

# Identification of the Main Odor-Active Compounds in Musts from French and Romanian Hybrids by Three Olfactometric Methods

Thierry Serot,<sup>\*,†</sup> Carole Prost,<sup>†</sup> Luminita Visan,<sup>‡</sup> and Mirella Burcea<sup>‡</sup>

Ecole Nationale d'Ingénieurs des Techniques et Industries Agro-Alimentaires, Laboratoire de Biochimie Industrielle Alimentaire, Rue de la Géraudière, B.P. 82225, 44322 Nantes Cedex 3, France, and  
Universitatea de Stiinte Agronomice si Medicina Veterinara, strada Bd Marasti 59,  
71331 Bucharest, Romania

Three olfactometric methods (frequency of detection, time–intensity method, and aroma extract dilution analysis) were used to evaluate the main odorants of three musts obtained from French–Romanian hybrids (Valerien, Admira, and Brumariu). The three methods allow detection of the same odor-active compounds. The results obtained from these methods were closely related. Nineteen odor-active compounds were detected, and 13 were identified. The three methods showed the importance of an unidentified compound with a grape and grape juice aroma note in the three musts. Among the other compounds, 3-hexen-1-al, (*E,Z*)-2,6-nonadien-1-ol, and 1-ccten-3-one seemed to contribute actively to the odor of Valerien must. 3-(Methylthio)propanal and hexanal were contributors to the Admira and Brumariu odor. Phenylacetaldehyde was one of the main odor-active compounds in must from Admira.

**Keywords:** Gas chromatography–olfactometry; French and Romanian hybrids; must; odor analysis

## INTRODUCTION

Romania produces well-appreciated wines. Traditionally, wine is made from indigenous cultivars (*Vitis vinifera*) such as Grasa, Feteasca alba, Tamanoiosa romoneasca, and Feteasca regala. However, several French and Romanian hybrids are used because of their potential as good quality grapes and, above all, their high resistance to diseases.

Many studies have reported results on wine volatile compounds (1–5). However, few have been devoted to the aroma of juice or musts obtained from grapes. Many of these aromas are formed during grape processing (destalking, crushing, and pressing) by chemical and enzymatic reactions (6). Some studies have shown the influence on aroma from grapes or musts of variety (7), of viticultural practices (8), and of the area of production (9). Much of this work concerns grapes produced in the United States (10) and Spain (7, 11), but no data exist for free varietal aromas from hybrid grapes grown in Romania.

Gas chromatography–olfactometry (GC-O) methods have been extensively used in aroma research and allow the determination of odor-active compounds in food. Olfactometric techniques can be classified into three categories: dilution methods, time–intensity methods, and detection frequency methods.

Dilution methods, Charm analysis (12), and aroma extract dilution analysis (AEDA) (13) are commonly applied and are suitable to screen the odorant com-

pounds in grape juice, must, or wine (14–16). The OSME method is a time–intensity method developed by McDaniel (17) to characterize the Pinot noir aroma. More recently, van Ruth and Roozen (18) have developed a new technique based on detection frequency of odorant area in a nondiluted extract by numerous panel members. This method, to our knowledge, has not been used in the determination of active odorant compounds in grape wine or must. However, the determination of odor-active compounds involves the preparation of samples prior to GC-O in order to obtain extracts that exhibit an odor similar to the odor of the initial food (19). The most frequently used methods for the isolation of flavor constituents from grape juice or wine involve extraction with solvent (10, 16) or Freon (5, 20), dynamic headspace analysis (7), or headspace solid phase micro-extraction (SPME) (21). The last two are quick methods, but they do not enable the similarity of extracts and corresponding samples to be assessed. In contrast, extraction with Freon seems to be time-consuming. Thus, the solvent extraction technique was used. Several methods and solvents were compared to obtain the most representative extracts.

Three white grape interspecific hybrids grown in Romania were used for this study, Brumariu [Saint Emilion (*Vitis vinifera*) × Rayon d'or (S 405 × S 2007)], Valerien (Seyve-Villard 23410, the exact origin of which is not known), and Admira [Perla de Csaba (*Vitis vinifera*) × Villard noir (SV 12375 × S 8745)]. Brumariu was created by the Blaj Vitivinicol Research Institute (Romania). This hybrid is characterized by yellowish green medium-sized grapes with shades of pink. It is very productive and exhibits a high resistance to diseases. Brumariu hybrid is used to make juice, wine, and alcohol. Valerien was created by La Maison Seyve-Villard (France). It produces small yellow grapes that are used for eating or wine-making. Admira is a creation of the Cluj Agricultural Research Institute (Romania).

\* Address correspondence to this author at the Laboratoire de Genie Alimentaire, IFREMER Centre de Nantes Rue de l'Île d'Yeu, B.P. 21105, 44311 Nantes Cedex 3, France (telephone 2-40 37 42 52; fax 2-40-37-40-71; e-mail tserot@ifremer.fr).

<sup>†</sup> Ecole Nationale d'Ingénieurs des Techniques et Industries Agro-Alimentaires.

<sup>‡</sup> Universitatea de Stiinte Agronomice si Medicina Veterinara.

The grapes of this hybrid are small with a yellowish green color. Admira grapes are used for wine-making or eating.

The aim of our study was to characterize the most odor-active compounds in musts obtained from three hybrids grown in Romania by using three GC-O analyses of representative extracts obtained by an appropriate liquid-liquid extraction method.

## MATERIALS AND METHODS

**Chemicals.** Dichloromethane (GC quality) and all standard compounds were purchased from Sigma-Aldrich Chemical Co. (Îsles d'Abeau, France). Pentane (GC quality) was purchased from Merck (Darmstadt, Germany).

**Juice Preparation.** The grapes from the three hybrids Brumariu, Admira, and Valerien were produced in the Dealu Mare area (Romania) by the Valea Calugareasca vitivinicole station. After harvesting (maturity was estimated by °Brix, titrable acidity, and berry size), grapes were picked off the bunches, crushed with a hand crusher, and then pressed at 40 kPa with a laboratory winepress (revolving cage hydraulic press Gabbia Girevole, Didacta Italia). The musts were homogenized and stored at  $-20^{\circ}\text{C}$  prior to extraction of volatile compounds.

**Extraction of Volatile Compounds.** Six liquid-liquid extraction methods were compared to optimize the production of representative extracts.

*Method A.* Two hundred milliliters of must, placed in a conical flask, was extracted with 5 mL of freshly distilled dichloromethane ( $\text{CH}_2\text{Cl}_2$ ) by stirring for 30 min at  $0^{\circ}\text{C}$  (19) and then centrifuged for 15 min at 10000g. The organic extract was dried with anhydrous sodium sulfate and stored at  $-20^{\circ}\text{C}$  until analysis.

*Method B* was the same as method A except that 20% NaCl was added to the must before extraction.

*Method C.* Two hundred milliliters of must, placed in a conical flask, was successively extracted ( $3 \times 20$  min) at  $0^{\circ}\text{C}$  with  $3 \times 25$  mL of freshly distilled dichloromethane and then centrifuged for 15 min at 10000g. The three organic extracts were pooled, dried with anhydrous sodium sulfate, and concentrated to 5 mL in a Kuderna-Danish concentrator ( $45^{\circ}\text{C}$ ) and then to 1 mL under a stream of nitrogen.

*Method D* was the same as method C except that 20% NaCl was added to the must before extraction.

*Methods E and F* were the same as methods C and D, respectively, except that dichloromethane was replaced by  $3 \times 25$  mL of dichloromethane/pentane (2:1).

### Sensory Analysis/Representativeness of the Extracts.

*Panel.* The panel was composed of 15 assessors (10 female and 5 male, between 25 and 44 years old, nonsmokers) from our department, previously trained in general odor recognition and sensory evaluation techniques.

*Sample Preparation and Presentation.* The must samples were thawed overnight at  $4^{\circ}\text{C}$  in a refrigerator. Five milliliters was placed in 15 mL black coded flasks, and then the temperature was raised to  $25^{\circ}\text{C}$  just before the musts were presented to the panel. An aliquot of each of the must extracts was adsorbed onto a cardboard smelling strip (reference 7140 B.P.S.I., Granger-Veyron, Lys, France), and after 30 s (the time necessary for solvent evaporation), the ends of the strips were cut and placed in dark coded flasks. These flasks were hermetically closed and presented to the panel after 30 min.

*Descriptive Analysis of Must Samples and Extracts.* The panel was asked to describe the odor of musts and extracts by choosing five attributes (19).

*Similarity of Must and Extract Evaluation.* A similarity test was performed to compare the odor of the extracts with the odor of the corresponding musts. The musts and their corresponding extracts were presented simultaneously to the panel, and assessors evaluated the similarity of the odor for each pair. A 100 mm unstructured scale was used, anchored with "very different from the must odor" on the left and "identical to the must odor" on the right. The positions of the extracts on the

scale were read as distance in centimeters from the left anchor. Results were analyzed with ANOVA and LSD tests.

*Odor Intensity Evaluation.* The odor intensities of the extracts were evaluated by using an unstructured scale anchored with "no odor" on the left and "very strong odor" on the right. The positions of the extracts were read as distance in centimeters from the left anchor. Results were analyzed with ANOVA and LSD tests.

### Gas Chromatography—Mass Spectrometry (GC-MS).

The GC-MS system consisted of an HP5890 II gas chromatograph and an HP5971 mass selective detector (Hewlett-Packard Co., Palo Alto, CA). Each extract ( $1 \mu\text{L}$ ) was injected in the splitless mode ( $250^{\circ}\text{C}$  injector temperature, 30 s valve delay) into a capillary column (DB-Wax, 30 m length  $\times$  0.32 mm i.d.  $\times$  0.5  $\mu\text{m}$  film thickness, J&W Scientific, Folsom, CA). The flow rate of carrier gas (helium) was 1 mL/min. Oven temperature was programmed from 50 to  $250^{\circ}\text{C}$  at a rate of  $5^{\circ}\text{C}/\text{min}$  with initial and final hold times of 5 and 10 min, respectively.

Mass selective detector conditions were as follows: ion source temperature,  $180^{\circ}\text{C}$ ; ionization energy, 70 eV; mass range, 30–300 amu; electron multiplier voltage, 2100 V; and scan rate,  $2 \text{ s}^{-1}$ .

Volatile compound identification was based on comparison of GC retention indices (RI) (22), mass spectra (comparison with MS spectra database, NBS 75K and internal library of the laboratory), and odor properties.

### Gas Chromatography—Flame Detection—Olfactometry (GC-FID-O).

The GC-FID-O system consisted of a 3400 Star GC (Varian, Palo Alto, CA), equipped with an FID and a sniffing port supplied with humidified air at  $40^{\circ}\text{C}$ . GC effluent was split 1:1 between the FID and the sniffing port. Each extract ( $2 \mu\text{L}$ ) was injected in the splitless mode ( $250^{\circ}\text{C}$  injector temperature) into a capillary column (DB-Wax, 30 m length  $\times$  0.32 mm i.d.  $\times$  0.5  $\mu\text{m}$  film thickness, J&W Scientific). The flow rate of carrier gas (helium) was 1 mL/min. Oven temperature was programmed from 50 to  $250^{\circ}\text{C}$  at a rate of  $8^{\circ}\text{C}/\text{min}$ .

**Frequency of Detection (FDT).** A panel of 10 judges (23) trained in odor detection and recognition and with experience in GC-O was selected from the 15 previous panelists. Sniffing of the chromatogram was performed for 23 min. The panelists were asked to assign odor properties to each odorant area detected. Detection of an odor at the sniffing port by fewer than 3 of the 10 assessors was considered to be noise (24). The 10 individual aromagrams were summed, yielding the final aromagram (detection frequency versus RI).

**Time—Intensity Method (TIM).** The time—intensity method was used to measure the perceived odor intensity of compounds in GC-O. The same panelists as before were trained to evaluate aroma intensity using a nine-point intensity scale (25) (1 = very weak intensity, 3 = weak intensity, 5 = moderate intensity, 7 = strong intensity, and 9 = very strong intensity). Sniffing conditions were the same as for frequency of detection, except that the panelists were also asked to assess intensity (according to a nine-point scale) for each odorous area. Times and intensities of areas detected by at least three judges were averaged, and a consensus aromagram (averages versus RI) was created.

**Aroma Extract Dilution Analysis (AEDA).** The AEDA method, described by Grosch (13), was used to assess the contribution of individual volatile compounds to the aroma of grape juices. Serial dilutions (1:3 in dichloromethane) of the extract were sniffed until no further odor-active area could be detected. AEDA was performed by two sensitive and repeatable panelists selected during olfactometric global and time—intensity method analyses. The result was expressed as the flavor dilution factor (FD factor =  $3^{n-1}$ , where  $n$  is the number of coincident responses). Data from AEDA were also represented in graphs by plotting FD versus RI.

**Statistical Analysis.** Statistical treatments were performed using Statgraphics Plus software (Manugistics, Inc., Rockville, MD). The Pearson test was used to establish the correlation among the three olfactometric methods.

**Table 1. Similarity Test To Measure the Difference between the Odors of the Musts and the Corresponding Extracts Obtained via the Six Extraction Methods (A–F) and the Odor Intensity of the Extracts<sup>a</sup>**

extract	similarity scale (cm)			intensity scale (cm)		
	Valerien	Admira	Brumariu	Valerien	Admira	Brumariu
A	5.2 <sup>a</sup>	5.1 <sup>a</sup>	5.0 <sup>a</sup>	4.6 <sup>a</sup>	5.2 <sup>a</sup>	5.0 <sup>a</sup>
B	6.4 <sup>c</sup>	6.2 <sup>c</sup>	6.7 <sup>c</sup>	5.9 <sup>b</sup>	5.5 <sup>b</sup>	5.2 <sup>b</sup>
C	6.9 <sup>c</sup>	6.5 <sup>c</sup>	6.1 <sup>c</sup>	7.1 <sup>c</sup>	6.7 <sup>c</sup>	6.5 <sup>c</sup>
D	5.2 <sup>a</sup>	4.9 <sup>a</sup>	4.4 <sup>a</sup>	6.0 <sup>b</sup>	5.8 <sup>b</sup>	5.4 <sup>b</sup>
E	4.8 <sup>b</sup>	4.4 <sup>b</sup>	4.1 <sup>b</sup>	5.0 <sup>a</sup>	4.0 <sup>a</sup>	5.1 <sup>a</sup>
F	4.0 <sup>b</sup>	3.7 <sup>b</sup>	3.9 <sup>b</sup>	4.6 <sup>a</sup>	4.2 <sup>a</sup>	4.8 <sup>a</sup>

<sup>a</sup> The data are the average distances measured in centimeters. In the same column values with the same letter are not significantly different at a level of 5%.

## RESULTS AND DISCUSSION

**Sensory Analysis.** Results from the similarity test (Table 1) show that methods B and C provide the most representative extracts from must. The average positions on the unstructured scale of both extracts B and C are not significantly different at a level of 5%. Furthermore, results from the intensity evaluation test (Table 1) show that the extracts obtained by method C present a stronger odor than those obtained by method B. These results have been verified by direct injection of both extracts in GC. The FID chromatograms obtained by direct injection of extracts C show some supplementary peaks compared to extracts B. Moreover, the areas of the peaks, with the same retention time, are greater in chromatograms of extracts C than in those of extracts B. Therefore, contrary to Moio (19), we did not observe the lack of some characteristic volatile compounds, due to the concentration steps. Consequently, we decided to perform descriptive analysis with extracts obtained by method C.

The descriptive analysis of musts and extracts C confirmed the previous results. Table 2 shows that the attributes used by panelists to describe the musts and associated extracts are very similar.

The panelists described the three musts and the three extracts principally by fruity notes such as grape, prune, red fruit, or wine-like odor. Green, grassy, and earthy notes were used to describe musts and extracts obtained from Admira grapes. An earthy note was also used to describe must obtained from Brumariu, but the corresponding extract was not defined by this note.

Yet we can observe that musts obtained from Admira and Brumariu grapes and corresponding extracts are described by more attributes than must and extract obtained from Valerien.

The results obtained by similarity, intensity evaluation tests, and descriptive analysis demonstrated that method C was convenient for the extraction of volatile

compounds from must and provided extracts which were representative of original musts. Consequently, the determination of potent odorants from musts was conducted by olfactometric analysis of extracts obtained by method C.

**Olfactometric Analysis.** Results of the Pearson correlation are given in Table 3 through the correlation coefficient and the *p* values. The three GC-O methods are compared two by two. Results from both judges of AEDA are also compared. It can be seen that the three olfactometric methods were positively correlated with *p* values  $\cong 0.00001$ , which means that all of the methods were significantly linked. The AEDA responses for judges 1 and 2 were well correlated. Correlations of the AEDA method with the other methods did not show that both judges had distinct sensitivities. According to Le Guen et al. (25), the time–intensity method correlated best with the other techniques. However, we can observe a weak correlation between the frequency of detection technique and AEDA in the case of Admira. This result was due to compounds with a low FD factor, although these compounds were perceived by numerous judges. Differences were due to the methods, which did not measure the same values and used a different number of panelists (25). However, these results will enable the odor-active compounds from the musts obtained from hybrid cultivars to be identified with great precision.

The results of olfactometric analysis are summarized in Table 4. According to the three olfactometric methods, 19 odorous areas were significantly perceived by the panelists in at least one extract.

Among the odorants perceived, some alcohols and aldehydes of six carbon atoms were identified. These compounds are known to exhibit grass-like and green odors. They are mainly formed by enzymatic oxidation of polyunsaturated fatty acids (PUFA) (26, 27). Their concentrations mainly depend on the grape cultivar, although there were variations due to geographical origin or to the ripening stage (11). Some differences in the perception of these compounds was observed for the three musts. Hexanal was perceived with green and grass-like odor mainly in Admira and Brumariu extracts, by eight and six judges, respectively. (*E*)-3-Hexen-1-al, described by grass and grape-like odor, was perceived by eight judges and had an FD = 243 in an extract from Valerien. This compound was perceived by fewer judges, with a weaker FD factor in Admira and Brumariu. (*E*)-3-Hexen-1-ol exhibited a green odor and was perceived only in Valerien and Brumariu extracts. The assessors did not generate common descriptors for this compound, which is usually described by a green leafy odor (28). These compounds must contribute to the green and grass aroma notes of the three musts and corresponding extracts.

**Table 2. Main Odorant Attributes Used by Panelists To Characterize the Three Musts and the Corresponding Volatile Extracts Obtained by Method C**

Valerien		Admira		Brumariu	
must	extract	must	extract	must	extract
grape (8)	grape (8)	grape (9)	grape (8)	grape (9)	grape (6)
wine (5)	prune (5)	fruity (6)	green, leafy (3)	wine (5)	fruity (5)
prune (5)	grass, green (3)	prune (5)	red fruits (3)	prune (5)	wine (3)
fruity (3)	fruity (3)	wine (5)	prune (3)	apple (5)	prune (3)
apple (3)		apple (3)	apple (3)	earthy (3)	green (3)
		grass, green (3)	earthy, woody (3)	alcohol (3)	
		earthy (3)	spicy (3)	red fruit (3)	

<sup>a</sup> Shown in parentheses is the number of judges who used this attribute to characterize the must or extract. Conditions of extraction by method C are indicated under Materials and Methods.

**Table 3. Pearson Correlation between the FDT, TIM, and AEDA Methods for the Three Musts<sup>a</sup>**

	TIM	FDT	AEDA 1	AEDA 2
Valerien				
TIM	1			
FDT	0.95 (0.00001)	1		
AEDA 1	0.86 (0.00001)	0.82 (0.00001)	1	
AEDA 2	0.82 (0.00001)	0.77 (0.00001)	0.85 (0.00001)	1
Admira				
TIM	1			
FDT	0.88 (0.00001)	1		
AEDA 1	0.68 (0.00001)	0.51 (0.0003)	1	
AEDA 2	0.63 (0.00001)	0.50 (0.0003)	0.72 (0.00001)	1
Brumariu				
TIM	1			
FDT	0.92 (0.00001)	1		
AEDA 1	0.71 (0.00001)	0.79 (0.00001)	1	
AEDA 2	0.73 (0.00001)	0.82 (0.00001)	0.82 (0.00001)	1

<sup>a</sup> Correlation coefficient (*p* value).

1-Octen-3-one and (*E,Z*)-2,6-nonadien-1-ol are also the result of PUFA oxidation. 1-Octen-3-one, which exhibited a mushroom-like odor, was mainly perceived in Valerien by six judges and presented an FD = 243. 2,6-Nonadien-1-ol was described with a cucumber-like odor mainly in Valerien by seven judges. These molecules were previously described in muscadine grape juice with a relatively high FD factor (10). Except for hexanal, compounds formed by unsaturated fatty acid oxidation were mainly perceived in Valerien. Saturated alcohols and aldehydes such as hexanal are exclusively formed during the oxidation of *n*-6 polyunsaturated fatty acids, whereas the unsaturated alcohols and aldehydes and (*E,Z*)-2,6-nonadienol are formed during *n*-3 polyunsaturated fatty acid oxidation (29, 30). A difference in PUFA composition or greater lipoxygenase activity in Valerien grape could explain the difference in perception of these compounds among the three musts.

According to the three olfactometric methods, 3-(methylthio)propanal was the most potent odorant in the

extracts of Admira and Brumariu, but it was weakly perceived in Valerien extract. This compound, which exhibited a boiled potato-like odor, has been reported in muscadine grape juice (10), in some fruit juices such as orange (31) and grapefruit (32), and in wine (33). Methional has been previously reported as the cause of an off-flavor in beer (34) and wine (35). The formation of this molecule generally occurs during Strecker degradation of methionine induced by cooking (36) or by *o*-quinone in wine (37). According to Escudero (35), methylthiopropional in wine would be formed from the peroxidation of methylthiopropional. Some differences in methylthiopropional precursors or differences in oxidation rate could explain the difference in perception of this compound among the three musts. However, no off-flavor of boiled potatoes was perceived in the three musts and extracts.

Three compounds exhibited the sweaty aroma notes of phenylacetaldehyde, ethyl 3-hydroxybutanoate, and an unidentified compound (peak 10, RI = 1404). Phenylacetaldehyde was perceived by eight judges in Admira and by only three judges in Brumariu. Phenylacetaldehyde was not detected in Valerien. This compound, which exhibits a sweaty and honey-like odor, has previously been identified in muscadine juice (10). It may be formed by the decarboxylation of acetophenylacetate. Ethyl 3-hydroxybutanoate did not seem to strongly contribute to the aroma note of the three musts; it was detected by only a few judges and had a relatively low FD factor. This result confirms that obtained by Baek et al. (10) with muscadine grape juice.

Two compounds with red fruit-like and grape aroma notes were detected: *p*-1-menthene-8-thiol and an unidentified compound (peak 18, RI = 1858). *p*-1-Menthene-8-thiol, the identification of which was based on RI and mass spectra, was principally perceived in Brumariu and characterized by fruity and red fruit-like odor. This compound was previously noted in grapefruit juice (32) but never reported in grape, grape juice, must,

**Table 4. Odor-Active Compounds Detected in the Three Musts by Three Olfactometric Methods**

peak	RI <sup>a</sup>	compound	odor description <sup>b</sup>	TIM		FDT			AEDA factor of dilution							
				av intensity			no. of judges			V <sup>g</sup>		A <sup>g</sup>		B <sup>g</sup>		
				V	A	B	V	A	B	1 <sup>h</sup>	2 <sup>h</sup>	1	2	1	2	
1	983	2,3-butanedione <sup>c-f</sup>	caramel, buttery	0	0	1.9	0	0	3	0	0	0	0	0	1	1
2	1044	2-methyl-3-buten-2-one <sup>d,e</sup>	buttery, cannell	1.6	1.1	0	3	1 <sup>i</sup>	0 <sup>i</sup>	0	0	0	1	3	0	0
3	1088	hexanal <sup>c-f</sup>	grass, green	1.1	4.7	3.6	2 <sup>i</sup>	8	6	0	0	27	81	9	9	
4	1149	( <i>E</i> )-3-hexen-1-ol <sup>c-f</sup>	grass, grape	4.5	1.9	3	8	4	5	243	243	9	27	81	81	
5	1309	1-octen-3-one <sup>c-f</sup>	mushroom, woody	3.5	2.9	2.5	6	3	3	243	81	81	27	27	9	
6	1345	unknown	flowery	1.7	1.7	1.8	3	2 <sup>i</sup>	2 <sup>i</sup>	0	3	0	0	0	0	
7	1353	6-methyl-1-heptenone <sup>c-e</sup>	grass, green	2.0	1.7	2.3	5	2 <sup>i</sup>	4	1	9	3	1	3	3	
8	1377	( <i>E</i> )-3-hexen-1-ol <sup>c-f</sup>	green	3	1.7	2.6	5	2 <sup>i</sup>	5	27	27	81	9	9	3	
9	1387	( <i>Z</i> )-3-hexen-1-ol <sup>c-e</sup>	no common descriptor	1.7	1.2	1.1	4	2 <sup>i</sup>	1	0	3	1	3	3	1	
10	1404	unknown	fruity, sweaty,	1.5	1.6	1.7	3	3	3	3	1	0	0	1	3	
11	1460	3-(methylthio)propanal <sup>c-f</sup>	boiled potatoes	0.9	8.1	4.7	1 <sup>i</sup>	9	8	1	0	243	81	81	27	
12	1522	ethyl 3-hydroxybutanoate <sup>c-e</sup>	hay-like, sweaty	1.2	2.3	2.4	2 <sup>i</sup>	3	4	1	3	9	1	3	81	
13	1530	unknown	grilled, burnt	1.1	1.3	2.1	1 <sup>i</sup>	1 <sup>i</sup>	4	0	0	3	0	1	3	
14	1598	<i>p</i> -1-menthene-8-thiol <sup>c,e</sup>	fruity, red fruit-like	1.2	2	5.7	2 <sup>i</sup>	3	6	3	9	9	27	9	81	
15	1657	phenylacetaldehyde <sup>c-e</sup>	sweaty, caramel, syrup	0	4.8	1.6	0 <sup>i</sup>	8	3	0	0	3	27	1	1	
16	1740	unknown	green, mint	3.3	1.9	1.8	4	3	4	27	9	3	1	3	27	
17	1784	( <i>E,Z</i> )-2,6-nonadien-1-ol <sup>c-f</sup>	melon, cucumber	3.1	2.1	2.8	7	3	3	81	81	3	9	3	1	
18	1858	unknown	grape, grape juice, prune	4.0	4.0	4.1	7	5	7	81	27	27	9	81	27	
19	2042	unknown	citrus fruit	1.1	2.3	0	1 <sup>i</sup>	3	0 <sup>i</sup>	0	1	0	0	0	0	

<sup>a</sup> Retention index on DB-Wax column. <sup>b</sup> Odor description as perceived by panelists during olfactometry analysis. <sup>c</sup> Identification based on coincidence of RI with those reported in the literature. <sup>d</sup> Identification based on mass spectrometric data (NBS 75K database). <sup>e</sup> Identification based on coincidence of odor description by assessors with odor description reported in the literature. <sup>f</sup> Identification based on coincidence of retention index and mass spectra with those of authentic standards. <sup>g</sup> V, Valerien; A, Admira; B, Brumariu. <sup>h</sup> Judges 1 and 2. <sup>i</sup> Compounds not significantly pointed by panel.

or wine. The unidentified compound (peak 18) is the only active odorant compound perceived by at least five judges in the three musts. Furthermore, this compound exhibited grape, grape juice, and prune aroma notes, which correspond to the attributes used with the highest frequency to characterize the three musts. We have to consider this compound's contribution to the odor of the three musts. Its RI was similar to that of  $\beta$ -damascenone (3, 5). However, an attempt to identify the chemical structure of this compound was unsuccessful. Its mass spectrum is as follows:  $m/z$  (% intensity) 43 (70), 57 (68), 73 (23), 87 (100), 101 (15), 129 (8). According to the NBS 75K database, this mass spectrum is very similar to the spectrum of ethanol-2-(2-butoxyethoxy) acetate. This hypothesis could not be verified with pure standard, particularly by comparing retention index and odorant properties.

In conclusion, it can be said that the odor of the three musts is due to three groups of odorant compounds. The first group is mainly constituted by aldehydes and alcohols that exhibit a green and grass-like odor. The second group represents compounds with sweaty odors, particularly phenylacetaldehyde. In the third group, volatile compounds with fruity and grape-like odors are found. Furthermore, GC-O analysis showed that, even if the main odor-active compounds are similar, their levels of perception are different in the three musts. Consequently, although it is difficult to quantify the contribution of a compound or a group of compounds to the odor of musts, it can be said that the extraction and GC-O methods carried out in this study are useful to discriminate the three musts on the basis of their main odor-active compounds.

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