

Supplementary materials

Table S1. Evaluation of repeatability performed on four extra-virgin olive oils of both compounds 3-Hydroxytyrosol and Tyrosol (day 2)

Compound	Sample	Average amount ¹ (in ppm)	RSD ² (in %)
3-Hydroxytyrosol	1	4.674	1.21
	2	2.987	0.39
	3	6.067	0.43
	4	4.242	0.46
Tyrosol	1	4.097	1.45
	2	4.909	0.88
	3	5.062	1.96
	4	5.457	0.27

¹ Average was determined on four-fold measurements of each hydrolyzed sample.

² Calculated relative standard deviation of the four injections put in relation to the detected average amount

Table S2. Evaluation of repeatability performed on four extra-virgin olive oils of both compounds 3-Hydroxytyrosol and Tyrosol (day 3)

Compound	Sample	Average amount ¹ (in ppm)	RSD ² (in %)
3-Hydroxytyrosol	1	4.709	0.38
	2	2.947	0.73
	3	6.070	0.78
	4	4.212	0.36
Tyrosol	1	4.132	1.23
	2	4.825	0.66
	3	5.090	1.04
	4	5.487	0.64

¹ Average was determined on four-fold measurements of each hydrolyzed sample.

² Calculated relative standard deviation of the four injections put in relation to the detected average amount.

Table S3. Optimization of hydrolysis time

Time (in min)	Average amount¹ (in ppm)	RSD² (in %)	Δ from the highest value³ (in %)
10	2.654	2.73	-30.2
20	3.156	1.28	-16.9
30	3.488	2.86	-8.20
40	3.611	0.64	-4.95
50	3.575	0.89	-5.91
60	3.755	1.18	-1.17
70	3.715	1.66	-2.24
80	3.776	1.76	-0.63
90	3.727	1.23	-1.93
100	3.699	1.28	-2.65
110	3.779	3.02	-0.55
120	3.800	1.66	-
150	3.666	1.56	-3.52
180	3.755	0.43	-1.19
240	3.618	1.09	-4.79
300	3.587	0.91	-5.61

¹Sum of tyrosol and hydroxytyrosol levels; average amount calculated over six injections.

² Calculated relative standard deviation of the four injections put in relation to the detected average amount.

³Difference from the highest value obtained during the hydrolysis time study was calculated.

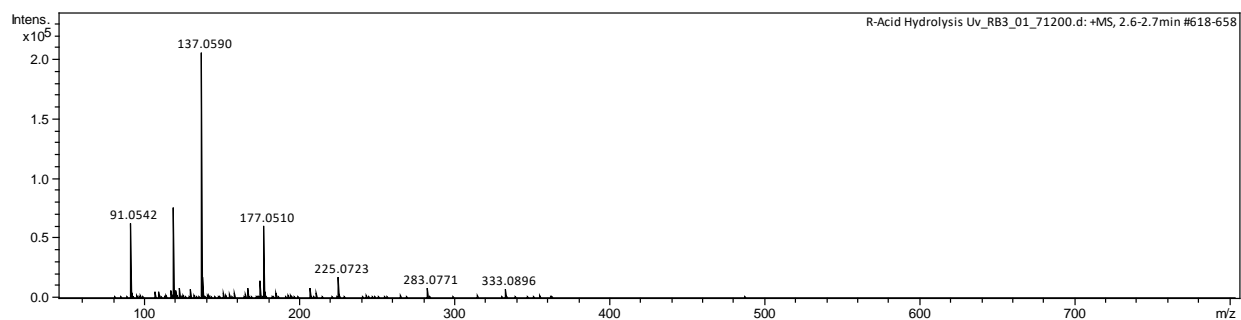


Figure S1. Recorded mass spectrum of hydroxytyrosol. 177.051 m/z was identified as $[M+Na]^+$, the basepeak at 137.059 corresponds to $[C_8H_9O_2]^+$. Electrospray ionization, positive mode, scan from 50-800 m/z, spectra rate 4 Hz.

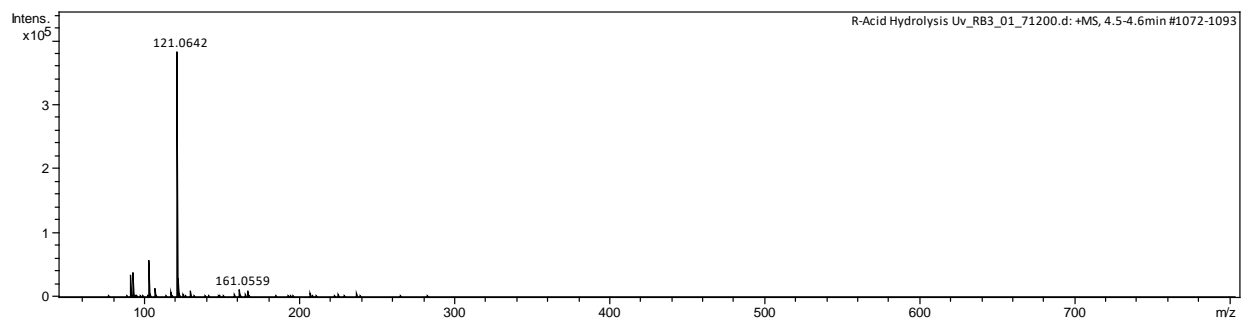


Figure S2. Recorded mass spectrum of tyrosol. 161.056 m/z was identified as $[M+Na]^+$, the basepeak at 121.064 corresponds to $[C_8H_9O]^+$. Electrospray ionization, positive mode, scan from 50-800 m/z, spectra rate 4 Hz.

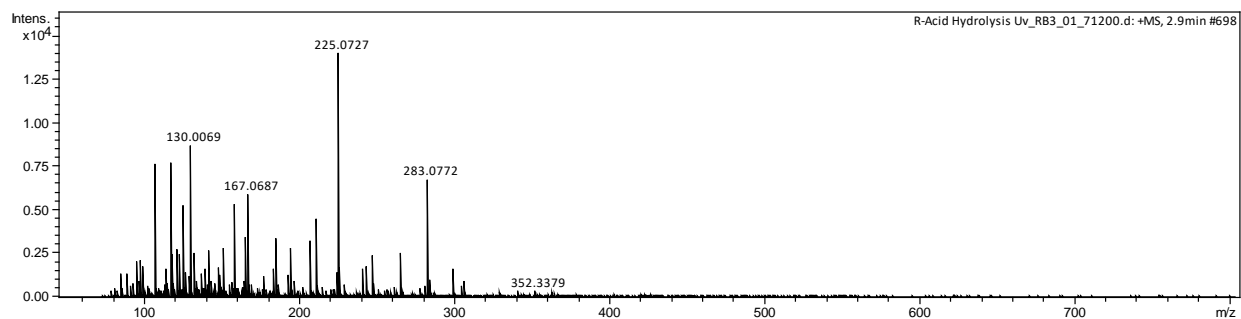


Figure S3. Recorded mass spectrum of the unknown signal eluting after hydroxytyrosol. Electrospray ionization, positive mode, scan from 50-800 m/z, spectra rate 4 Hz.

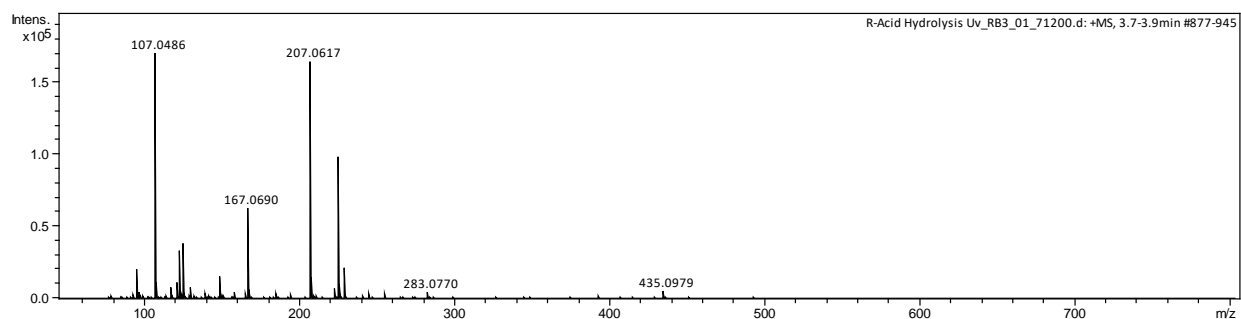


Figure S4. Recorded mass spectrum of the broad signal eluting between 3.9 min and 4.4 min. Electrospray ionization, positive mode, scan from 50-800 m/z, spectra rate 4 Hz.