

Table 1. Gradients: UPLC-MS (A: formic acid 1% and B: methanol:acetonitrile:iso-propanol (49:49:2)) and HPLC-UV (A: water and B: methanol)

UPLC-MS gradient				HPLC-UV gradient			
Time (min.)	Flow (mL/min)	Eluent A (%)	Eluent B (%)	Time (min.)	Flow (mL/min)	Eluent A (%)	Eluent B (%)
0.0	0.4	91	9	0	0.45	95	5
3.0	0.4	91	9	8	0.45	95	5
3.1	0.4	30	70	9	0.45	0	100
4.0	0.4	0	100	11	0.45	0	100
5.0	0.4	0	100	12	0.45	95	5
5.1	0.4	91	9	20	0.45	95	5
6.5	0.4	91	9				

Table 2. Linearity parameters of the UPLC-MS and HPLC-UV methods

Method	Matrix	R ²	LOD (µg/L)	LOQ (µg/L)
HPLC-UV	Wine	0.9986	0.259	0.862
	Model wine	0.9957	1.394	4.643
UPLC-MS	Wine	0.9953	0.001	0.003
	Model wine	0.9598	0.004	0.013

Table 3. Repeatability and accuracy parameters of the UPLC-MS and HPLC-UV methods

Method	Matrix	Repeatability peak area		Accuracy
		Level, $\mu\text{g/L}$	Average \pm SD	%
HPLC-UV	Wine	10	7.59 \pm 0.57	75.50
		20	12.79 \pm 1.42	78.61
	Model wine	10	7.54 \pm 0.65	
		20	15.18 \pm 2.53	
UPLC-MS	Wine	10	7.45 \pm 3.04	77.44
		20	14.32 \pm 2.54	87.72
	Model wine	10	8.43 \pm 0.72	
		20	12.16 \pm 0.83	

Table 4. Description and sotolon quantification of the dry and sweet white wines analysed (n.d. not detected; n.q. not quantified). The wines were listed according to vintage (from the youngest wine to the oldest). For wines 52 and 34, the cultivar was unknown and for wine 34 the vintage was unknown.

Code	Cultivars	Vintage	Description	Sotolon (µg/L) <i>UPLC-MS</i>	Sotolon (µg/L) <i>HPLC-UV</i>
58	Chenin blanc	2013	Dry white wine	0.62	n.d.
59	Chenin blanc	2013	Dry white wine	0.68	n.d.
60	Chenin blanc	2013	Dry white wine	0.49	n.d.
63	Sauvignon blanc	2011	Dry white wine	n.d.	n.d.
64	Sauvignon blanc	2011	Dry white wine	n.d.	n.d.
65	Semillon	2011	Dry white wine	1.96	2.56
16	Semillon	2010	Dry white wine	0.67	n.d.
17	Semillon	2010	Dry white wine	5.31	4.68
18	Pinot Grigio	2010	Dry white wine	1.55	n.d.
19	Chenin blanc	2010	Dry white wine	2.95	3.55
20	Colombard	2010	Dry white wine	0.55	n.d.
49	Chardonnay	2010	Dry white	9.11	8.13

			wine		
50	Sauvignon blanc	2010	Dry white wine	1.07	n.d.
61	Sauvignon blanc	2010	Dry white wine	0.79	n.d.
62	Semillon	2010	Dry white wine	0.57	n.d.
52	-	2010	Sweet wine	n.d.	n.d.
10	Chenin blanc	2010	Dry white wine	2.90	2.97
1	Sauvignon blanc	2009	Dry white wine	n.d.	n.d.
6	Viognier	2009	Dry white wine	1.36	n.d.
8	Sauvignon blanc	2009	Dry white wine	n.d.	n.d.
22	Chenin blanc	2009	Dry white wine	0.55	n.d.
21	Sauvignon blanc	2009	Dry white wine	4.96	7.17
25	Grenache blanc	2009	Dry white wine	1.34	n.d.
28	Semillon	2009	Dry white wine	0.88	n.d.
31	Chardonnay/Viognier	2009	Dry white wine	1.08	n.d.
43	Sauvignon blanc	2009	Dry white wine	n.d.	n.d.
48	Chardonnay	2009	Dry white	4.50	5.28

			wine		
51	Sauvignon blanc	2009	Dry white wine	n.d.	n.d.
9	Viognier	2009	Dry white wine	1.36	n.d.
3	Chardonnay	2008	Dry white wine	3.39	5.75
4	Chenin blanc	2008	Dry white wine	4.94	5.86
5	Viognier	2008	Dry white wine	4.72	7.36
12	Chardonnay	2008	Dry white wine	2.03	2.05
23	Chardonnay	2008	Dry white wine	4.73	3.34
24	Sauvignon blanc	2008	Dry white wine	2.17	n.q.
27	Viognier	2008	Dry white wine	1.31	n.d.
39	Chenin blanc	2008	Dry white wine	1.78	n.d.
44	Sauvignon blanc	2008	Dry white wine	n.d.	n.d.
42	Sauvignon blanc	2008	Dry white wine	n.d.	n.d.
47	Chardonnay	2008	Dry white wine	3.46	3.60
2	Chardonnay	2007	Dry white wine	4.98	7.73

7	Chenin blanc/Colombard	2007	Dry white wine	1.59	n.d.
14	Chardonnay	2007	Dry white wine	1.56	n.d.
15	Semillon	2007	Dry white wine	0.63	n.d.
29	Semillon	2007	Dry white wine	4.32	6.12
32	Viognier/Chenin blanc/Chardonnay	2007	Dry white wine	n.d.	n.d.
33	Rhine Riesling	2007	Dry white wine	n.d.	n.d.
41	Sauvignon blanc	2006	Dry white wine	n.d.	n.d.
45	Sauvignon blanc	2006	Dry white wine	n.d.	n.d.
11	Chardonnay	2005	Dry white wine	5.41	n.q.
13	Sauvignon blanc	2005	Dry white wine	4.97	6.16
26	-	2005	Dry white wine	n.d.	n.d.
30	Sauvignon blanc	2005	Dry white wine	1.34	n.d.
46	Sauvignon blanc	2005	Dry white wine	n.d.	n.d.
57	Chardonnay	2003	Dry white wine	1.62	n.d.
55	Chenin blanc	2002	Dry white wine	2.62	n.q.

53	-	2002	Dry white wine	0.80	n.d.
40	Chardonnay	2001	Dry white wine	2.60	n.q.
38	Chenin blanc	2001	Dry white wine	1.36	n.d.
54	Rhine Riesling	2000	Dry white wine	0.82	n.d.
56	-	1999	Sweet wine	8.72	n.q.
36	Gewurztraminer	1988	Dry white wine	2.19	n.q.
35	Colombard	1984	Dry white wine	0.85	n.d.
37	Colombard	1983	Dry white wine	2.04	n.q.
34	-	-	Dry white wine	1.57	1.42
<hr/>					
A	Sauvignon blanc	2011	Dry white wine	23.97	27.09
B	Sauvignon blanc	2011	Dry white wine	23.47	15.28
C	Sauvignon blanc	2011	Dry white wine	21.55	25.87
D	Sauvignon blanc	2011	Dry white wine	36.08	27.29
E	Sauvignon blanc	2011	Dry white wine	27.58	24.72
<hr/>					

Fig 1

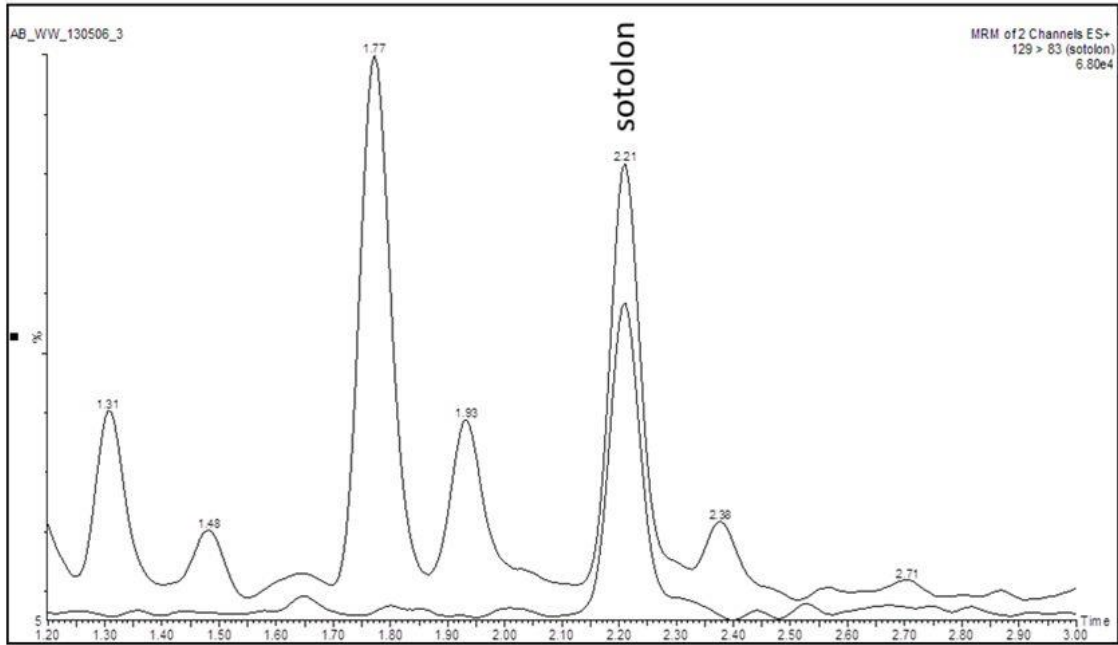


Fig 2

