^{-1} difference was due to the paraffin contribution of talc (0.50 mg kg^{-1} referred to olive fruit).

Olive Leaves

The amount of UCM paraffins found in olive leaves was about 15 mg kg^{-1} (184 mg kg^{-1} n-alkanes). On the assumption that olives containing 1% w/w of leaves were crushed, the UCM content of olive pomace increased 0.15 mg kg^{-1} referred to olive fruit.

White mineral oil

Mineral oil named “white mineral oil” is mineral oil prepared to be used in food industries and is constituted mainly by a complex mixture of saturated hydrocarbons since unsaturated and aromatic has been eliminated. This is extensively used in olive mills and extraction plant as hydraulic liquids and machinery lubricants, so accidental contaminations may occur. The hydrocarbon content of various white mineral oils used in Spanish oil mills is shown in Table IV. The HRGC profiles show humps of saturated hydrocarbons similar to those from olive-pomace oils but the presence of the n-alkane series is negligible (Figure 4).

On the assumption that one tonne of humid olive pomace containing 2.0% of residual oil was contaminated with 5 g of “white mineral oil” containing 60% of UCM, the resultant olive-pomace oil might contain up to 150 mg kg^{-1} UCM. Therefore, white mineral oil may be a significant source of contamination.

Influence of oil extraction process

Figure 5 shows the scheme for the determination of n-alkanes and paraffins described herein.

In a large-scale olive mill olives of the Lechín de Sevilla variety were processed using a two-phase decanter with the addition of 2% talc. The olive paste (olive pomace) was dried at 105° C and divided into equal portions: one was extracted with hexane at room temperature and other in Soxhlet. Clearly, the use of solvent at high temperature is more efficient to extract total hydrocarbons, rendering oils with significant UCM content (Table V).

To verify the effect of type of obtaining processes, also experiments with the Abencor® system were carried out simulating the industrial processes. First and second centrifugation olive oils from olives of the Aloreña, Leccino, and Manzanilla cultivars were obtained meaning that after the first oil extraction (first centrifugation olive oil) the solids that remained in the centrifuge were *immediately* processed as if they were olive paste giving rise to second centrifugation olive oil. The pomaces obtained from both centrifugation processes were dried at 105° C, crushed, and taken to Soxhlet extraction, obtaining first and second centrifugation olive-pomace oils. Table VI shows that first centrifugation olive oil has high amounts of n-alkanes and negligible UCM whereas second centrifugation olive oil contains similar concentration of n-alkanes and noticeable UCM presence. First centrifugation pomace -extracted with hexane- yielded oils with higher concentration of both types of hydrocarbons than oils obtained just by centrifugation due to the major efficiency of hexane as extracting agent.

Similar UCM contents were observed when using hot hexane to extract pomace oil from diverse varieties after passing through the Abencor® system without talc (Table VII). The oils extracted with solvent from pomace subjected to two consecutive centrifugation processes showed higher UCM concentrations due to the lower yield (Table VI).

In order to study the influence of the oil content still in the pomace, Hojiblanca and Lechín olive fruits were processed in the Abencor® system and the pomaces dried to 105 °C, crushed, and extracted with hexane via Soxhlet. The solids were once more dried, crushed and extracted. Results in Table VIII indicate that in the oils obtained from a second hexane extraction of pomace, the UCM concentrations are very high and exceed those of n-alkanes because the amounts of oil is very low and a great quantity of n-alkanes are removed in the first hexane extraction.

From these results we conclude that n-alkanes are mainly contained into the fatty portion of olives whereas UCM paraffins are firmly included in the solid part of the fruit. Therefore, oils obtained by centrifugation contain lower amounts of UCM than those coming from solvent extraction. In addition, the lower the oil yield, the higher the UCM concentration.

Storage of olive pomace

To check if hydrocarbons might be generated during olive pomace storage due to some kind of fermentation process, samples were taken from a 200 kg wet pomace pool placed at open air and sheltered from the rain. Sampling was carried out directly from the surface and also at medium depth and extracted with hexane. n-Alkanes and UCM (Table IX) did not show significant changes after months, leading to the conclusion that the storage time has no influence on the paraffin content, in agreement with that previously reported (Moret *et al.*, 2003).

Hydrocarbons in olive fruit constituents: cuticle, pulp, stone woody shell, and seed

Olive fruit is essentially composed of cuticle (1-3% of dry weight), pulp (70-80%), and stone (18-22%) including the seed (2-4%). The pulp contains 15-30% oil, the seed 22-27%, and the woody shell of stone 1% maximum (Bianchi 2003). In order

to find out where exactly those hydrocarbons were located, the different parts of the olive fruit were separately analysed.

Since the olive peel could not be neatly separated from the flesh, the washings with hexane of olive fruits in an ultrasonic bath were analysed and the results (Table X) pointed towards the presence of n-alkanes and UCM paraffins on the cuticle. Therefore, hydrocarbons were determined in the cuticle of an olive variety such as Uovo di Pichone, whose fruits are big enough (about 72 olives per kg) to allow separation of the skins with very small quantities of pulp. In the oil obtained from the pulp, the n-alkane content was clearly measurable (4.87 mg kg^{-1} calculated on olive fruit), but the UCM paraffin concentration was definitely below the method's LOQ. In the extract from the cuticle we found 6.36 mg kg^{-1} n-alkanes and 2.61 mg kg^{-1} UCM paraffins, both calculated on the total weight of olive fruit. From this point on, cuticle and pulp were analysed together in the 2011 crop-year olives, taking into account that the measured UCM would mainly correspond to that contained from the cuticle (Table XI). In both hexane and cuticle + pulp extracts of olive varieties growing in the same orchard, there is no relationship between the UCM contents and the olive fruit size, in contrast to the direct relationship found between the PAHs content and olive surface (Rodríguez-Acuña *et al.*, 2008). This fact suggests that environmental contamination of the fruit surface during the growing stage is not responsible for the presence of UCM and that, at least, part of the UCM paraffins present in the cuticle are actually of endogenous origin, even though absorption or deposition of atmospheric contaminants may contribute to the total content.

The hexane washing of stones coming from the Arbequina variety yielded 2.24 mg kg^{-1} and 1.65 mg kg^{-1} of n-alkanes and UCM paraffins, respectively (both

calculated on stone weight), supporting the hypothesis of the endogenous origin of both kinds of hydrocarbons.

In the hexane extracts obtained by Soxhlet from the stone woody shells, the n-alkanes/UCM ratios (Table XII) were lower than those from olive washings and cuticle + pulp fractions. The amounts of hydrocarbons in seeds (Table XIII) were lower than in woody shells, although the A/U ratios were higher. These results were consistent with those determined in stones (woody shell plus seed) coming from olives gathered in the 2011 crop year (Table XIV).

It was then deduced that cuticle + pulp portions showed the highest UCM concentration although the results lack accuracy due to the fact that the UCM concentrations lie near to the LOQ of the method (the amount of oil in the sample is large). Tentatively the 4.75 mg kg^{-1} value of the Ascolano cultivar may be considered as maximum and the 2.45 mg kg^{-1} as average value. The maximum UCM content in stone is that of the Empeltre cultivar (1.82 mg kg^{-1}), being the average value 0.76 mg kg^{-1} . Therefore, 6.57 mg kg^{-1} is the maximum UCM paraffin concentration found in the entire fruit being 3.21 mg kg^{-1} the average concentration.

To summarize, n-alkanes and UCM paraffins are present in cuticle, woody shell, and seed of olives, being the former greater than the latter. The n-alkane profiles are similar in all parts of olives. Pulp contains n-alkanes but UCM paraffins are absent or in a very low concentration. In entire olives the concentrations of both types of hydrocarbons and the A/U ratio depend on the olive variety. During the process to obtain olive oil, by pressing or centrifugation, a greater part of n-alkanes passes to a significant amount of oil whereas the UCM paraffins remain totally or mainly in the solid residue and, consequently, the olive oil shows negligible concentration of UCM paraffins.

Pomace oil, containing the remaining n-alkanes and the greatest part of UCM paraffins, can be extracted by mixing followed by one or several consecutive centrifugation steps, or subjecting the dried residue to extraction with hexane. Moreover, the residue obtained from centrifugation of the olive pomace can be dried and extracted with hexane. In all cases, both hydrocarbon types are transferred to the pomace oil in amounts that depend on the fruit variety, the oil depletion of the solid residue, the efficiency of the extraction procedure, and the use of talc. Consequently, the hydrocarbon content and the A/U ratio in olive-pomace oil is very variable: If hexane extraction is used, major quantity of both hydrocarbons will be found. If the solid residue is poor in oil, the pomace oil will show a low A/U ratio.

In second centrifugation olive oils a maximum concentration of 50 mg kg^{-1} of UCM paraffins is suggested because it is the limit adopted by EU for sunflower oil (EC, 2009). This is quantifiable by simple analytical methods with sufficient reliability, and includes the values found in these types of oils (Table VI).

In olive-pomace oils the presence of UCM paraffins is usual even if the oils have been produced under controlled conditions. This is due to the passage from the olive residue to the oil as a result of different and consecutive extraction processes.

Since it is not possible to take into account all the factors that influence the presence of UCM paraffins in olive-pomace oil, a value of 250 mg kg^{-1} is suggested as maximum content for oils obtained following the normal extraction procedures. This UCM concentration is higher than the value (194 mg kg^{-1}) obtained adding up the average contents of olive fruit (3.21 mg kg^{-1}), talc (0.50 mg kg^{-1}), and leaves (0.15 mg kg^{-1}) assuming that all the UCM is transferred to a minimum of 2% of olive-pomace oil. Consequently, UCM paraffin concentrations above this value would clearly indicate contamination being white mineral oil the most probable source.

Paraffin values below 250 mg kg⁻¹ may be due to a minor content of the olive fruit increased by contamination with mineral oil. In such cases, a minimum of 1.0 for the A/U ratio must be required because this is a value slightly lower than those of oils obtained by solvent extraction from olive pomaces subjected to earlier centrifugation (Table VI) and the white mineral oil does not contain noticeable amounts of n-alkanes.

These limits are compatible with the values found nowadays in the olive-pomace oil from market in contrast with a lot of oil samples analysed during the 2008-2009 crop year (Table I). This fact indicates that a quality policy has been adopted in oil extraction industries to prevent the contamination.

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Table I. Content of n-alkanes and quantification of the unresolved complex mixture (UCM) of paraffins in crude olive-pomace oils obtained during the 2008-2009 crop year.

UCM (mg kg ⁻¹)	Number of samples									
	62-100	100-150	150-200	200-250	250-300	300-350	350-400	400-500	500-600	>600
Samples from Spain. n-Alkanes (mg kg ⁻¹)*	3 (225)	5 (240)	8 (226)	9 (220)	13 (263)	11 (291)	8 (283)	2 (276)	2 (201)	1 (361)
Samples from Greece. n-Alkanes (mg kg ⁻¹)*	1 (721)	1 (710)	2 (542)	2 (588)	2 (600)	0	2 (580)	0	0	1 (668)
Samples from Tunisia. n-Alkanes (mg kg ⁻¹)*		1 (480)	2 (471)							
Samples from Morocco. n-Alkanes (mg kg ⁻¹)			1 (319)							
Total	4	7	13	11	15	11	10	2	2	2

(*) Mean values

Table II. Aliphatic hydrocarbons content in commercial talc utilized as aid in the olive industry extracted with hexane in Soxhlet. UCM = unresolved complex mixture of paraffins.

Talc samples	n-Alkanes mg kg ⁻¹	UCM mg kg ⁻¹	Range	Maximum
Talcoliva (2010)	4.0	27	C14-C54	C29
Talc LNA-M	3.3	< 0.4	C16-C54	C29
Talcoliva M (2011)	2.8	21	C14-C50	C29
Talc (olive mill)	3.2	25	C12-C54	C35
Talc (Abencor 09-11)	2.8	25	C12-C54	C22, C35
Talc (Abencor 09-11)*	1.5	< 0.4	C14-C54	C17, C35

*n-Hexane extraction at room temperature, during 10 min in ultrasonic bath.

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Table III. Hydrocarbon concentration in olive oil and in olive pomace oil obtained from fruits of the Lechín de Sevilla variety, with and without the addition of 3% talc. Olive oils were extracted through centrifugation in Abencor® system whereas pomace was taken to solvent extraction in Soxhlet. UCM = unresolved complex mixture of paraffins

		Oil yield ^a %	Saturated hydrocarbons			
			Oil (mg kg ⁻¹)		Olives (mg kg ⁻¹) ^a	
			<i>n</i> -Alkanes	UCM	<i>n</i> -Alkanes	UCM
Olive oil (Abencor®)	No talc	15.6	44	<15	6.86	NQ
Olive oil (Abencor®)	Talc	16.6	41	<15	6.81	NQ
Olive-pomace oil (Soxhlet)	No talc	3.9	161	27	6.28	1.05
Olive-pomace oil (Soxhlet)	Talc	2.8	229	49	6.41	1.37

^aValues refer to olives at origin.

NQ = non-quantifiable.

Table IV. Hydrocarbon content of “white mineral oils” used in olive oil industries.

UCM = unresolved complex mixture of paraffins.

White mineral oil	UCM (%)	Range	Maximum
Fluid 1	76.0	C14-C47	C22
Fluid 2	64.0	C21-C47	C35
Viscous 1	48.8	C27-C54	C35
Viscous 2	32.7	C27-C54	C35

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Table V. Hydrocarbon concentration in olive oil and in olive pomace obtained from fruits of the Lechín de Sevilla variety, with the addition of 2% talc, in industrial plant.

Olive oil was obtained by centrifugation whereas two portions of pomace paste were taken either to cold or to hot solvent extraction. UCM = unresolved complex mixture of paraffins

Oil	Oil yield on dried pomace %	Saturated hydrocarbons mg kg ⁻¹	
		n-Alkanes	UCM
Olive oil (centrifugation)		46	< 15
Olive-pomace oil (n-hexane extraction, room temperature)	5.8	67	< 15
Olive-pomace oil (Soxhlet extraction)	8.0	373	78

Table VI. Hydrocarbon content of oils obtained through the Abencor® system from olives of different varieties using 2% talc. UCM = unresolved complex mixture of paraffins.

Cultivar	Oil yield ^a %	First centrifugation olive oil				A/U ^b
		mg kg ⁻¹ oil		mg kg ⁻¹ olives ^a		
		n-Alkanes	UCM	n-Alkanes	UCM	
Aloreña	10	23	< 15	2.30	NQ	>1.5
Leccino	12	127	< 15	15.20	NQ	>1.8
Manzanilla	15	26	< 15	3.90	NQ	>2.3
Second centrifugation olive oil						
Aloreña	0.6	37	22	0.22	0.13	2.0
Leccino	1.0	128	29	1.28	0.29	4.4
Manzanilla	1.0	35	< 15	0.35	NQ	>1.5
Oil extracted with solvent from 1 st centrifugation pomace						
Aloreña	3.5	94	42	3.29	1.47	2.2
Leccino	3.0	145	124	4.64	3.72	1.2
Manzanilla	3.1	123	58	3.81	1.80	2.1
Oil extracted with solvent from 2 nd centrifugation pomace						
Aloreña	2.8	105	81	2.94	2.27	1.3
Leccino	2.1	219	155	4.60	3.25	1.4
Manzanilla	2.0	148	61	2.96	1.22	2.4

^aValues refer to the olives at origin.

^bA/U = ratio between n-alkanes and UCM paraffins.

NQ = non-quantifiable.

Table VII. Hydrocarbon content in olive-pomace oils obtained via Soxhlet extraction from first centrifugation pomace coming from olives processed by the Abencor® system without talc. The corresponding data in the case of stones are also given.

UCM = unresolved complex mixture of paraffins.

Cultivar	Oil extracted with solvent from 1 st centrifugation pomace			Stones		
	Oil yield ^a %	mg kg ⁻¹ oil		mg kg ⁻¹ olives ^a		A/U ^b
		n-Alkanes	UCM	n-Alkanes	UCM	
Frantoio	11.4	241	40	27.50	4.56	6.0
Lechín de Granada	3.4	181	35	6.15	1.19	5.2
Blanqueta	5.8	272	20	15.80	1.16	13.6
Carolea	4.0	290	34	11.60	1.36	8.5
Hojiblanca	4.0	75	20	3.00	0.80	3.8
Ascolano	7.4	111	22	8.21	1.62	5.1
Uovo de Pichone	3.4	122	28	4.15	0.95	4.4

^aValues refer to the olives at origin.

^bA/U = ratio between n-alkanes and UCM.

Table VIII. Hydrocarbon content in olive pomace oils obtained by first and second Soxhlet extraction from pomaces obtained by the Abencor® system. UCM = unresolved complex mixture of paraffins

Cultivar	Oil yield ^a %	1st Solvent extraction of olive pomace				A/U ^b
		mg kg ⁻¹ oil		mg kg ⁻¹ olives ^a		
		n-Alkanes	UCM	n-Alkanes	UCM	
Hojiblanca	4.1	96	20	3.94	0.82	4.8
Lechín de Sevilla	5.6	183	33	10.24	1.85	5.5
2nd Solvent extraction of olive pomace						
Hojiblanca	0.09	68	360	0.06	0.33	0.19
Lechín de Sevilla	0.06	160	486	0.10	0.31	0.33

^aValues refer to the olives at origin.

^bA/U = ratio between n-alkanes and UCM paraffins.

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Table IX. Saturated hydrocarbon content in olive-pomace oils obtained by hexane extraction from olive pomace at different storage times.

Sampling	Oil yield on dried pomace %	Saturated hydrocarbons		
		n-Alkanes (mg kg ⁻¹)	UCM (mg kg ⁻¹)	A/U ^a
27/02/2010 Surface of pile	12.5	302	126	2.40
27/02/2010 Interior of pile	13.8	271	114	2.38
28/07/2010 Surface of pile	13.0	290	115	2.52
28/07/2010 Interior of pile	13.5	276	122	2.26

^aA/U = ratio between n-alkanes and UCM paraffins.

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Table X. Hydrocarbon content on olive fruit surface obtained from hexane washings of various olive varieties

Cultivar	Fruits per kg	Hydrocarbons in hexane washings mg kg ⁻¹ olives ^a		
		n-Alkanes	UCM	A/U ^b
Crop year: 2010				
Blanqueta	502	3.00	0.53	5.7
Arbequina	434	6.25	1.03	6.1
Pajarera	220	1.41	0.29	4.9
Ascolano	131	1.94	0.39	4.9
Crop year: 2011				
Leccino	492	2.58	0.26	10.3
Manzanilla	209	3.27	0.28	11.7
Aloreña	158	1.30	0.18	7.2

^aValues refer to olives at origin.

^bA/U = ratio between n-alkanes and UCM paraffins.

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Table XI. Hydrocarbon content in the cuticle+pulp fraction -as a whole- in olive fruits coming from different olive varieties, obtained through Soxhlet extraction. Crop year: 2011.

Cultivar	Fruits per kg	Oil Yield ^a %	Hydrocarbons in cuticle+pulp fractions				A/U ^b
			mg kg ⁻¹ oil		mg kg ⁻¹ olives ^a		
			n-Alkanes	UCM	n-Alkanes	UCM	
Leccino	492	8.6	76	16	6.53	1.38	4.7
Frantoio	472	11.9	164	16	19.52	1.90	10.3
Lechín	430	16.6	88	20	14.54	3.32	4.4
Granada	410	10.8	152	29	16.42	3.13	5.3
Empeltre	379	11.3	167	19	18.87	2.15	8.8
Blanqueta	379	11.3	167	19	18.87	2.15	8.8
Carolea	279	14.8	63	15	9.32	2.22	4.2
Hojiblanca	244	11.5	69	18	7.93	2.07	3.8
Manzanilla	209	10.1	85	15	8.59	1.52	5.7
Aloreña	158	9.6	36	< 15	3.46	NQ	> 2.4
Ascolano	98	14.4	104	33	14.98	4.75	3.2
Uovo di Pichone	72	11.0	54	19	5.94	2.09	2.7

^aValues refer to the olives at origin.

^bA/U = ratio between *n*-alkanes and UCM paraffins.

NQ: non-quantifiable.

Table XII. Hydrocarbon content in the woody shells of olive stones from various olive varieties, obtained through Soxhlet extraction. Crop year: 2010.

Cultivar	Oil Yield ^a %	Hydrocarbons in woody shell of stones mg kg ⁻¹ oil		mg kg ⁻¹ olives ^a		A/U ^b
		n-Alkanes	UCM	n-Alkanes	UCM	
Blanqueta	0.55	301	106	1.66	0.64	2.8
Arbequina	0.58	128	64	0.74	0.37	2.0
Lechín de Sevilla	0.29	347	133	1.01	0.39	2.6
Pajarera	0.33	312	128	1.02	0.43	2.4
Ascolano	0.23	194	157	0.45	0.36	1.2

^aValues refer to the olives at origin.

^bA/U = ratio between n-alkanes and UCM paraffins.

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Table XIII. Hydrocarbon content in seeds coming from different olive varieties, obtained through Soxhlet extraction. Crop year: 2010.

	Oil yield ^a %	Hydrocarbons in seeds				
		mg kg ⁻¹ oil		mg kg ⁻¹ olives ^a		A/U ^b
		n-Alkanes	UCM	n-Alkanes	UCM	
Blanqueta	0.75	148	43	1.11	0.27	3.4
Arbequina	0.70	72	16	0.50	0.11	4.5
Lechín de Sevilla	0.40	163	44	0.64	0.18	3.7
Pajarera	0.48	123	25	0.60	0.12	4.9
Ascolano	0.38	163	48	0.62	0.18	3.4

^aValues refer to the olives at origin.

^bA/U = ratio between n-alkanes and UCM paraffins.

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Table XIV. Hydrocarbon content in stones (woody shell plus seed) coming from various olive varieties, obtained through Soxhlet extraction. Crop 2011

Cultivar	Oil yield ^a %	Hydrocarbons in stones				
		mg kg ⁻¹ oil		mg kg ⁻¹ olives ^a		
		n-Alkanes	UCM	n-Alkanes	UCM	A/U ^b
Frantoio	1.02	469	41	4.78	0.42	11.3
Lechín de Granada	1.16	166	69	1.93	0.80	2.4
Empeltre	1.94	141	94	2.74	1.82	1.5
Blanqueta	1.13	244	84	2.75	0.94	2.9
Carolea	0.99	180	31	1.79	0.31	5.8
Hojiblanca	0.90	243	48	2.18	0.44	5.1
Ascolano	0.56	241	105	1.35	0.59	2.3
Uovo de Pichone	0.72	220	103	1.59	0.74	2.1

^aValues refer to the olives at origin.

^bA/U = ratio between n-alkanes and UCM.

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Figure captions

Figure 1. HRGC profile of saturated hydrocarbons from crude olive-pomace oil with n-alkane content = 271 mg kg⁻¹ and UCM content = 114 mg kg⁻¹: (1) C20:0 (Internal standard); (2) C21:0; (3) C22:0; (4) C23:0; (5) C24:0; (6) C25:0; (7) C26:0; (8) C27:0; (9) C28:0; (10) C29:0; (11) C30:0; (12) C31:0; (13) C32:0; (14) C33:0; (15) C34:0; (16) C35:0. UCM = Unresolved Complex Mixture of paraffins.

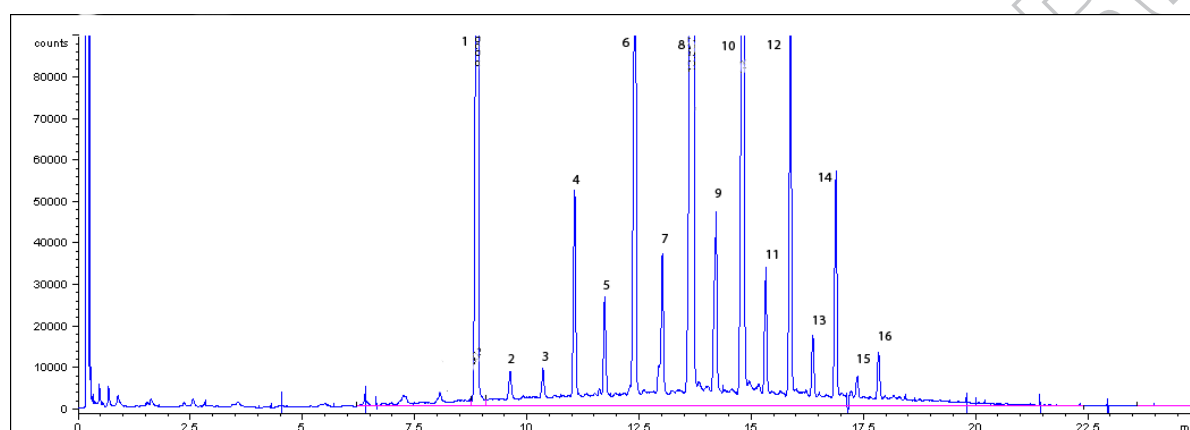


Figure 2. HRGC profile of saturated hydrocarbons from crude olive-pomace oil with n-alkane content = 59 mg kg⁻¹ and UCM content = 340 mg kg⁻¹. Peaks identification as in Figure 1. UCM = Unresolved Complex Mixture of paraffins.

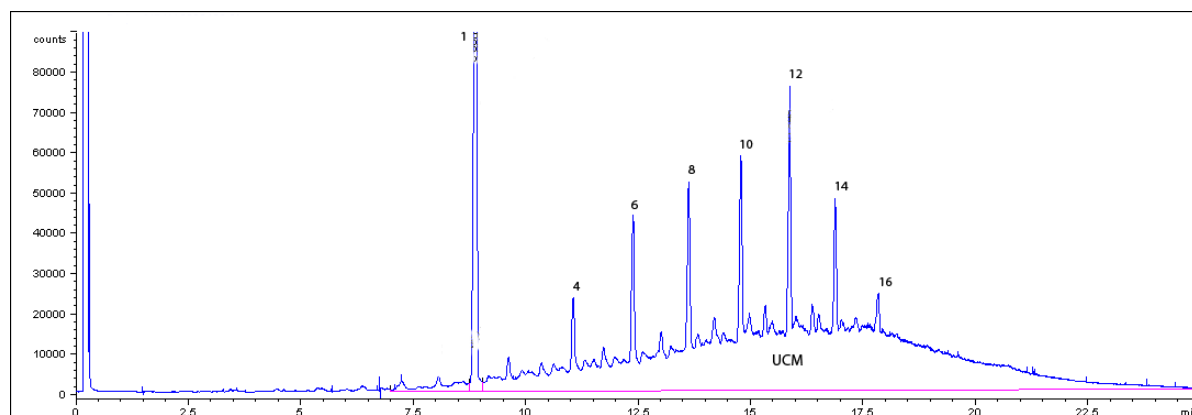


Figure 3. HRGC profile of saturated hydrocarbons from talc. Peaks identification as in Figure 1. UCM = Unresolved Complex Mixture of paraffins.

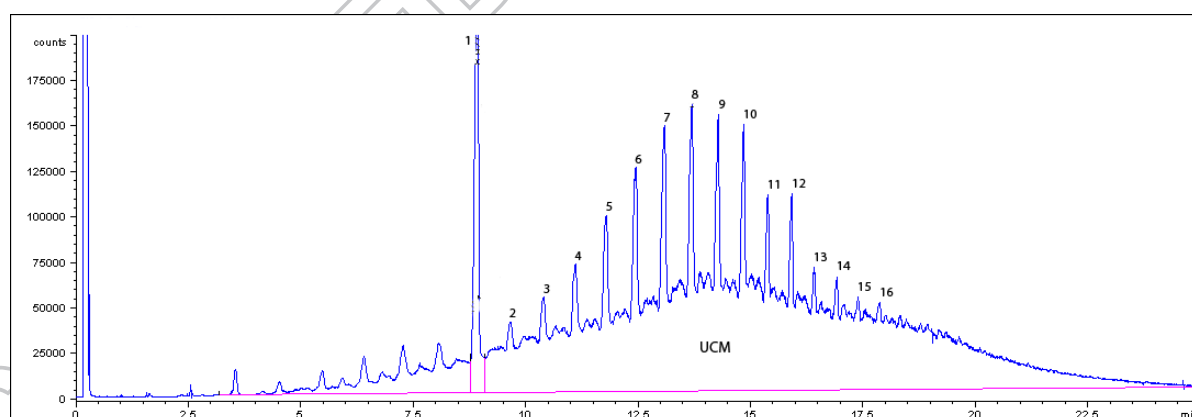


Figure 4. HRGC profile of saturated hydrocarbons from “white mineral oil”. IS = C20, internal standard.

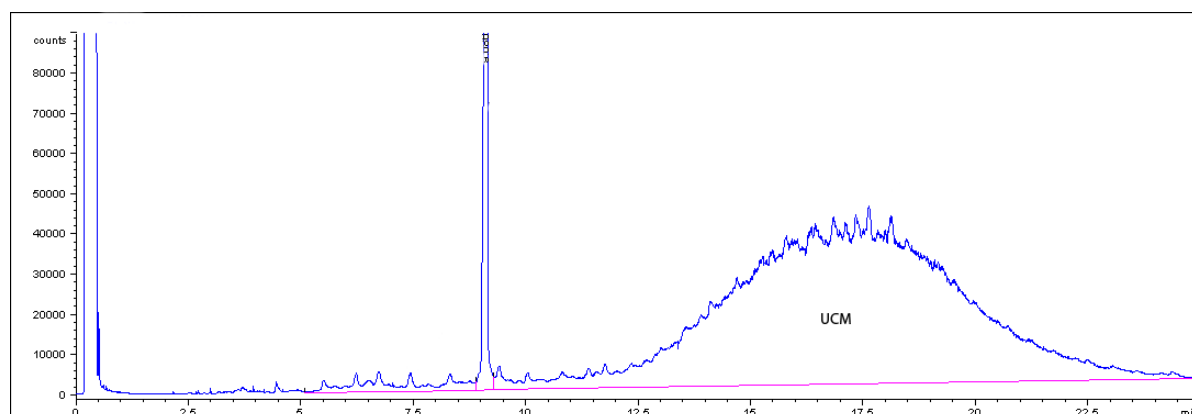
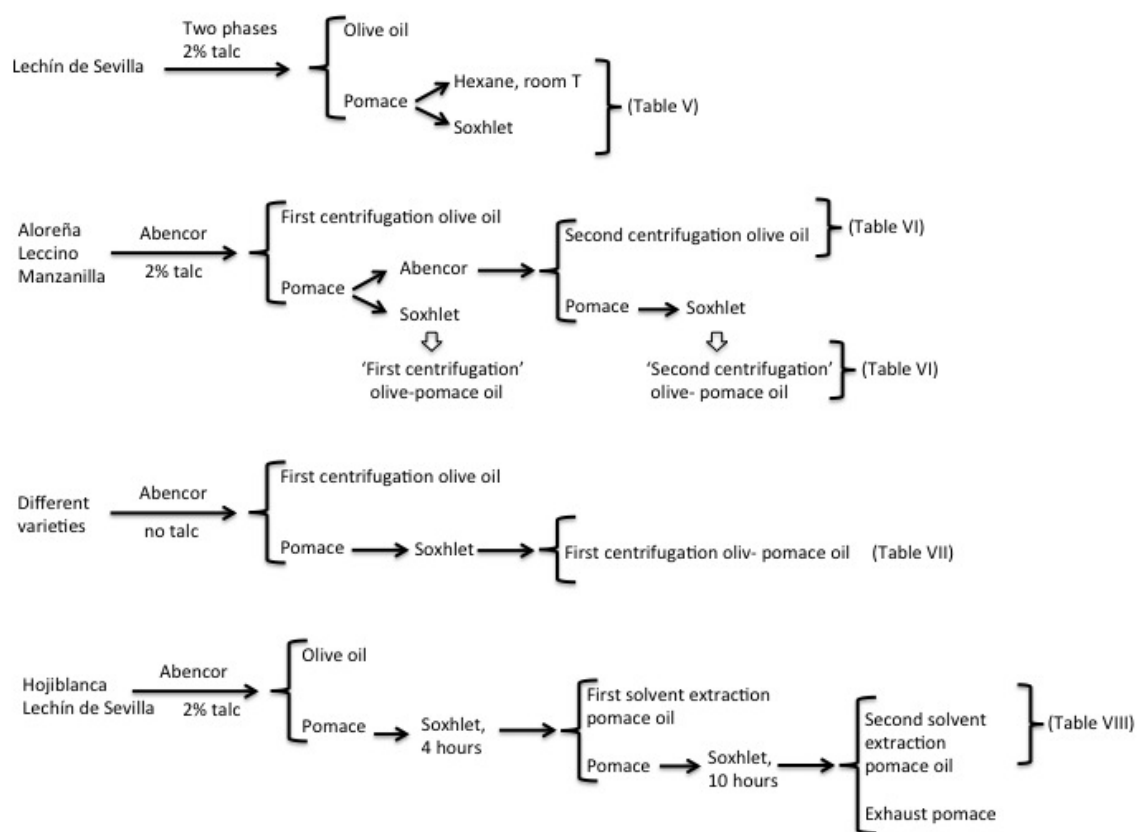


Figure 5. Scheme followed for the determination of n-alkanes and paraffins in olive-pomace oil according to different extraction treatments.



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