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Characterization and evaluation of volatile compounds of three grape varieties *(Vitis labrusca*) from the region of Bento Gonçalves – RS

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In grape must, the aroma is an indication of adequate conservation status. It is able to indicate losses due to thermal degradation, besides representing a great contribution in the properties of the sensorial quality of the grape juices most appreciated by consumers. This study identified the volatile compounds in different must cultivars of *Vitis labrusca* grapes. The volatile composition was evaluated by the solid phase microextraction method (SPME), using the polydimethylsiloxane/divinylbenzene fiber (PDMS/DVB). The separation and identification of the volatile compounds were carried out through gas chromatography by mass spectrometry (GC-MS). The results showed the identification of forty-four compounds belonging to the following chemical classes: carboxylic acids, alcohol, aldehydes, ketones, furans esters and terpenes. The class of esters was the most numerous compounds identified and is largely responsible for the fresh and fruity aroma of the tested musts. Solid-phase microextraction proved to be a reproducible, sensitive and fast-response technique for the identification of chemical biomarkers in musts of different grape cultivars.

Key words: Aroma, gas chromatography by mass spectrometry (GC-MS), headspace, solid phase microextraction method (SPME).

INTRODUCTION

The production and consumption of whole grape juice in Brazil and in the world increase every year. The grape is the fruit of the vine *(Vitis* sp*.),* a plant of the family *Vitaceae*, and is used in the production of juices, sweets, wines, and raisins *in natura*.

Generally, in the grape juice production are used the species *Vitis vinifera* grapes from both white and purple varieties. In Brazil, the whole grape juice comes from the American group grapes (*Vitis aestivalis, Vitis labrusca, Vitis bourquina*). In Serra Gaúcha, the most widespread cultivars of *V. labrusca* species are Bordeaux, Concord, and Isabel, which are mainly used for the production of grape juice.

One of the decisive attributes in the selection and

acceptance of foods and beverages is the aroma, being perceived by the retronasal and gustatory sense, so that the demand for new flavors has attracted the aromatization industry's attention, for the characterization of volatile compounds that are responsible for these characteristics, which explains the importance they play in the quality of the fruits and their derivatives (Jiang and Song, 2010).

The fruity aroma is formed by volatile compounds of low molecular weight and low polarity, belonging to several chemical classes such as carboxylic acids, alcohols, aldehydes, esters, ethers, lactones and terpenes (mainly mono and sesquiterpenes), as well as amino or sulfur compounds (Bicas et al., 2011).

The volatile aroma compounds are thermolabile substances that can undergo cyclization, rearrangement, and oxidation when exposed to high temperatures. For the separation and identification of these volatile compounds, one of the most commonly used techniques is gas chromatography coupled to mass spectrometry (GC-MS). For the extraction of the volatile compounds (VCs), we have several methods: solid phase extraction (SPE), solid phase microextraction (SPME), liquid-liquid extraction (LLE), packaged sorbent microextraction (PSME) and stir bar sorbent extraction (SBSE) (Huang and Lee, 2012; Xu et al., 2016; Uekane et al., 2017).

Among these methods, the solid phase microextraction (SPME) is widely used for both volatile and semi-volatile fruits and their derivatives, having benefits such as sensitivity, reproducibility, besides being solvent-free. Sample preparation is simple and demands low temperatures (Kataoka et al., 2000; Gutiérrez-Rosales, 2010; Merkle et al., 2015; Mesquita et al., 2017; Rocha et al., 2017).

Volatile compounds are important indicators of the quality and provenance of the food and the varieties used. Thus, the objective of this study was to identify the volatile compounds in musts of *V. labrusca* cultivars, searching for the main markers of each cultivar.

METHODOLOGY

Samples

The samples of *V. labrusca* cultivars grape musts (Bordeaux, Concord, and Isabel) were supplied by Embrapa Grape and Wine from Rio Grande do Sul during the month of August 2016.

Solid phase microextraction (SPME)

For extraction of the volatile compounds, the solid phase microextraction method was used in *headspace* mode (HS-SPME). It has been used for these operations that create the link between the chemical matrix and the analytical instruments, being particularly interesting for Gas Chromatography (GC). The method has high concentration power (adapting to GC detectors sensibility), it is suitable for many types of analytes and facilitates the transport of the extracted material to the chromatograph (Pawliszyn, 1997), based on the retention of the analytes of interest on a stationary phase, which is attached or deposited in a fused silica capillary microfiber. A Polyimethylsiloxane/divinylbezene (PDMS/DVB) 65 µm semi-polar fiber (SUPELCO) was used and conditioned according to the instructions provided by the manufacturer.

Preparation of the samples

For extraction, 2.0 g of samples of each variety were weighed in SPME-specific 20-mL vials, sealed with aluminum and rubber septum (Garcia et al., 2016).

Thereafter, the sample vial was placed in an aluminum block, preheated at 60°C, on a non-shaking heating plate and left for 5 min to establish its balance. After 5 min, the SPME fiber (PDMS/DVB) adapted to a holder was exposed in the vial in *headspace* mode for 15 min, then the fiber was collected and taken for manual insertion into the gaseous chromatograph injector, being exposed for 5 min, for the desorption of volatile organic compounds (VOCs) (Garcia et al., 2016).

The GC-MS analysis parameters

The VOCs identification was done at the Laboratory of Mass Spectrometry of the Department of Chemistry - UFMG, using a gas chromatograph (Trace GC Ultra) coupled to a mass spectrometer (Polaris Q) by Thermo Scientific (San Jose, CA), with an "ion-trap" type analyzer, with a split/splitless injector, in "splitless" mode. Helium gas was used as a carrier gas at a constant flow of 1 mL min^{-1} by electron impact ionization (EI), with energy rate of 70 eV. The chromatographic column used was a TR-1 MS capillary column (100% dimethylpolysiloxane, 60 m long × 0.25 mm of internal diameter × 0.25 µm of a thick film) by Supelco (*Sigma Aldrich*). The chromatographic analysis conditions were: injector temperature at 250°C, 5 min of desorption time, source temperature at 200°C, and interface temperature at 275°C. The column heating was performed with programmed temperature, which started at 40°C, remaining for 2 min in this temperature, and then, it was increasing at a heating rate of 10°C/min reaching 100°C, maintaining the isotherm for 2 min. Next, it was heated at a rate of 15°C/min reaching 180°C, when the isotherm was maintained for 2 min. Then, the heating process was kept at 15°C/min until it reached 245°C,when the isotherm was maintained for 3 min. Data acquisition occurred in the *Full Scan* mode with a range of 50 to 400 m/z (Garcia et al., 2016).

Identification and correlation of detected volatiles

The VOCs spectra were identified according to their fragmentation profile, which was compared to the mass spectra of the *National Institute of Standards and Technology* (NIST) using a similarity level (RSI) higher than 500. In addition, the data were confirmed by

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comparison with the compounds already reported in the literature

RESULTS

V. labrusca musts chromatograms (Bordeaux, Concord, and Isabel) are presented in the Appendix Figures 1 to 3, respectively, showing the profile of the cultivars. Volatile organic compounds (VOCs) were identified according to the m/z ratio shown in Table 1. The main chemical classes found were carboxylic acids, alcohols, aldehydes, ketones, esters, furans, terpenes, and sesquiterpenoids, amounting to 41 compounds.

For the three investigated cultivars, octanoic acid was identified in Bordeaux type, in which was also identified the decanoic acid. For Concord type, nonanoic acid was identified.

The common alcohols for the three cultivars evaluated were: 4-penten-2-ol and benzene ethanol. (*Z*) -2-hexen-1-ol was found in Concord and Isabel.

For Bordeaux cultivar musts, cis-2-octane-1-al, ionone and 3,4,5,6,7,8-hexahydro-4,8-dimethyl-1H-naphthalene were identified.

The esters were the most numerous compounds identified, being especially responsible for the fresh and fruity aroma of the juice. The common esters found in all three varieties were ethyl caproate, acetic acid phenyl ester, anthranilic acid methyl ester, capric acid ethyl ester and ethyl-2,4-decadienoate ethyl ester acid (E, Z). The ethyl 2-furancarboxylic acid was detected in the musts of Bordeaux and Isabel cultivars.

DISCUSSION

Among the classes of the compounds identified, the carboxylic acids have an influence on the flavor. They are present in whole grape juice and the aliphatic acids such as octanoic and decanoic acids are produced from fatty acids, influencing negatively the aroma of the juices (Clarke and Bakker, 2004). Carboxylic acids were identified in Merlot and Moscatel wines *(V. viniferas*), in addition to octanoic, nonanoic and decanoic acids using HS-SPME-GCxGC/TOFMS and HS-SPME-1D-G/MS, respectively (Nicolli et al., 2015). In Chardonnay grapes, octanoic acid was detected using GC-MS (Liberatore et al., 2010).

Alcohols can be produced from sugars during processing, causing positive and negative effects to the aroma of the must (Fariña et al., 2015). The 4-penten-2-ol and (E) -2-hexen-1-ol alcohols were identified by HS-SPME using PDMS/DVB, CAR/DVB/PDMS and CW/DVB fibers in Cabernet Sauvignon and Muscat grapes (*V. viníferas*) (Canuti et al., 2009; Sánchez-Palomo et al., 2005). For the Chardonnay and Merlot grapes (*V. viniferas*), the 4-penten-2-ol compound was detected using CAR/DVB /PDMS fiber (Canuti et al., 2009; Welke et al., 2012), and (Z) -2-hexen-1-ol compound was detected in Cabernet Sauvignon grapes (*V. viníferas*) using the PDMS and DVB/CAR/PDMS fibers (Canuti et al., 2009).

Aldehydes and ketones such as cis-2-octaen-1-al, ionone, and 3,4,5,6,7,8-hexahydro-4,8-dimethyl-1H-naphthalene, as well as lactones, contribute to the floral and fruity scent of the must (Webb and Muller, 1972).

The esters contribute to the fresh and fruity aroma (Welke et al., 2012), as the ethyl-2,4-decadienoate ethyl ester acid (*E, Z*), which was detected in Merlot grapes*(V. viníferas)* and in commercial fruits juice (pear, apricot and peach) by using HS-SPME (Welke et al., 2012; Riu-Aumatell et al., 2004). In Chardonnay grapes *(V. viniferas)* 2-furancarboxylic acid ethyl ester was identified (Liberatore et al., 2010).

The terpenes, mainly monoterpenes, contribute to the varietal aroma of the juice because they have low thresholds of perception, favoring floral scents (Welke et al., 2012). The element *cis*-geraniol, which was identified in Moscato Bianco and Moscato Giallo grapes (Berger, 2007; Xin et al., 2013; Closs et al., 2014) was also detected in the Bordeaux cultivar under study. The 3-carene was identified in Merlot grapes (*V. viníferas*) (Welke et al., 2012). The same was detected in Bordeaux and Isabel grape musts. The p-menth-1-en-4-ol was detected in Muscat grapes (*V. viniferas*) (Kang et al., 2010) and was also identified in the three musts of *V. labrusca*. The cis-z-a-bisabolene epoxide was found in Merlot grapes (*V. labrusca*) and the *α*-selinene was identified in Chardonnay wines (*V. labrusca*) (Welke et al., 2012). In commercial fruit juices (pear, apricot and peach), naphthalene-1,2,4,5,8,8-a-hexahydro-4,7-dimethyl-1- (1-methylethyl) were identified (Riu-Aumatell et al., 2004). The sesquiterpenoids identified in Bordeaux grape musts were the cis-*Z*-a-bisabolene epoxide, *trans*-longipinocarveol, naphthalene-1,2,4,5,8,8a-hexahydro-4,7-dimethyl-1- (1- methylethyl), α-selinene, epi-γ-eudesmol and valerianol.

In commercial fruit juices (pear, apricot, and peach), the volatile compound a-damascenone was detected (Riu-Aumatell et al., 2004), being also detected in the three musts of *V. labrusca* evaluated.

Conclusion

The extraction method and the PDMS/DVB fiber used were efficient in identifying forty-one volatile compounds of several chemicals, responsible for the flavor of the grape must. The classes that determined the volatile profile of *V. labrusca* musts studied are carboxylic acids, alcohols, aldehydes, ketones, esters, furans, and terpenoids. Among these forty-one compounds identified, twelve are common volatile compounds to musts of *V. labrusca* cultivars studied, such as: octanoic acid, 4-pent-2-ol, benzene ethanol, ethyl caproate, acetic acid linalool ester, anthranolic acid methyl ester, acetic acid phenyl ethyl ester, capric acid ethyl ester, ethyl 2,4-decadienoate

Table 1. Volatile compounds identified in grape musts of *Vitis labrusca.*

#	Compounds	m/z	Cultivars		
			Bordeaux	Concord	Isabel
CarboxylicAcids					
1	Octanoicacid[a,b,c,d,]	43, 55, 60, 73, 135	X	X	X
2	Nonanoicacid[b,c]	41, 55, 73,24; 115,09; 129,05	ND	X	ND
3	Decanoicacid[a,c,d]	41,27; 55,33; 73,29; 87,22; 129,15; 172	X	ND	ND
Alcohol					
4	4-penten-2-ol	42; 43;86	X	X	X
5	(Z)-2-hexen-1-ol [d,e]	39,24; 43,20; 57,17; 67,13; 82,05; 100	ND	X	X
6	Benzene ethanol (phenylethyl alcohol)	91,25;92,14, 121,27; 65,46; 122	X	X	X
Aldehyde					
7	Cis-2-octane-1-al	41,18; 55,19; 70,08,83,06; 126	X	X	ND
Ketones					
8	Ionones	41; 93; 121; 135; 177; 192	X	X	ND
9	3,4,5,6,7,8-hexahydro-4,8-dimethyl-1H-naphthalen-2-one	97,39; 123,29; 137,23; 179,14; 212,99; 204	X	ND	ND
Furans					
10	5- (Hydroxymethyl) furan-2-carbaldehyde [d]	39,21; 69,39; 97,10; 126	ND	X	X
Esters					
11	Ethylcaproate (ethylhexenoate)	43,25; 55,36; 61,25; 73,25; 88,12; 99,01; 145,09	X	X	X
12	Acid-2-furancarboxylic methylester (2-furoate methyl)[f]	39,24; 95,12; 125,92	X	ND	X
13	*Trans*-2-hexenoate ethyl	39,24; 55,29; 73,32; 99,04;142	ND	ND	X
14	Aceticacidlinalool ester	43; 71; 91; 137; 159; 196	X	ND	ND
15	Methylsalicylic ester	92,3; 120,18; 151,99 e 121,17; 92,31; 120,14; 152,01	X	X	X
16	Acetic acid-phenyl-ethyl ester	65,30; 91,16; 164	X	X	X
17	Benzoicacidphenyl ester	79,44; 103,28; 104,15; 105,11; 226	X	ND	ND
18	Anthranilicacid, methyl ester	92,52; 119,22; 151,07	X	X	X
19	Naphthalene-1,2-dihydro-2,5,8-trimethyl	43,22; 142,35; 157,17; 172,10	ND	X	ND
20	Decenoicacidethyl ester	39,82; 41,27; 55,26; 67,31; 81,33; 152,11	ND	X	ND
21	Capricacid, ethyl ester	55,45; 61,31; 70,24; 73,27; 157,14; 200	X	X	X
22	Ethyl-2-4-decadienoate ethyl ester (E, Z)[d,g]	67,51; 81,34; 97,30; 125,11; 196	X	X	X
23	Anthranilicacid – ethyl ester	92,34; 119,24; 120,22; 137,21; 165,08	X	ND	ND
24	Linolenicacid-ethyl ester	67,35; 79,33; 121,20; 136,18; 178,06; 306	X	ND	ND
Terpenes					
25	(S)-cis-verbenol((1S, 2S, 5S) -4,6,6-trimethylbicyclo [3.1.1] hept-3-en-2-ol)	39;59;95;111;152	X	ND	ND
26	2-ethenyl-6-methyl-5-hepten-1-ol	39; 41; 79; 121; 136; 154	X	ND	ND
27	P-menth-1-en-4-ol[h]	43,38; 71,32; 91,29; 93,20; 111,11; 154	X	X	X
28	Allo-ocimene((4E, 6E) -2,6-dimethylocta-2,4,6-triene)	93,51; 121,15; 136	X	ND	ND
29	Dihydroumbellulone (bicyclo [3,1,0] hexan-2-one, 4-methyl-1- (1-methylethyl) - (1a, 4a, 5a))	39,22; 67,29; 81,25; 97,09; 152	X	ND	ND
30	6-isopropyl-3-methyl-7-oxabicyclo-heptan-2-one	43,23; 55,44; 83,32; 125,14; 168; 182,03	ND	ND	X
31	3-carene (4,7,7-trimethylbicyclo [4.1.0] hept-3-ene)[d]	59,50; 81,50; 93,30; 121,19; 136,10	X	ND	X
32	cis-geraniol (cis-3,7-dimethyl-2,6-octadien-1-ol)[i,j]	41,18; 67,28; 93,16; 121,11; 154	X	ND	ND
Sesquiterpenoids					
33	(Z) -bisaboleneepoxide[d]	41,29; 57,51; 67,33; 97,45; 102,21; 220	X	ND	ND

Table 1. Contd.

34	1,2,4a, 5,8,8a-hexahydro-4,7-dimethyl-1- (1-methylethyl) -naphthaleno[g]	105,58; 133,34; 161,29; 189,19; 204,12	X	ND	ND
35	α-selinene[d]	41;67; 81; 107; 134; 162; 204	X	ND	ND
36	-2epi-γ-Eudesmol	105,50; 133,29; 149,31; 161,20; 189,16; 204,17; 222	X	ND	ND
Others					
37	4-hydroxy-2-butanone-ethyl acetate	42;45;61;70;88	X	X	ND
38	Benzylcyanide	89,34; 90,32; 116,22; 117,09	X	X	X
39	1H-2-indonone, 2,4,5,6,7,7a-hexahydro-3- (1-methylethyl) -7a-methyl	93,32; 121,29; 177,12; 192,07	X	X	X
40	1,2-Dihydro-1,5,8-trimethylnaphthalene	43,22; 142,35; 157,17; 172,10	ND	X	ND
41	A-damascenone (E) -1- (2,6,6-trimethylcyclohexa-1,3-dien-1-il) -but-2-en-1-one [g]	41,82; 69,47; 105,31; 121,21; 190,02; 199	X	X	X

Solid phase microextraction fiber: Polydimethylsiloxane/divinylbenzene (PDM/DVB) with 65 µm. The letters indicate compounds that have already been identified by other authors: [a](Etievant, 1991; Gürbüz et al., 2006); [b](Guio et al., 2010); [c] (Nicolli et al., 2015); [d](Welke et al., 2012); [e](Canuti et al., 2009); [f](Liberatore et al., 2010); [g](Riu-Aumatell et al., 2004); [h](Kang et al., 2010); [i](Xin et al., 2013); [j](Closs et al., 2014). X: Detected, ND: Not detected.

acid ethyl ester (E, Z), p-meth-1-em-4-ol, benzyl cyanide and 1-H-2-indonone-2,4,5,7,7a-hexahydro-3- (1-methylethyl) -7a-methyl - some of them being reported in the literature.

The parameters used in the study were sensitive, fast, easy, and useful for the quantification and identification of volatile compounds according to data reported in the literature and, in the future, it will be possible to determine the aromatic maturity for different grapes.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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Appendix

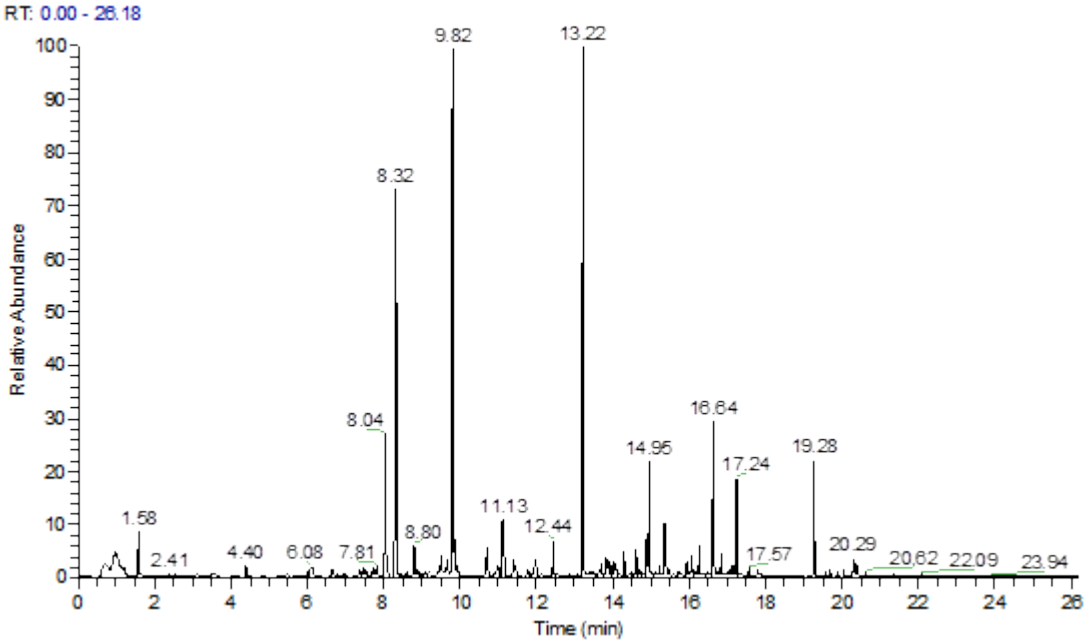

Figure 1. Chromatogram profile of volatile compounds of grape musts belonging to Bordeaux cultivars (*Vitis labrusca*).

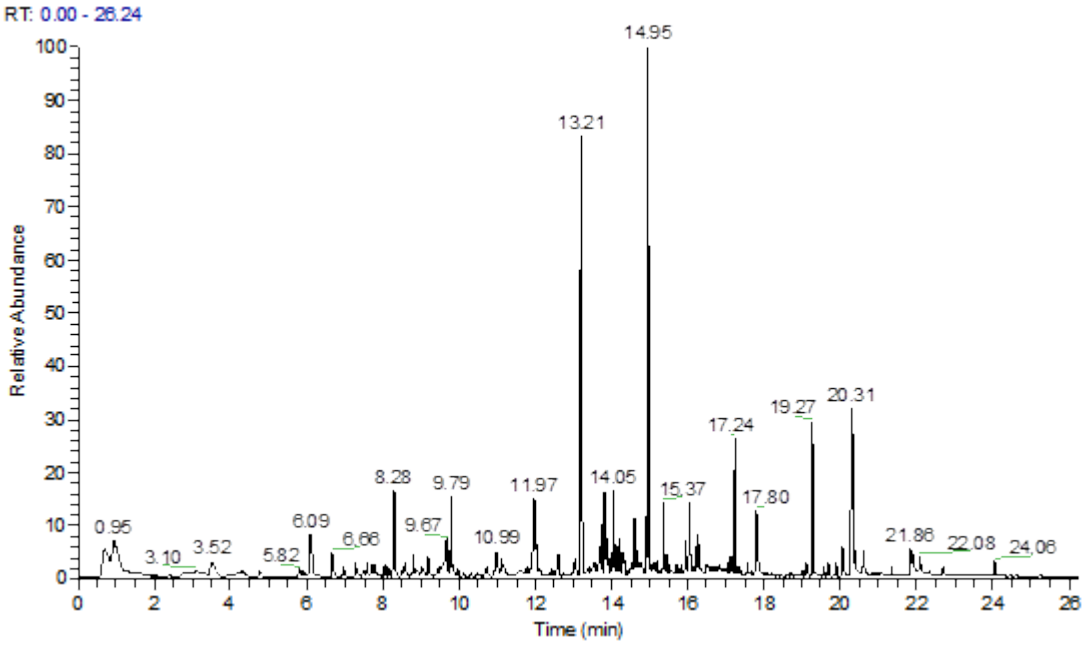

Figure 2. Chromatogram profile of volatile compounds of grape musts belonging to Concord cultivars (*Vitis labrusca*).

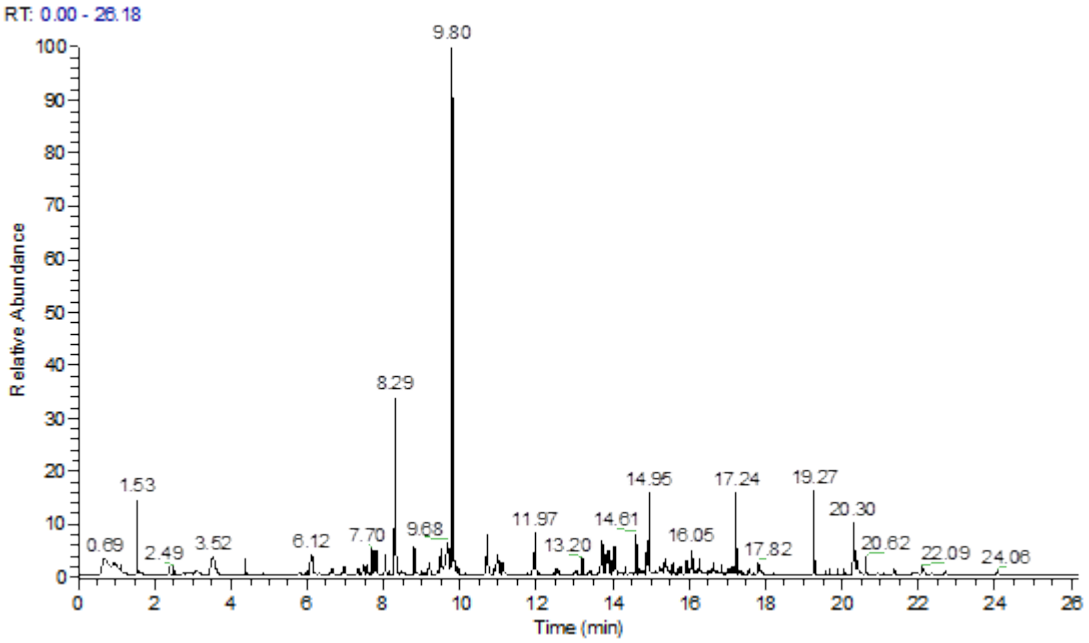

Figure 3. Chromatogram profile of volatile compounds of grape musts belonging to Isabel cultivars (*Vitis labrusca*).