Supplementary Information

Maturation and Maceration Effects on Tropical Red Wines Assessed by Chromatography and Analysis of Variance - Principal Component Analysis

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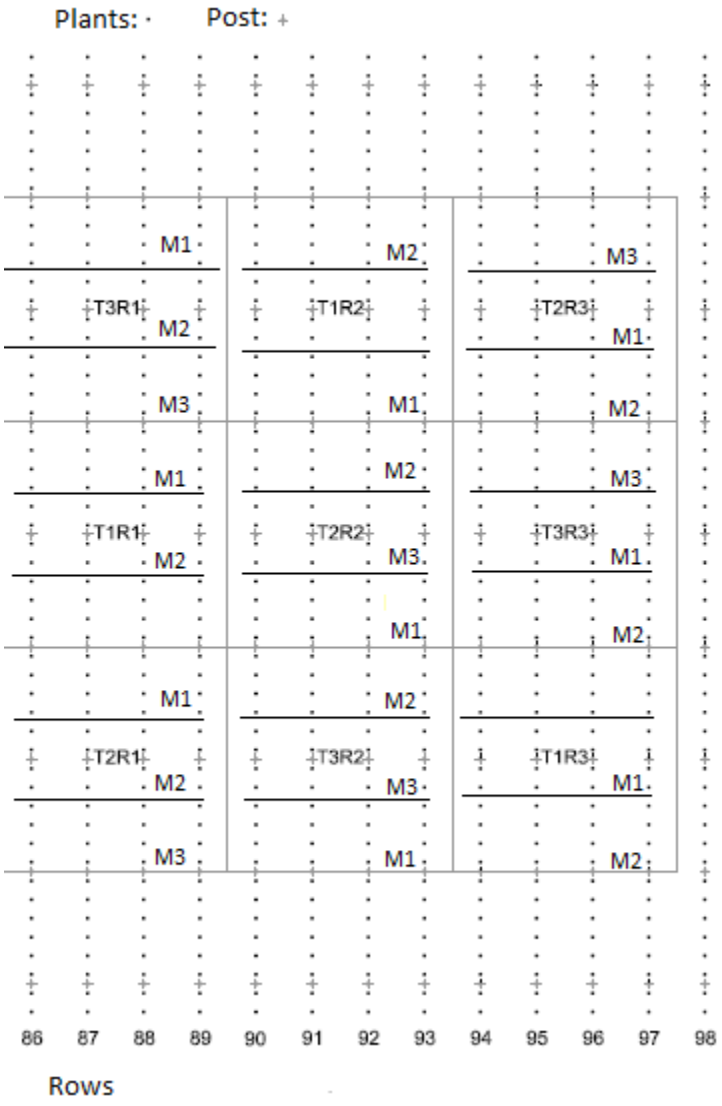

Figure S1. Scheme of design of the field experiments that originated the wines under study. T_1: grapes harvested at 113 days after pruning (DAP) for grapes, with total soluble solid content of 19.0 °Brix; T_2: 120 DAP and 21.0 °Brix; T_3: 127 DAP and 23.0 °Brix; M_1, M_2, M_3 designate three periods of maceration during fermentation: 10, 20 and 30 days, respectively. Plants: grapevines; Rows: rows of grapevines; R: field experiment replicates that were vinified separately; Post: a post for every six grapevines using a conduction system on trellis.

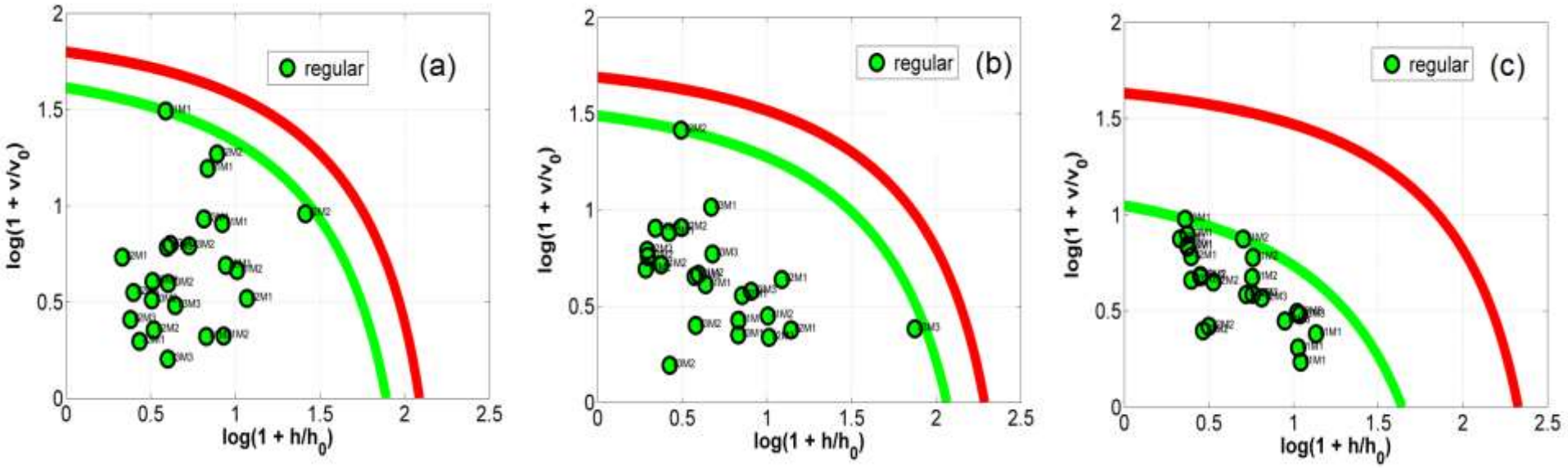

Figure S2. Acceptance area for outlier detection based on DD-SIMCA of the three main components relating wine samples derived from different maturation and maceration times and (a) physicochemical parameters, (b) phenolic compounds, (c) volatile compounds. The green and red solid lines are the border (at 95% confidence) of regular and extreme samples, respectively. Position of a particular sample is defined by v (orthogonal distance) and h (scores distance). The terms h_0 and v_0 are scaling factors.

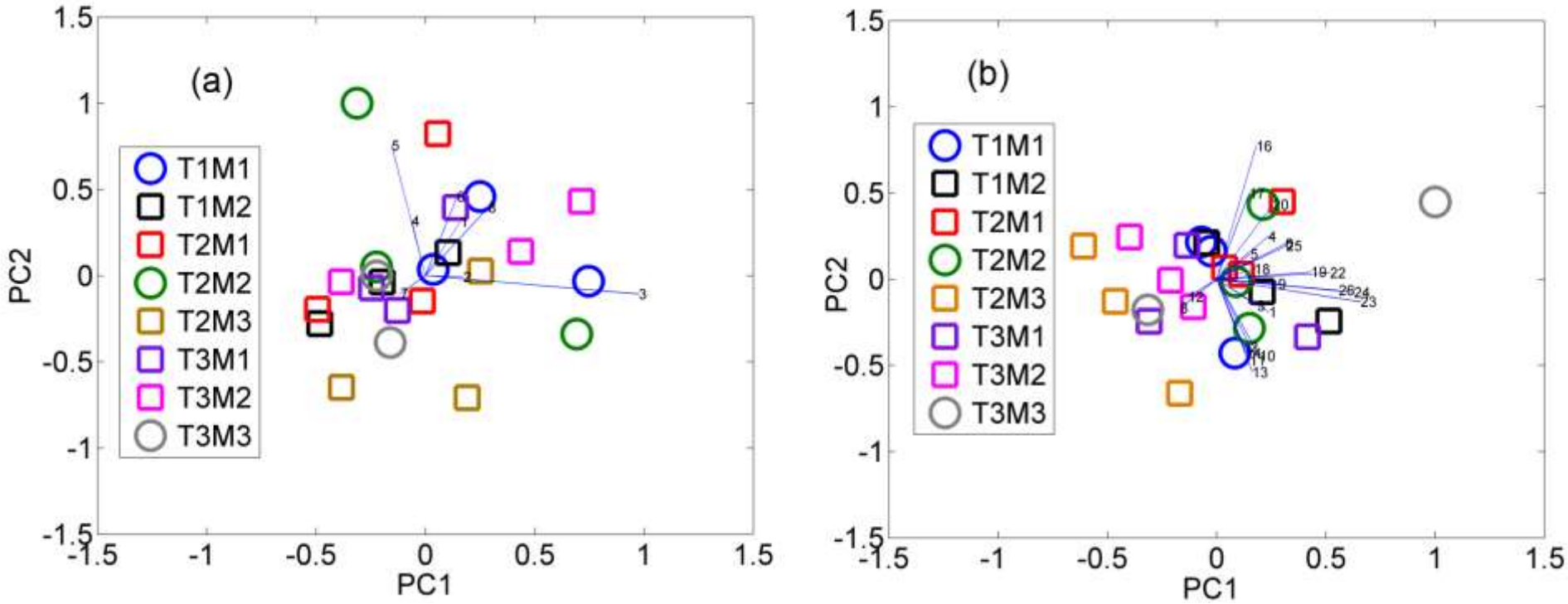

Figure S3. Bi-plots of (a) physico-chemical parameters (PC1 = 27.7% and for PC2 = 21.8%) and (b) phenolic compounds (PC1 = 23.2% and for PC2 = 18.2%) of Syrah wines showing the first two principal components of the interaction between the factors maturation (T) *versus* maceration (M), by means ANOVA-PCA.

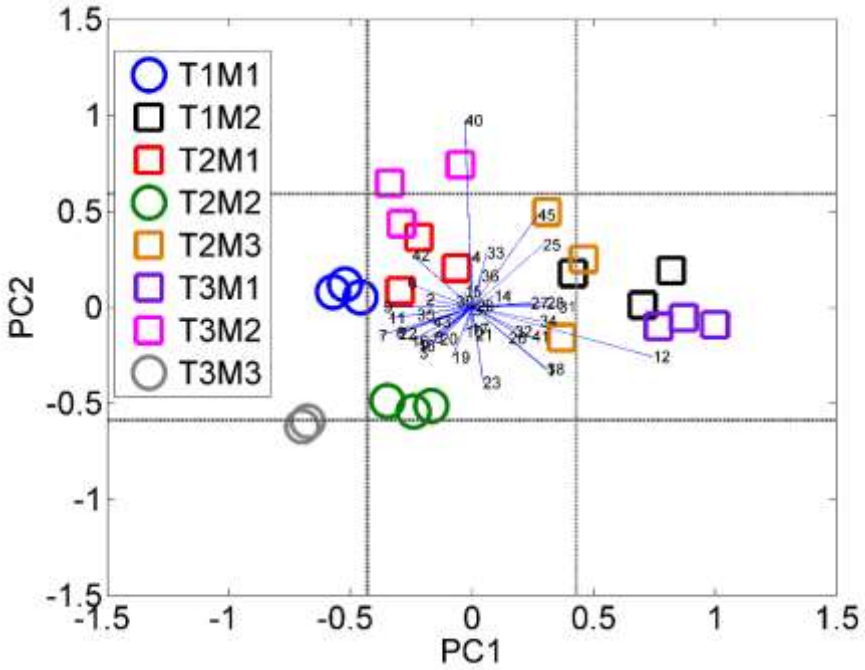

Figure S4. Bi-plot of volatile compounds of Syrah wines showing the first two principal components (PC1 = 15% and for PC2 = 12%) of the interaction between the factors maturation (T) *versus* maceration (M), by means ANOVA-PCA.

Table S1. Description of the samples of Syrah wines made with grapes harvested in different stages of maturation due to distinct periods of time of maceration

Wine code	Treatment (number of the maturation treatment) / °Brix	Days after pruning (DAP)	Maceration time / days
T_1M_1	1-19	113	10
T_1M_2	1-19	113	20
T_2M_1	2-21	120	10
T_2M_2	2-21	120	20
T_2M_3	2-21	120	30
T_3M_1	3-23	127	10
T_3M_2	3-23	127	20
T_3M_3	3-23	127	30

Table S2. Tentatively identified compounds of Syrah wines headspace with their respective odor threshold, odor activity value (OAV) and odor descriptor

	Volatile compound	CAS No.	LTPRI exp. DB-Wax[a]	LTPRI lit. DB-Wax	LTPRI exp. DB-5[b]	LTPRI lit. DB-5	OTS[e] / (μg L^{-1})	OAV								Odor descriptor
								T_1M_1	T_1M_2	T_2M_1	T_2M_2	T_2M_3	T_3M_1	T_3M_2	T_3M_3	
							Ester									
I	ethyl acetate	141-78-6	894	885[11d]			12000[13]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	fruity, melon[13]
II	2-methylpropyl acetate	110-19-0	1018	1018[21]	780	786[43]	1600[19]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	fruity, apple, banana[19]
1	3-methylbutyl acetate	123-92-2	1118	1127[46]	876	876[47]	30[2]	30.7	14.4	25.4	34.1	15.4	48.4	18.7	8.8	fruity, banana, sweet[13]
2	ethyl butanoate	105-54-4	1038	1034[44]	802	802[45]	20[2]	3.8	3.8	6.8	16.0	14.8	10.0	24.3	25.1	strawberry, apple[13]
3	ethyl hexanoate	123-66-0	1231	1244[11]	999	999[37]	14[5]	56.1	38.4	19.3	30.1	13.0	17.4	15.1	16.4	fruity, green apple, banana[13]
4	ethyl lactate	97-64-3	1345	1347[48]	813	813[25]	150000[13]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	fruity, buttery[13]
5	ethyl octanoate	111-11-5	1433	1446[11]	1197	1198[30]	580[49]	4.0	3.1	1.9	2.2	1.7	2.0	1.7	1.6	fruity, sweet, brandy, pear[13]
6	ethyl decanoate	110-38-3	1637	1651[11]	1394	1395[45]	200[5]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	fruity, grape, brandy[13]
7	3-methylbutyl octanoate	2035-99-6	1655	1658[31]	1444	1450[22]	152[29]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	oily[1], sweet, light fruity, cheese, cream[29]
8	diethyl butanedioate	123-25-1	1677	1677[38]	1179	1181[22]	1200[13]	< 1	1.0	2.8	2.3	2.5	< 1	2.1	6.3	fruity, melon[13]
9	ethyl 2-phenylacetate	103-45-7	1809	1805[51]	1252	1248[22]	1800[13]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	flowery[13]
10	ethyl 9-decenoate	67233-91-4	1688	1689[38]	1385	1386[30]	100[50]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	rose[1]
III	ethyl dodecanoate	106-33-2	1841	1835[51]	1592	1591[45]	3500[52,f]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	sweet, floral, fruity, cream[17]
11	ethyl tetradecanoate	124-06-1	2048	2048[53]	1793	1796[47]	800[17]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	Nf
12	ethyl hexadecanoate	628-97-7	2252	2261[24]			1500[29]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	fatty, rancid, fruity, sweet [29]
							Acid									
14	acetic acid	64-19-7	1476	1461[1]			200000[2]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	pungent, vinegar[3]
15	2-methylpropanoic acid	79-31-2	1591	1581[4]			2300[5]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	cheese[6]
16	butanoic acid	107-92-6	1651	1637[7]			173[5]	< 1	< 1	< 1	< 1	< 1	< 1	1.1	< 1	rancid, cheese[3]
17	3-methylbutanoic acid	503-74-2	1680	1682[8]	848	848[9]	33[5]	42.3	41.7	55.5	54.4	57.4	49.9	50.1	57.5	rancid, acid[10]
18	hexanoic acid	142-62-1	1864	1863[11]			420[5]	2.3	2.1	1.3	1.3	1.2	1.4	1.5	1.7	cheese, fatty
19	octanoic acid	124-07-2	2076	2083[11]	1184	1180[12]	500[5]	7.2	6.9	4.5	4.3	3.8	4.5	3.8	4.7	fatty, rancid[13]

20	nonanoic acid	112-05-0	2189	2193[14]	1272	1277[15]	3000[10,f]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	fatty[10]
21	decanoic acid	334-48-5	2292	2296[11]	1370	1371[16]	1000[5]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	fatty, unpleasant[17]
22	dodecanoic acid	143-07-7	2509	2517[11]	1558	1559[20]	1000[17]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	dry, metallic[17]
23	9-decenoic acid	14436-32-9	2355	2348[18]			40[19]	1.1	< 1	< 1	< 1	< 1	1.2	< 1	1.1	waxy, fatty, soapy[19]
							Alcohol									
IV	1-propanol	71-23-8	1044	1036[11]			306000[19]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	alcohol-like, ripe fruit[19]
V	2-methyl 1-propanol	78-83-1	1107	1093[1]			40000[17]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	fusel, alcohol[17]
25	3-methyl 1-butanol	137-32-6	1221	1221[21]	762	760[22,d]	30000[2]	2.0	3.0	2.5	3.2	3.5	2.1	2.8	< 1	solvent, nail polish[19]
26	(*Z*)-3-hexen-1-ol	928-96-1	1384	1382[26]	853	853[27]	1000[13]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	green, bitter, fatty[13]
27	1-octanol	111-87-5	1558	1552[31]	1069	1071[32]	800[19]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	jasmine, herbaceous [19]
28	1-nonanol	143-08-8	1660	1661[33]	1170	1172[34]	Nf	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nf
29	phenyl methanol	100-51-6	1874	1879[24]	1029	1032[35]	200000[3,f]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	fruity, sweet[3]
30	2-phenyl ethanol	60-12-8	1908	1909[36]	1110	1117[37]	14000[5]	1.5	2.2	2.3	3.0	3.2	2.3	3.3	3.9	rose, honey[13]
31	1-dodecanol	112-53-8	1968	1981[1]	1471	1471[25]	1000[17]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	unpleasant in higher concentration, flowery in low concentration[17]
32	4-methyl 1-pentanol	626-89-1	1316	1330[11]	836	Nf	50000[3,f]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	almond, toasted[3]
33	3-methyl 1-pentanol	589-35-5	1328	1331[1]	843	843[23]	50000[3,f]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	vinous, herbaceous, cacau[3]
34	1-hexanol	111-27-3	1354	1357[24]	868	868[25]	1100[13]	<1	2.5	1.4	2.2	2.5	1.6	2.0	2.3	herbaceous, grass, woody[13]
35	1-butanol	71-36-3	1155	1165[11]			150000[19]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	medicinal, wine-like[19]
VI	2-ethyl-1-hexanol	104-76-7	1491	1491[28]			8000[29]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	floral[10], sweet fruity[29]
36	2,3-butanediol	513-89-3	1542	1545[1]	786	789[30]	150000[3,f]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	fruity[3]
37	1,3-butanediol	107-88-0	1580	1578[33]	794	789[30]	Nf	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nf
38	1-hexadecanol	36653-82-4	2379	2382[38]			Nf	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nf
							Terpene									
40	linalool	78-70-6	1551	1549[14]	1097	1099[27]	15[13]	< 1	< 1	1	< 1	1.1	< 1	1.3	< 1	citrus, floral, sweet, grape-like[13]
41	geraniol	106-24-1	1849	1850[56]			30[2]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	roses, geranium[3]
42	(*E*)-nerolidol	7212-44-4	2043	2045[57]	1561	1563[43]	1000[13]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	rose, apple, green, citrus[13]
43	citronellol	106-22-9	1767	1771[55]	1226	1224[25]	100[2]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	sweet, citrus-like[55]
							Sulfur compound									
45	3-(methylthio)- 1-	505-10-2	1713	1715[4]			500[2]	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	cooked vegetable[3]

	propanol															
VII	benzothiazole	95-16-9	1938	1947[58]			Nf	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	ruber[10]
							Aldehyde									
VIII	benzaldehyde	100-52-7	1513	1529[21]	953	953[39]	2000[13]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	almond, fragant[13]
							Ketone									
IX	2,3-butanedione	431-03-8	984	998[40,c]			100[2]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	buttery[1]
X	2,3-pentanedione	600-14-6	1063	1064[41]			Nf	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	caramel, sweet[42]
							Lactone									
XI	butyrolactone	96-48-0	1616	1617[7]	905	901[54]	20000[13]	Nc	Nc	Nc	Nc	Nc	Nc	Nc	Nc	caramel, sweet[13]

CAS No.: Chemical Abstract Service number; [a]LTPRI expDB-Wax: experimental linear temperature programmed retention index (LTPRI) calculated using *n*-alkanes (C9-C24) in a polar column (DB-Wax, 100% polyethyleneglycol); [b]LTPRI expDB-5: experimental linear temperature programmed retention index (LTPRI) calculated using *n*-alkanes (C9-C24) in a non-polar column (5% phenyl-methylpolysiloxane); [c]experimental isothermal Kovats retention index calculated using *n*-alkanes (C6-C9) in a polar column (DB-Wax, 100% polyethyleneglycol); [d]experimental isothermal Kovats retention index calculated using *n*-alkanes (C6-C9) in a non-polar column (DB-5, 5% phenyl-methylpolysiloxane); LTPRI lit DB-Wax-isothermal or linear temperature programmed retention index (LTPRI) reported in scientific literature for a polar column (DB-Wax, 100% polyethyleneglycol); LTPRI lit.DB-5-isothermal or linear temperature programmed retention index (LTPRI) reported in scientific literature for a non-polar column (DB-5, 5% phenyl-methylpolysiloxane); [e]OTS: odor threshold determined in 10-12% (v/v) ethanol from literature; [f]odor threshold obtained in water; Nf: not found; Nc: OAV was not calculated because signal to noise for these specific compounds was less than ten or OTS was not found; Roman numbers represent the compounds identified and not quantified due to their low signal to noise ratio lower than three. Arabic numbers represents compounds quantified in Syrah wines.

Table S3. Results for the parameters of method validation for determination of volatiles compounds in Syrah wines by GC-MS

No. Volatile compound[a]	Regression equation	Concentration range / (µg L^{-1})	R^2	LOD[b] / (µg L^{-1})	LOQ[c] / (µg L^{-1})	Repeatability		Intermediate precision		Recovery[d] / %	Recovery[e] / %
						RSD[d] / %	RSD[e] / %	RSD[d] / %	RSD[e] / %		
					Ester						
(2) Ethyl butanoate	$y = 0.0006\,x + 0.0114$	30-2 500	0.993	9.9	30.0	14.5	1.2	18.6	9.0	106.6	94.6
(3) Ethyl hexanoate	$y = 0.0011\,x + 0.0499$	94-2 500	0.997	30.9	93.5	1.2	4.6	6.8	17.8	98.4	107.4
(5) Ethyl octanoate	$y = 0.0061\,x - 0.0430$	34-500	0.999	11.2	34.1	5.4	8.0	6.9	17.3	97.4	102.3
(6) Ethyl decanoate	$y = 0.0065\,x - 0.0759$	18-500	0.993	6.1	18.5	13.7	16.7	18.1	17.5	110.1	101.2
(8) Diethyl butanedioate	$y = 0.0024\,x - 0.7898$	344-10 000	0.999	113.4	343.8	6.5	7.2	19.4	6.4	99.3	99.4
					Acid						
(18) Hexanoic acid	$y = 0.0003\,x + 0.0143$	225-1 250	0.995	74.3	225.1	10.6	2.6	18.0	10.5	119.7	97.5
(19) Octanoic acid	$y = 0.0025\,x + 0.5624$	244-2 500	0.991	80.4	243.5	5.4	12.4	14.1	12.7	98.7	95.8
					Alcohol						
(29) Phenylmethanol	$y = 0.0003\,x + 0.0059$	102-1 000	0.990	33.6	101.8	16.6	15.5	13.1	15.7	97.2	95.7
(30) 2-Phenyl ethanol	$y = 0.0004\,x + 0.6670$	1101-50 000	0.996	363.3	1101.8	10.2	13.3	8.3	18.6	91.2	113.8
					Terpene						
(40) Linalool	$y = 0.0072\,x - 0.0486$	11-250	0.995	3.6	11.0	12.4	2.9	11.8	4.5	98.3	106.6
(42) (*E*)-Nerolidol	$y = 0.0052\,x - 0.0072$	25-250	0.998	8.2	25.0	9.8	14.0	19.4	18.5	107.3	103.1

[a]Compounds numbered as in Table S2; [b]LOD: limit of detection; [c]LOQ: limit of quantification; [d]calculated at the lower level of calibration; [e]calculated at the upper level of calibration.

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