

Table S1. Total Area of volatile compounds determined in organic Picual olive oil from washed olives by the HSSE-PDMS-GC-MS method.

Compounds				LRI				ID				Odour Descr.				WASHED												TT*	
																Day 1				Day 15				Day 32					
																MORNING (WM1)		AFTERNOON (WA1)		T	MORNING (WM15)		AFTERNOON (WA15)		T	MORNING (WM32)		AFTERNOON (WA32)	
Mean		±SD		Mean	±SD		Media	±SD		Media	±SD		Media	±SD		Media	±SD		M	A									
Acetic acid esters																													
Hexyl acetate				1247	A	Fruity	295318	9490	162483	87966	b	269551	29926	165826	228825	b	820213	22747	633566	84722	a								
(Z)-3-Hexenyl acetate				1291	A	Green	2234069	244982	1224258	574755	b	1645140	216040	777045	1313698	b	4123737	174669	3031396	701208	a								
Acids																													
Hexanoic acid				1841	A	Fatty	231558	117398	604222	346628		918927	154870	200350	389097		509981	397925	399836	707077									
Nonanoic acid				2168	B <sup>1</sup>	Waxy	657898	694488	690724	377810		676645	120820	407964	269276		344677	662294	737104	521137									
Alcohols																													
Methyl Alcohol				884	B <sup>2</sup>	Alcoholic	1169038	1055841	7230893	349024		1398755	167436	805153	6075672		631659	213016	1524508	526253									
Ethanol				912	A	Alcoholic	593238	167035	846285	93638	b	2743913	83174	639363	606854	b	3322023	684458	4145507	1830374	a								
1-Penten-3-ol				1138	A	Green	1212044	34675	555698	475776		876366	186475	424138	25426		469707	24510	400972	332032	a	b							
2-Hexyn-1-ol				1200	B <sup>2</sup>	-	2372765	156086	1013047	1013624	a	1676786	500976	328715	788075	b	282231	14599	357329	960533	b								
(Z)-2-Penten-1-ol				1310	A	-	1915287	20855	885381	711537		1197800	183098	435050	200897		712397	34588	614156	556643	a	b							
						Herbal,																							
1-Hexanol				1345	A	Green	443418	45296	225412	135629		464048	38173	1543780	357046		664828	21435	532903	752764									
(Z)-3-Hexen-1-ol				1374	A	Green	2062936	145145	1692921	186674		1699105	237851	7672215	902458		1519889	73836	1196197	4186705									
(E)-2-Hexen-1-ol				1396	A	Fruity	101330	31043	226368	106507		109753	13017	1348470	16299		75602	7462	65531	879632									
2-Furanmethanol				1654	A	Bready	2815666	603231	1449111	735841		1358478	1183802	1230065	546050		1694765	192640	1305560	187125									
1-Undecanol				1861	A	Waxy	1608656	303021	935589	819949		642526	243598	928441	6543		1523691	193320	728269	105474									
1-Dodecanol				1964	A	Waxy	2025033	990583	1600993	181878		1326333	866350	1490232	381742		953913	785634	1828283	276926									
Phenol				2003	B <sup>2</sup>	Phenolic	366227	107725	255883	49079		193444	115785	193267	28278		205388	57711	233056	29055									
2-Phenoxyethanol				2152	B <sup>2</sup>	Floral	374629	159926	160840	247203		180098	20039	175381	10671		157185	85454	185455	46081									
1-Hexadecanol				2376	B <sup>3</sup>	Waxy	412892	67847	450201	136947		370906	38147	588281	435823		482251	192452	743951	57478									
Aldehydes																													
Hexanal				1037	A	Green	3372282	62518	2674095	454086		4947488	202071	1741337	1114623		2965551	480975	2566368	2507582	a	b							

3-Methyl-2-butenal	1089		Green	495564	3916	348175	126267	393348	69491	0	171998	147026	10900	162375	283589		
(Z)-2-Hexenal	1091	B <sup>2</sup>	Green	1027580	285962	367141	623033	856412	324	0	441398	113069	141546	124317	534802	<i>a</i>	<i>b</i>
(E)-2-Hexenal	1193	B <sup>2</sup>	Green	17699433	1008983	8346466	6056111	23355622	647163	1308938	621470	10864006	801967	8633047	15990347	<i>a</i>	<i>b</i>
Nonanal	1372	B <sup>1</sup>	Aldehycic	3524212	921461	3706600	1197623	2712983	38109	3201342	573328	2560991	32196	2991517	329149		
Decanal	1484	A	Aldehycic	1358374	713879	1160936	488685	820568	113950	775398	222548	669749	80789	1078262	72338		
Benzaldehyde	1504	B <sup>2</sup>	Fruity	820631	294862	463061	100874	559334	59578	304478	135921	396236	47225	504171	203861		
(E)-2-Nonenal	1522	A	Fatty Caramelli	214806	33869	194554	13842	250348	5898	140844	41677	153838	16988	172686	85928		
5-Methylfurfural	1559	B <sup>4</sup>	c	166169	11192	101310	48582	93412	21172	75635	25676	104156	3769	90185	14492		
(E)-2-Decenal	1641	B <sup>5</sup>	Waxy	392955	77307	466740	106925	478968	30456	362124	96141	338410	52334	435636	108834		
2-Undecenal	1740	B <sup>2</sup>	Fruity	488662	168115	838816	339024	710192	61269	539272	295988	536789	120645	761410	181185		
<b>Hydrocarbures</b>																	
Hexadecane	1586	B <sup>2</sup>	-	429722	140259	338239	50655	389239	10005	262600	164194	269068	147863	404252	163482		
Nonadecane	1886	B <sup>2</sup>	-	211381	86438	274010	42725	261718	28070	205012	166661	203274	118770	199419	99485		
(Z)-1-Methoxyhex-3-ene	965	B <sup>2</sup>	Green	1224610	7272	551063	115435	1413941	240137	749901	398145	1710065	32494	1260785	485798		
3-Ethyl-1,5-octadiene	982	B <sup>2</sup>	-	1654513	146	942976	475054	1496310	151900	592661	228026	581359	113353	533067	695656	<i>a</i>	<i>b</i>
Pentadecane	1489	C	-	572629	148520	302359	104544	308661	68373	240435	110327	273599	33125	296873	64809		
Heptadecane	1700	B <sup>2</sup>	-	475797	295609	461060	6513	453969	4140	239834	285907	280164	232375	373595	267643		
Octadecane	1800	B <sup>2</sup>	-	347781	242852	385546	11347	430862	64	233626	266701	249244	198560	232613	238750		
<b>Esters</b>																	
Isopropyl Myristate	2023	B <sup>2</sup>	-	1201802	240743	1138782	400607	977683	149612	1230700	1048223	898534	354080	1966556	1866		
Methyl palmitate	2203	B <sup>2</sup>	Waxy	412185	215546	597447	371834	537873	177280	454701	1460433	353577	282736	673172	200183		
Isopropyl Palmitate	2230	B <sup>2</sup>	-	371486	216699	799716	352164	482864	320684	574118	1013616	383012	287051	1282093	79003	<i>b</i>	<i>a</i>
Methyl dihydrojasmonate	2280	B <sup>2</sup>	Floral	514117	348233	621948	104915	1560915	92849	381831	138650	296125	286663	462158	977077		
<b>Ketones</b>																	
4-Methyl-2-hexanone	842	C	-	6760720	2731197	10046807	3929562 a	4606456	549724	3948662	532109 b	3096752	805502	6546884	864349	ab	
2-Pentanone	940	B <sup>2</sup>	Fruity	1486259	278974	840093	300902	749163	254601	6465266	514377	875565	47562	733593	4018110		
1-Penten-3-one	976	B <sup>2</sup>	Spicy	2274106	52591	1090078	846696	2085248	300348	20596	825553	970197	95331	948853	1507630	<i>a</i>	<i>b</i>
2-Octanone	1262	A	Earthy Caramelli	555307	43983	723890	155198 a	205850	32883	145318	70839 b	147034	19417	163142	52515	b	
1-Hydroxy-2-propanone	1283	A	c	6536297	1513555	2799448	1946475	2544120	3148689	2686679	3002409	3264391	484389	2159632	141394		

6-Methyl-5-hepten-2-one	1316	A	Green	674811	139713	467830	74448	a	306942	26521	359247	49725	b	221123	19711	358359	27126	b
1-Hydroxy-2-butanone	1358	B <sup>2</sup>	Coffe	463585	189319	155550	146216		149737	281080	176018	231412		222990	8355	120495	14369	
Acetophenone	1637	A	Floral	195514	17245	310622	184953		204335	17622	113176	97403		117712	108579	324547	118752	
1,2-Cyclopentanedione	1771	B <sup>2</sup>	Floral	1429999	10875	709764	621887		743416	673583	848281	193979		1012135	4082	665223	72106	a b
2-Hydroxy-3-methyl-2-cyclopenten-1-one	1834	B <sup>2</sup>	-	336573	124835	150328	106605		130506	142938	152782	208142		181493	1763	112598	14867	
<b>Lactones</b>																		
5-Ethyl-2(5H)-furanone	1581	B <sup>2</sup>	-	741866	483466	273502	517351		620805	303247	0	333785		305461	34543	260149	471465	a b
γ-Butyrolactone	1620	A	Creamy	767850	164385	319821	231698		297185	384848	281480	263856		351041	50105	282029	36161	
2(5H)-Furanone	1747	A	Buttery	1132047	108662	551985	380484		492142	485209	481938	254176		620351	63685	506154	39062	
2-Hydroxy-γ-butyrolactone	2189	C	Creamy	550441	113124	279571	145643		262984	240311	395388	185185		411501	71871	220339	57684	
<b>(Sesqui)Terpenes</b>																		
Copaene	1468	C	Woody, Spicy	155003	30830	76472	40791		103519	25571	124322	23393		105396	13214	75110	8066	
α-Farnesene	1724	B <sup>2</sup>	Woody, Green	167019	42082	111362	32466	b	248249	39441	133926	53485	a b	328618	68894	246392	115289	a
<b>Others</b>																		
Pyrrole	1492	B <sup>2</sup>	Nutty	179452	58749	105071	25727		83696	51208	75933	26548		86256	13215	94149	12100	
Acetamide	1790	B <sup>2</sup>	-	207485	110355	160756	3900		90070	123688	85855	91597		98729	33406	175329	19863	

LRI: Experimental Linear Retention Index values estimated by linear regression. ID: reliability of identification: A. mass spectrum and LRI agreed with standards; B. mass spectrum agreed with mass spectral data base and LRI agreed with the literature data; C. mass spectrum agreed with mass spectral data base. Odour Descriptor: information obtained from The good scents company (<http://www.thegoodscentscompany.com>). Literature reference agreed with LRI data: <sup>1</sup>da Silva, Freitas, Cabrita & Garcia, (2012) [67]. <sup>2</sup>National Center for Biotechnology Information (2005) [68]. <sup>3</sup>Liang, Chen, Reeves & Han, (2013) [69]. <sup>4</sup>Fan & Qian (2006) [70]. <sup>5</sup>Sales et al., (2019) [71]. Mean: mean area values. SD: standard deviation. TTdays: Tukey's test (p<0.05) between the morning and afternoon harvest for each day. Different lower case letters indicate significant difference between days (where a>b). T\*: Tukey's test (p<0.05) among between the morning and afternoon harvest, regardless of the day. Different lower case italic letters indicate significant difference between days (where a>b). M: Morning; A: Afternoon.

**Table S2.** Total area of volatile compounds determined in organic Picual olive oil from non-washed olives by the HSSE-PDMS-GC-MS method.

			NON-WASHED																		
			Day 1				Day15				Day 32										
Compounds	LRI	ID	Odour Descr.	MORNING (NWM1)		AFTERNOON (NWA1)		T	MORNING (NWM15)		AFTERNOON (NWA15)		T	MORNING (NWM32)		AFTERNOON (NWA32)		T	TT		
				Media	±SD	Media	±SD		Media	±SD	Media	±SD		Media	±SD	Media	±SD				
Acetic acid esters																				a	b
Hexyl acetate	1247	A	Fruity	272711	251	139295	311259	b	441100	6816	246392	488867	b	761543	88081	543043	216237	a			
(Z)-3-Hexenyl acetate	1291	A	Green	2031009	74	1029263	1590334	b	2896687	55636	1619090	2168711	ab	3900895	330117	2633383	565995	a			
Acids																					
Hexanoic acid	1841	A	Fatty	170427	25154	290475	620709		291720	399001	353563	214882		195896	35120	343105	136464				
Nonanoic acid	2168	B <sup>1</sup>	Waxy	764275	3452	264623	432164		150058	7166	746147	257770		349076	414748	579778	340588				
Alcohols																					
Methyl Alcohol	884	B <sup>2</sup>	Alcoholic	1350368	312	1577160	318082		1386536	1982444	1071761	616015		788414	185513	453986	377204				
Ethanol	912	A	Alcoholic	1310486	7	16871965	3358745		4735263	66388	4365702	149442		4677828	257574	3510761	2159747				
1-Penten-3-ol	1138	A	Green	1122675	215	238794	82817		696469	8631	471587	52594		429824	36940	411558	497535	a	b		
2-Hexyn-1-ol	1200	B <sup>2</sup>	-	2468968	694	150513	14545		1939985	44167	1188785	21903		259013	49452	228287	1529911				
(Z)-2-Penten-1-ol	1310	A	-	1812679	4512	466796	287046		978226	17790	703961	93273		723863	62904	664813	822646	a	b		
1-Hexanol	1345	A	Herbal, Green	372282	541	1753531	705928		806984	40570	339223	144898		676352	59887	485353	91179				
(Z)-3-Hexen-1-ol	1374	A	Green	1989706	212	7826732	4436267		2304470	64028	1126244	70501		1515249	116941	1097073	566249				
(E)-2-Hexen-1-ol	1396	A	Fruity	93289	2556	1030591	902947		87004	23982	60663	31234		64671	4812	74751	14886				
2-Furanmethanol	1654	A	Bready	2691135	153	1029416	874351		1786234	362985	1409545	527101		1391741	347052	1169436	1153107	a	b		
1-Undecanol	1861	A	Waxy	1651083	25	935526	91464		748492	1219650	1361458	918770		906768	176324	613402	528710				
1-Dodecanol	1964	A	Waxy	2250822	315	898601	399154		1134304	784599	1758153	565966		1167883	1158964	1823112	818425				
Phenol	2003	B <sup>2</sup>	Phenolic	385886	254	159627	116830		221135	51355	222397	46739		206128	79791	235637	185585				
2-Phenoxyethanol	2152	B <sup>2</sup>	Floral	264810	541	166256	3762		160273	50869	214590	66284		107814	24260	171112	72215				
1-Hexadecanol	2376	B <sup>3</sup>	Waxy	519243	62	447104	64775		181217	209188	497195	410469		279673	490060	451008	211515				

Aldehydes																		
Hexanal	1037	A	Green	3193183	4113	1787056	1597414	3596590	165614	2370628	1068446	2317005	276551	2161385	827926	a	b	
3-Methyl-2-butenal	1089		Green	480584	2551	0	228347	345674	2711	264177	166928	160425	12036	155495	181961	a	b	
(Z)-2-Hexenal	1091	B <sup>2</sup>	Green	1060727	21563	0	76049	906405	4858	521488	11747	88872	579	83908	641451			
(E)-2-Hexenal	1193	B <sup>2</sup>	Green	18847598	1553	1829105	4781636	17909314	69266	13831812	4364939	9671635	949465	7668852	7338040	a	b	
Nonanal	1372	B <sup>1</sup>	Aldehycic	4127582	213	2736017	7620	3390059	847203	2570819	488121	2742919	1275648	2829201	1694032	a	b	
Decanal	1484	A	Aldehycic	1478251	2215	501755	32690	1257069	443049	774034	351913	724693	632456	775658	519607	a	b	
Benzaldehyde	1504	B <sup>2</sup>	Fruity	864647	22036	307295	236372	587416	43463	313535	47824	364141	103544	353636	375254	a	b	
(E)-2-Nonenal	1522	A	Fatty	194983	1256	133905	45097	ab	227358	82180	169105	33564	a	106432	47125	133210	49381	b
5-Methylfurfural	1559	B <sup>4</sup>	Caramellic	172149	2156	76802	59658	125721	17152	88139	26463	89281	31462	75270	82741	a	b	
(E)-2-Decenal	1641	B <sup>5</sup>	Waxy	398320	3165	347408	173847	ab	439605	131928	421133	66162	a	253698	112135	358833	74444	b
2-Undecenal	1740	B <sup>2</sup>	Fruity	600575	36102	547058	277141	532460	230159	773939	153452	401084	184757	649378	36536			
Hydrocarbures																		
Hexadecane	1586	B <sup>2</sup>	-	458767	51525	281097	28766	299437	129481	389569	80510	259892	191992	355409	91548			
Nonadecane	1886	B <sup>2</sup>	-	358358	22563	212570	10427	137968	56822	391233	22608	213343	79066	259292	175654			
(Z)-1-Methoxyhex-3-ene	965	B <sup>2</sup>	Green	1152245	2515	508003	202755	1562680	30470	883479	831556	1641580	218152	1145106	21804	a	b	
3-Ethyl-1,5-octadiene	982	B <sup>2</sup>	-	1784845	154641	856464	39132	a	1278233	1043	884274	5874	ab	609768	48665	535637	966994	b
Pentadecane	1489	C	-	487654	2113	193708	136904	394685	80205	277934	87477	255399	112696	235706	244316	a	b	
Heptadecane	1700	B <sup>2</sup>	-	609552	222665	351088	74902	248438	125039	516580	59402	322259	142764	542013	144808			
Octadecane	1800	B <sup>2</sup>	-	567775	64565	316435	6388	184971	115802	555541	40977	292138	93150	419717	212415			
Esters																		
Isopropyl Myristate	2023	B <sup>2</sup>	-	1440100	225623	1029976	580205	600473	572424	1909365	479530	720464	405119	1371174	452704			
Methyl palmitate	2203	B <sup>2</sup>	Waxy	711179	65526	840676	164666	275070	227348	875731	64498	444324	579250	500238	574298			
Isopropyl Palmitate	2230	B <sup>2</sup>	-	398262	55621	645217	100770	214920	335896	882711	212862	285796	312652	1335852	282806	b	a	
Methyl dihydrojasmonate	2280	B <sup>2</sup>	Floral	949087	5211	537622	81832	368853	200000	940045	87035	339019	2548	635865	603101			
Ketones																		
4-Methyl-2-hexanone	842	C	-	11359800	65413	6362457	23188	4501480	313712	5483010	30584	4136400	2268634	3851985	7123572			
2-Pentanone	940	B <sup>2</sup>	Fruity	1552972	3161	2369907	3739915	a	867812	43719	617944	26640	b	979447	179775	633952	798786	b
1-Penten-3-one	976	B <sup>2</sup>	Spicy	2138943	215633	29982	1097289	1706216	117175	1274142	529338	967179	139281	862830	873586	a	b	
2-Octanone	1262	A	Earthy	689932	25512	1553877	89433	a	175132	19037	274717	21240	b	182901	51221	158793	404459	b

1-Hydroxy-2-propanone	1283	A	Caramellic	5984501	55264	1842868	926278	3179873	552776	2588823	1421832	2940142	454847	2117855	3115667	a	b	
6-Methyl-5-hepten-2-one	1316	A	Green	610627	25113	322527	80410	938578	68782	329175	52155	213820	62486	250881	203374			
1-Hydroxy-2-butanone	1358	B <sup>2</sup>	Coffe	398299	5215	136902	69037	203147	30984	149891	72663	178719	12670	147475	177505	a	b	
Acetophenone	1637	A	Floral	399024	525	129569	14647	211238	17761	179424	24250	112522	256227	128010	342171			
1,2-Cyclopentanedione	1771	B <sup>2</sup>	Floral	1215705	2563	705180	228282	998439	347002	778302	372502	773824	3974	588377	393986	a	b	
2-Hydroxy-3-methyl-2-cyclopenten-1-one	1834	B <sup>2</sup>	-	280923	253	93050	65680	184442	61490	137816	87671	146854	2860	130129	105350	a	b	
Lactones																		
5-Ethyl-2(5H)-furanone	1581	B <sup>2</sup>	-	259302	25411	85354	972	b	739424	47535	465452	104604	a	210531	14063	64415	6849	b
γ-Butyrolactone	1620	A	Creamy	686135	2551	196986	164330	381036	57538	305841	152004	321875	79398	203765	353858	a	b	
2(5H)-Furanone	1747	A	Buttery	1085334	551	368114	312721	698299	145335	546069	212517	539666	128745	404765	569415	a	b	
2-Hydroxy-γ-butyrolactone	2189	C	Creamy	506245	23512	191791	554	268159	66204	276196	243276	268029	54752	190055	261771			
(Sesqui)Terpenes																		
Copaene	1468	C	Woody, Spicy	159086	2255	73432	18850	116479	13473	98103	28219	85791	12268	66888	59553	a	b	
α-Farnesene	1724	B <sup>2</sup>	Woody, Green	182041	6314	86902	109696	b	197397	94272	184804	162959	ab	297967	21145	222196	52988	a
Others																		
Pyrrole	1492	B <sup>2</sup>	Nutty	186217	2511	63826	57650	114980	25558	82748	30314	86332	33459	85888	89635	a	b	
Acetamide	1790	B <sup>2</sup>	-	235761	52654	73295	129835	120587	39207	112836	31344	105404	100254	115034	91938			

LRI: Experimental Linear Retention Index values estimated by linear regression. ID: reliability of identification: A. mass spectrum and LRI agreed with standards; B. mass spectrum agreed with mass spectral data base and LRI agreed with the literature data; C. mass spectrum agreed with mass spectral data base. Odour Descriptor: information obtained from The good scents company (<http://www.thegoodscentscompany.com>). Literature reference agreed with LRI data: <sup>1</sup>da Silva, Freitas, Cabrita & Garcia, (2012) [67]. <sup>2</sup>National Center for Biotechnology Information (2005) [68]. <sup>3</sup>Liang, Chen, Reeves & Han, (2013) [69]. <sup>4</sup>Fan & Qian (2006) [70]. <sup>5</sup>Sales et al., (2019) [71]. Mean: mean area values. SD: standard deviation. TTdays: Tukey's test (p<0.05) between the morning and afternoon harvest for each day. Different lower case letters indicate significant difference between days (where a>b). T\*: Tukey's test (p<0.05) among between the morning and afternoon harvest, regardless of the day. Different lower case italic letters indicate significant difference between days (where a>b). M: Morning; A: Afternoon.