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# Aroma characterization of various apricot varieties using headspace–solid phase microextraction combined with gas chromatography–mass spectrometry and gas chromatography–olfactometry

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#### Abstract

The characterization of the aromatic profile of several apricot cultivars with molecular tracers in order to obtain objective data concerning the aromatic quality of this fruit was undertaken using headspace–solid phase microextraction (HS–SPME). Six apricot cultivars were selected according to their organoleptic characteristics: Iranien, Orangered, Goldrich, Hargrand, Rouge du Roussillon and A4025. The aromatic intensity of these varieties measured by HS–SPME–Olfactometry were defined and classified according to the presence and the intensity of grassy, fruity and apricot like notes. In the six varieties, 23 common volatile compounds were identified by HS–SPME–GC–MS. Finally, 10 compounds, ethyl acetate, hexyl acetate, limonene,  $\beta$ -cyclocitral,  $\gamma$ -decalactone, 6-methyl-5-hepten-2-one, linalool, b-ionone, menthone and (E)-hexen-2-al were recognized by HS–SPME–GC–O as responsible of the aromatic notes involved in apricot aroma and considered as molecular tracers of apricot aromatic quality which could be utilized to discriminate apricot varieties.

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Keywords: Apricot; Aromatic quality; Tracers; HS–SPME–O; HS–SPME–GC–MS; HS–SPME–GC–O

# 1. Introduction

Several studies concerning the volatile fraction of apricot showing a great variability concerning the nature and the concentration of the aroma compounds isolated from different cultivars are available ([Crouzet,](#page-7-0) Etiévant, & Bayonove, 1990; Takeoka, Flath, Teranishi,

[& Guentert, 1990](#page-7-0)). However, studies devoted to volatile compounds involved in apricot flavour are scarce ([Chai](#page-7-0)[rote, Rodriguez, & Crouzet, 1981; Guichard, Schlich, &](#page-7-0) [Issanchou, 1990; Takeoka et al., 1990; Toth-Marcus,](#page-7-0) [Boross, Blazso, & Kerek, 1989\)](#page-7-0). Sensory descriptive analysis indicated that lactones were involved in the typical, basic apricot flavour, whereas several compounds, like terpene alcohols, 2-phenylethanol,  $\beta$ -ionone and hexyl acetate and in some cases benzaldehyde were described as contributors of the flower and fruity notes of different apricot cultivars.

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<span id="page-1-0"></span>Several techniques were used for these determinations, correlation between sensory and instrumental data [\(Chairote et al., 1981; Guichard et al., 1990](#page-7-0)) or odour unit concept [\(Takeoka et al., 1990; Toth-Marcus](#page-8-0) [et al., 1989](#page-8-0)). However, at the best of our knowledge, no recent work using gas chromatography coupled to olfactometry (GC–O) technique was undertaken for apricot aroma characterization ([Acree, Barnard, & Cunning](#page-7-0)[ham, 1984; Da Silva, Lundhal, & Mc Daniel, 1994; Ul](#page-7-0)[rich & Grosch, 1987](#page-7-0)). One of the most important problems in olfactometry studies is that of the extract representativeness (Etiévant et al., 1994; Reinecius, [2000\)](#page-7-0). solid phase microextraction (SPME) constituted a simple, solvent-free method for the isolation and the concentration of the volatile compounds present in the headspace without modifications of these compounds due to temperature or solvent effect ([Harmon, 1997;](#page-7-0) [Pawliszyn, 2001](#page-7-0)). SPME was used for analysis of several flavour compounds in several food products, and more particularly in fruits [\(Azodanlou, Darbellay, Luisier,](#page-7-0) Villetaz, & Amadò, 2003; Beaulieu & Grimm, 2001; [Riu-Aumatel, Castellari, Lopez-Tamames, Galassi, &](#page-7-0) [Buxaderas, 2004; Song, Fan, & Beaudry, 1998; Steffen](#page-7-0) [& Pawlizyn, 1996; Yang & Peppard, 1994\)](#page-7-0). Moreover, SPME could be used to determine, by GC–O ([Bezman,](#page-7-0) [Rouseff, & Naim, 2001; Deibler, Acree, & Lavin, 1999;](#page-7-0) [Frank, Owen, & Patterson, 2004\)](#page-7-0), the contribution of volatile compounds to apricot aroma.

The aim of the present work was to characterize the aroma of apricot cultivars using SPME coupled with GC–MS and with GC–O in order to identify volatile compounds with olfactive impact.

# 2. Materials and methods

# 2.1. Plant material

Apricot cultivars, of different precocity were gathered during the 2000 season at full maturity in Institut National de la Recherche Agronomique orchards of Avignon and Melgueil. Six cultivars recognized as having the typical aroma (Robini, 2000 Personal communication) or possessing an interest according to their matury period were selected for this study. Orangered and Iranien were precocious apricots; Goldrich, half season apricot; Hargrand, Rouge du Roussillon and one hybrid A 4025, full season apricot. Apricot were utilized in samples as pulp pieces or purée obtained by crushing.

## 2.2. Standard mixture

Target aroma compounds previously reported to be key compounds for apricot flavour were selected: β-ionone, linalool, benzaldehyde, 2-phenyl ethanol,  $\gamma$ -decalactone and hexyl acetate [\(Chairote et al., 1981;](#page-7-0) [Guichard et al., 1990; Takeoka et al., 1990; Toth-Mar](#page-7-0)[cus et al., 1989](#page-7-0)). Their volatility, corresponding to the Henry constant =  $\gamma_i \times \text{ps}_i$  calculated at 40 °C from previously reported infinite dilution activity  $(\gamma_i)$  data [\(Baudot](#page-7-0) [& Marin, 1997; Fichan, Larroche, & Gros, 1999;](#page-7-0) [Sadafian & Crouzet, 1987; Voilley, Fares, Lorient, &](#page-7-0) [Simatos, 1988](#page-7-0)) and vapour pressure determined at 40 °C according to the Physical and Chemical Handbook ([Weast, 1972\)](#page-8-0) were given in Table 1. It was postulated that the variation of  $\gamma_i$  with temperature could be neglected in the range  $25-40$  °C [\(Bomben, Bruin, This](#page-7-0)[sen, & Merson, 1973](#page-7-0)).

The temperature of 40  $\degree$ C was chosen for equilibrium temperature according to the literature data [\(Jia, Zhang,](#page-7-0) [& Min, 1998\)](#page-7-0) and preliminary determinations.

An aqueous model solution with 1% of absolute ethanol including these six compounds (Sigma, St. Louis, MO, USA) at concentrations near of those found in Rouge du Roussillon apricot variety ([Guichard & So](#page-7-0)[uty, 1988](#page-7-0)) was used for determination of the extraction time and for the choice of the fibre (Table 1).

Calibrations curves were also made with standard solutions of each compound at four different concentrations between 1 and 50 ppm in ethanol. These calibration curves were all linear in this range and allow to

Table 1





<sup>a</sup> [Baudot and Marin \(1997\).](#page-7-0)<br><sup>b</sup> [Voilley et al. \(1988\)](#page-8-0).<br><sup>c</sup> [Sadafian and Crouzet \(1987\)](#page-8-0).

<sup>d</sup> [Fichan et al. \(1999\).](#page-7-0)

give concentration of compound on fibre for the SPME extraction conditions used.

# 2.3. HS–SPME conditions

The SPME device and the fused silica fibres coated with polydimethylsiloxane (PDMS) with  $100$ - $\mu$ m thickness, polydimethylsiloxane/divinylbenzene (PDMS/ DVB) 65  $\mu$ m and Carboxen/polydimethylsiloxane (CAR/PDMS) 75 um, were used all obtained from Supelco Co. (Bellefonte, PA, USA). The fibres were conditioned prior use according to supplier's prescriptions,  $0.5$  h at 250 °C for PDMS and PDMS/DVB and 1 h at 300 °C for CAR/PDMS.

Seven mL of standard mixture or apricot purée were placed in a 20 mL vial; 50 g of apricot pulp (about 20 mL) were placed in a 60 mL vial. Vials were hermetically closed by a teflon/silicone septum and equilibrated at  $40^{\circ}$ C for  $40$  min. The headspace sampling was performed at the same temperature for 20 min. The desorption of the analytes from the fibre coating was made in the injection port of GC at  $250\text{ °C}$  during 4 min. For each sample four SPME extractions and desorptions were realized.

### 2.4. Gas chromatography

A Varian 3300 (Walnut Creek, CA, USA) gas chromatograph fitted with a FID and a  $30 \text{ m} \times 0.25 \text{ mm}$ , 0.25-um film thickness DB-WAX (J&W Scientific, Folson, CA, USA) fused silica capillary column was used. The temperature of the injector was held at  $250^{\circ}$ C during analysis. The oven temperature was programmed from 60 to 200 °C at 5 °C/min, then to 250 °C at 6 °C/ min and held at this temperature for 5 min. Hydrogen at 1.8 mL/min was used as carrier gas.

A Combipal (CTC Analytics, Swingen, Switzerland) automatic sampler was used for analysis of the model solution.

Linear retention indices (RI) of the compounds were calculated using a serie of n-alkanes (C8–C32) (Sigma, St. Louis, MO, USA) injected in the same conditions.

## 2.5. Gas chromatography–mass spectrometry

A chromatograph Hewlet–Packard 5989 (Palo Alto, CA, USA) coupled to a quadrupole mass spectrometer Hewlet–Packard 5890 fitted with the same DB-Wax column and operated in the conditions used for GC analysis were used. Helium at 1.5 mL/min was used as carrier gas. The transfer line was 250 °C. Electron impact mass spectra were scanned at 70 eV in the  $m/z$  range 60–600 mass units and then compared with those present in user generated (INRA Mass, Dijon, France) or commercial libraries (Registry of Mass Spectral Data with Structures, Wiley, NY, USA).

#### 2.6. Gas chromatography–olfactometry

A chromatograph operated in the same conditions as those used for GC determination was equipped with a SGE sniffing port (SGE, Villeneuve Saint-Georges, France). At the end of the column, the gas flow was split into two equal parts according to the small amount injected through SPME: one part going to the FID and the other going to the sniffing port. The split occurred through an SGE capillary splitter connected to two fused silica deactivated capillary tubing of the same length. Purified air saturated with water at a 25 mL/ min flow was combined to the GC effluent to avoid dehydration of the nasal mucous membrane.

The effluents were analysed by three trained panelists which were requested to assign odour properties and intensity to each volatile compound detected. The panelists previously defined the vocabulary used for aroma descriptors after training with apricot extracts. The respective retention times and intensity were recorded manually. Panelists rated the intensity of aroma on a six-point sliding scale: the score of 0 is given if no aroma is perceived and 5 for intense aroma.

Moreover, the three trained panelists verified the representativeness of SPME extracts by direct SPME– Olfactometry. A short deactivated fused silica column was used in the place of the chromatography column. The panelists gave descriptors of global aroma with their intensity on a six-point sliding scale. The intensity of overall SPME–O responses were obtained by the FID integrated signal.

# 3. Results and discussion

### 3.1. Determination of the extraction time

A PDMS fibre was used at  $40^{\circ}$ C for the extraction time determination after 40 min of equilibrium to achieve the partition equilibration of volatile compounds between the sample and the headspace. The concentrations and coefficients of variation of key compounds were calculated according to calibration curves. The coefficients of variation ranged from 1.1% to 3.8%. These results indicated that SPME–GC under the analytical conditions used was appropriate for the analysis of flavour compounds in apricot. Calculated concentrations showed that the SPME extraction allow a concentration of compounds on fibre ([Fig. 1](#page-3-0)) according to contents of standard solution ([Table 1\)](#page-1-0). The evolution of compound concentration after SPME extraction ([Fig. 1](#page-3-0)) showed that the equilibrium of extraction was obtained after about 20 min for hexyl acetate, benzaldehyde and linalool. For  $\gamma$ -decalactone and b-ionone the equilibrium was not reached for 40 min but between 20 and 40 min the concentration

<span id="page-3-0"></span>

Fig. 1. SPME extraction of the model solution compounds performed at  $40 °C$  with PDMS fibre.

did not significantly increased. However, 2-phenylethanol was not extracted in detectable amount. This fact was probably the result of the low concentration of this compound in the solution, 0.25 ppm and of its low volatility (Henry constant =  $1.97 \times 10^4$  Pa) relatively to benzaldehyde, linalool and b-ionone [\(Table 1\)](#page-1-0). According to these results, an extraction time of 20 min at 40  $^{\circ}$ C was finally retained.

The release of volatile compounds from apricot purée or apricot pieces is different to release of volatile compounds from aqueous solution. In the case of apricot samples the release is hindered by the interactions occurring between volatiles and matrix constituents. Moreover, the mass transfer resistance is different in water and in apricot samples. However, the chromatogram obtained after extraction of apricot fruit (cv Rouge du Roussillon) by SPME used in the conditions reported above was qualitatively similar of those obtained using other headspace extraction methods (Chairote et al., 1981) and the coefficient of variation of apricot sample extraction (sum of peak area) did not exceed 4.0%. The SPME conditions obtained on model solution were appropriate to the apricot samples.

# 3.2. Choice of fibre

The concentration of compounds extracted by PDMS, PDMS/DVB and Carboxen/PDMS fibres, reported in Fig. 2 showed that hexyl acetate, benzaldehyde, linalool and  $\beta$ -ionone were extracted in more important quantities by Carboxen/PDMS than by the other fibres. Conversely PDMS fibre is more convenient for  $\gamma$ -decalactone extraction. The three fibres in these conditions did not extract 2-phenyl ethanol. Therefore,



Fig. 2. Comparison of different SPME fibres for extraction of model solution compounds after 20 min at 40  $^{\circ}$ C.

Carboxen/PDMS fibre allowed a good compromise for the extraction of the aroma compounds present in the model solution and belonging to different series of compounds.

# 3.3. Extract representativeness and characteristics

The representativeness of extract was checked by the three panelists by comparison between each extract and the corresponding apricot purée: a sample of apricot purée equilibrated at  $40^{\circ}$ C was sniffed directly by the panelists; then sniffing of extract was performed by GC–O using a short inactivated silica column in place of chromatography column. Moreover, two categories of results were obtained by GC–O.

Firstly, quantitative data were obtained for the several apricot cultivars by integration of the FID signal giving intensity of the overall response (one peak) (Table 2). The most important quantities of aroma compounds were extracted using SPME from Iranien, Hargrand and Rouge du Roussillon. These results showed that there is no correlation between aroma intensity and harvesting date of the variety: Iranien, which is a precocious variety gave the highest signal intensity.

Table 2

Intensity of the global FID response and sum of the intensity of the ofactive notes perceived by the panelists after SPME–O analysis

Apricot varieties	$FID$ intensity <sup>a</sup> (peak area $\times 10^4$ )	Aroma intensity <sup>a</sup> (AU)
A 4025	$75.6 \pm 2.2$	$4.0 \pm 0.0$
Goldrich	$77.8 \pm 5.2$	$4.0 \pm 0.0$
Hargrand	$119.6 \pm 14.1$	$5.8 \pm 0.4$
Iranien	$103.6 \pm 3.9$	$5.2 \pm 0.7$
Orangered	$60.6 \pm 3.3$	$4.3 \pm 0.5$
Rouge Roussillon	$106.5 \pm 5.1$	$5.0 \pm 0.0$

Means of six analysis  $\pm$  standard deviation.

<span id="page-4-0"></span>

Fig. 3. Classification of apricot varieties according to the intensity of their aromatic global note obtained by SPME–O total extract.

Secondly, panelists defined by apricot sniffing by SPME–O three global olfactive notes: grassy, fruity and apricot. The sums of the intensities of notes perceived were reported in [Table 2](#page-3-0): a good correlation was found between these results and the FID intensities for the same analysis for the different cultivars. The apricot cultivars were then classified according to the intensity of these notes given by the panelists (Fig. 3). The extracts obtained by SPME from the several cultivars studied are representative of the product, but each of them had a specific characteristic, A 4025 possessed the most intense apricot note, Rouge du Roussillon was perceived as fruity whereas a grassy note was detected for Goldrich. These results were in good agreement with those obtained by sensory analysis on the same cultivars: A 4025 is also found having the most typically apricot aroma, Rouge de Roussillon is considered as the most fruity and Goldrich is also characterized by grassy notes (Robini, 2000 Personnal Communication).

# 3.4. Identification of common apricot volatile compounds by HS–SPME–GC–MS

From the extraction performed by SPME from six apricot cultivars, more than 200 volatile compounds were identified from a rich chromatogram as it can be show on the SPME–GC profile of Rouge du Roussillon cultivar (Fig. 4). Among these compounds 23 were present, in different amount, in all the cultivars studied ([Table 3\)](#page-5-0). All the compounds of the model solution used for the determination of the SPME experimental conditions are present in this list except 2-phenyl ethanol that was not extracted by SPME conditions due to this low concentration in model solution and also in apricot cultivars. According to their perception threshold and their volatility as indicated in [Table 1](#page-1-0) these compounds were considered as good candidates for apricot aromatic quality tracers. Limonene, b-cyclocitral, 6-methyl-5-hepten-2-one and menthone are reported for the first time as being involved in apricot flavour.  $\beta$ -Cyclocitral was known as resulting of thermal, photo oxygenation or enzymatic degradation of  $\beta$ -carotene the main carotenoid pigment found in apricot ([Demole & Berthet,](#page-7-0) [1972; Drawert, Schreier, Bhiwapurkar, & Heindze,](#page-7-0) [1981; Gloria, Grulke, & Gray, 1993; Kanasawud &](#page-7-0) [Crouzet, 1990a](#page-7-0)), whereas 6-methyl-5-hepten-2-one was described as resulting of the degradation of lycopene ([Drawert et al., 1981; Kanasawud & Crouzet, 1990b;](#page-7-0) [Wache´, Bosset-De Ratuld, & Belin, 2002](#page-7-0)). According to the extraction mode used in the present work these compounds resulted probably from enzymatic or photochemical reactions occurring during the maturation process of fruits. Menthone was previously reported as aroma component of fruit-like raspberry ([Broderick &](#page-7-0) [Raspberry, 1976; Winter & Sundt, 1962\)](#page-7-0) or cherimoya ([Idstein, Herres, & Schreier, 1984](#page-7-0)).



Fig. 4. HS–SPME–GC chromatogram of Rouge du Roussillon apricot.

<span id="page-5-0"></span>



 $a$  Means of three analysis  $\pm$  standard deviation.

<sup>b</sup> Retention index on D-WAX column.

Table 3 showed that Hargrand and Rouge du Roussillon presented high amount of aroma compounds selected. These results confirmed the high aroma intensity perceived by SPME–O [\(Table 2\)](#page-3-0). Hargrand was especially the richest in hexyl acetate, Rouge du Roussillon presented high contents of ethyl acetate and p-cymene whereas the highest amount of E-hexen-2-al was found in Goldrich. A 4025 presented high amount of limonene but is the richest in  $\nu$ -decalactone. However, Iranien did not confirmed the result of SPME–O and no particular compounds detected and identified in this variety and not included in the list of common compounds could explain the intensity perceived by panelists and recorded by FID.

The next step of the study was to determine among these common compounds, which were really contributors of apricot aroma.

# 3.5. Characterization of aroma key compounds by HS– SPME–GC–O

Olfactive descriptors were attributed to most of the volatile compounds detected by SPME–GC–O from the apricot cultivars selected. On the 23 common compounds cited in Table 3, only 10 compounds were selected for their olfactive impact (Table 4). Some of them were previously reported as contributors of apricot flavour: linalool [\(Chairote et al., 1981; Takeoka et al.,](#page-7-0) [1990\)](#page-7-0),  $\gamma$ -decalactone [\(Guichard et al., 1990; Takeoka](#page-7-0)

[et al., 1990\)](#page-7-0), hexyl acetate [\(Guichard et al., 1990](#page-7-0)), b-ionone and trans hexen-2-al [\(Takeoka et al., 1990\)](#page-8-0). According to [Toth-Marcus et al. \(1989\)](#page-8-0) only  $\gamma$ -decalactone possessed an apricot, apricot jam-like flavour, whereas linalool and hexyl acetate could be contributors of the flavour of some cultivars. Limonene,  $\beta$ -cyclocitral, 6-methyl-5-hepten-2-one and menthone confirmed their importance in apricot flavour. Limonene and bcyclocitral gave fruity and especially citrus note to apricot aroma of cultivars studied whereas 6-methyl-5 hepten-2-one developed floral note. Moreover, it could be assumed that menthone could contribute to the fresh or refreshing notes of apricot. Menthol was otherwise detected in some mango varieties [\(Koulibaly, Sakho,](#page-7-0) [& Crouzet, 1992\)](#page-7-0).

Table 4

Aromatic notes of volatile compounds identified in the six apricot cultivars having a typical apricot aroma

Volatile compounds	Aromatic note
Ethyl acetate	Fruity
Hexyl acetate	Fruity
β-Cyclocitral	Fruity
$\gamma$ -Decalactone	Peach/apricot jam
Limonene	Citrus
6-Methyl-5-hepten-2one	Floral
Linalool	Floral
$\beta$ -Ionone	Floral
$(E)$ -hexen-2-al	Grassy
Menthone	Minty, fresh



Fig. 5. Spider-web of key aroma compound intensities for HS–SPME extracts of apricot varieties.

Compared with the SPME–O study these results confirmed the importance of fruity and grassy notes in the apricot aroma. However, these results indicated the importance of compounds with floral note not detected by SPME–O probably because of a recovering by the fruity note. In order to characterize the cultivars, the average intensities of the notes of the compounds having olfactive impact perceived by the panelists were reported in Fig. 5. The intensities of these notes on a six-point scale given by three analyses by each panelist were in good correlation with the amount of the compounds given by the [Table 4.](#page-5-0) However, some compounds found in low amount in cultivars were perceived with high aroma intensities especially  $\gamma$ -decalactone. We can show a good correlation of the SPME–O and the SPME–GC–O analyses: Table 5 gives

Table 5

Correlation matrix between key compounds identified by SPME-GC-	
O and global notes obtained by SPME-O	



correlation coefficient between key odorant compounds identified by SPME–GC–O and global olfactive notes obtained by SPME–O. The cultivar A 4025 judged to be characterize by the apricot note by SPME–O ([Fig. 3\)](#page-4-0), was discriminated by the spider-web graphic (Fig. 5) as the most rich in  $\gamma$ -decalactone recognized to having an apricot jam olfactive impact; limonene in high amount in this variety could also participated to this typical apricot note. Moreover, these compounds have the higher correlation coefficient with the apricot olfactive note obtained in SPME–O (Table 5). Goldrich, characterized by the grassy note in the SPME–O, presented the highest amount of  $(E)$ -hexen-2-al, which was identified as grassy contributor in SPME–GC–O and correlated to SPME–O grassy note (Table 5). His highest amount of menthone could intensify the grassy note by his refreshing contribution, moreover this compound was the most correlated to SPME–O grassy olfactive on correlation matrix (Table 5). The fruity characterization of Rouge du Roussillon by SPME–O was also confirmed by the olfactive impact of esters, ethyl acetate and especially hexyl acetate with higher correlation coefficient. Whereas the variety Hargrand was the most influenced by the floral notes of 6 methyl-5-hepten-2-one, linalool and  $\beta$ -ionone and also by fruity notes given by b-cyclocitral and especially hexyl acetate. These results could explain that floral notes were not perceived in SPME–O and the low correlation coefficients of these floral key compounds with the other global olfactive note. Moreover, these balance between <span id="page-7-0"></span>fruity and floral note confirmed the high aromatic quality of this cultivar which had the highest aromatic intensity in SPME–O [\(Table 2\)](#page-3-0) and the highest amount of common volatile compounds [\(Table 3\)](#page-5-0).

## 4. Conclusions

The apricot aroma extracted by HS–SPME was characterized by a mixture of fruity notes, the more important, and of floral notes and grassy notes contributed equally to the aroma of apricot. Differences perceived among the six cultivars studied are related to the quantities of 10 volatiles identified as key aroma compounds by the SPME–GC–O analysis: ethyl acetate, hexyl acetate,  $\beta$ -cyclocitral,  $\gamma$ -decalactone, limonene, 6-methyl-5-hepten-2one, linalool,  $\beta$ -ionone, menthone,  $(E)$ -hexen-2-al. These compounds could be used as tracers to characterize and discriminate apricot varieties on aromatic criteria.

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