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## DELIVERABLE 4.5

Title: Final report on the selection and sensory/chemical analyses of two surely genuine EVOOs

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LEAD BENEFICIARY: UNIBO

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Dissemination Level		
PU	Public	X
PP	Restricted to other programme participants (Including the Commission Services)	
RE	Restricted to a group specified by the consortium (Including the Commission Services)	
CO	Confidential, only for members of the consortium (Including the Commission Services)	



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### 1. Executive summary

This deliverable is focused on the selection of the extra virgin olive oils (EVOOs) to be sampled in the context of Tasks 4.1/4.2 and Task 4.3, as well as on the related information and the results of the sensory and chemical analyses.

These samples were collected, as described in the Task 4.1 (ST 4.1.2), along two different samplings, then analyzed as raw and used for preparing blends for the Task 4.2. Some of the same samples were also be object of the analytical activities scheduled in the Task 4.3.

### 2. Objectives

For Tasks 4.1 and 4.2, the EVOOs were adequately blended with soft-deodorized olive oils, by following the guidelines reported in the D.4.1, during two samplings and with the goal to obtain the desired blends to be analyzed.

For each of the two samplings, it has been decided to sample an EVOO with medium/high intensity of fruitiness and another one characterized by low fruitiness, in order to better simulate a realistic fraudulent scenario. For the same reason, the selected EVOOs were all commercial products sold at low price.

In particular, within the first sampling, each partner had the possibility to prepare blends at different percentages in its own laboratory, while in the framework of the second sampling Fera was responsible for preparing the blends by mixing two EVOOs with ten soft-deodorized olive oils at three different percentages, thus obtaining 60 blind blends shipped to the T.4.2 analytical partners.

The partners received also the corresponding raw EVOOs, for analyzing them and for preparing further blends at different percentages.

For Task 4.3, two out of the four EVOOs samples, collected for the T.4.1 and T.4.2, were used for preparing legal and illegal blends, respectively with virgin hazelnut and virgin avocado oils and with refined conventional sunflower and refined high oleic sunflower oils.

For preparing all these blends, the D.4.1 was carefully followed. In particular, for the first sampling, each T.4.3 analytical partner had the possibility to prepare in lab its own illegal blends, while Fera prepared the legal blends. On the contrary, for the second sampling each T.4.3 analytical partner had the possibility to prepare its own legal blends, while Fera prepared blind illegal blends.

### **3. Samples information and results of the sensory and chemical analyses**

A technical protocol addressed to the olive oil companies providing EVOOs was prepared for each sampling, according to the D.3.1; herein some instructions were specified on how to perform a proper sampling and subsequent storage of the oils. The volumes needed for each sampled oil and the specific requested information were also detailed. Moreover, an analytical plan was agreed among the Partners for defining the determinations to be performed on the samples.

Both the samples information and the results of the sensory and chemical analyses were made available to all the T.4.2 and T.4.3 analytical partners in the OLEUM Project Basecamp and can be found in the next pages of this D.

Only sure information were indicated in the table; for all the others, "not available" was stated. The names of the company providing the samples were not shown in the following labels, as agreed with the olive oil companies according to the technical protocol.

All the limits for EVOOs reported in the tables refer to the ones established in the current EU Reg. 2095/2016; all the chemical and sensory results on the four samples were compliant with these limits.

<b>OLIVE OIL COMPANY:</b>	<b>CODE SAMPLE:</b>
-	EVOO_H_1
Date of sampling	March 2017 (first sampling)
Olive typology <i>(geographic origin of the olives, specify here if the olives were collected from: i. a single member of EU ii. EU iii. a third country outside EU)</i>	Spain
Olive typology <i>(olive variety/varieties, PDO, PGI)</i>	Main cultivar: Manzanilla
Olive typology <i>(sanitary state of olives, time and storage conditions of olives before milling)</i>	Fresh and healthy olives collected only from the trees. Conventional antiparasitic treatments.
Mill location <i>(location of the mill in which the oil was obtained)</i>	Seville, Spain
Technology parameters <i>(press/2-phases/3-phases, capacity of the production line)</i>	2 phases
Date of production of the oil	18-24/11/2016
Date of starting of oil storage	18/01/2017
Type of tank/bottles <i>(material, insulation, inert gas)</i>	Steel tank
Technology parameters <i>(filtered, not filtered)</i>	Not filtered
Volume of tank / oil mass & volume of the sampled oil	Volume of tank 18000 kg / oil mass 17000 kg & about 45 L
Oil temperature <i>(at the moment of sampling)</i>	17 °C
Oil quality grade <i>(extra virgin, virgin, lampante); please specify here also the defect and its median value (if found)</i>	EVOO

*-IOC Panel test (EU Reg. 1227/2016)*

Fruity: 6.1; Bitter: 6.3; Pungent: 5.6

*-Determination of the fatty acids composition, as fatty acid methyl esters (EEC Reg. 2568/1991)*

<b>Fatty acids</b>	<b>%</b>	<b>limit EU %</b>
C14:0	0.01	≤ 0.03
C16:0	12.44	7.50-20.00
C16:1	1.04	0.30-3.50
C17:0	0.22	≤ 0.40
C17:1	0.3	≤ 0.60
C18:0	3.63	0.50-5.00
C18:1	74.37	55.00-83.00
C18:2	6.3	2.50-21.00
C20:0	0.47	≤ 0.60
C18:3	0.77	≤ 1.00
C20:1	0.25	≤ 0.50
C22:0	0.13	≤ 0.20
C22:1	n.d	
C24:0	0.07	≤ 0.20

*-Basic quality parameters (free acidity, peroxide values, UV spectrophotometric indexes), according to EEC Reg. 2568/1991 and following amendments*

		<b>limit EU</b>
Free acidity	0.26%	≤ 0.8
Peroxide value	3 meq O <sub>2</sub> /kg oil	≤ 20
K <sub>268</sub>	0.17	≤ 0.22
K <sub>232</sub>	1.82	≤ 2.50

*-Determination of the ethyl esters of fatty acids and waxes (EU Reg. 61/2011)*

Fatty acid ethyl esters: 0.4 mg/kg (limit EU:  $\leq 35$  mg/kg)

*-Determination of sterols composition (EEC Reg. 2568/91)*

<b>Sterols</b>	<b>%</b>	<b>limit EU %</b>
cholesterol	0.28	$\leq 0.5$
brassicasterol	n.d	$\leq 0.1$
24-methylencholesterol	0.1	
campesterol	2.58	$\leq 4.0$
campestanol	0.07	
stigmasterol	0.64	< camp.
delta-7-campesterol		
delta-7-campesterol		
delta-5,23-stigmastadienol		
chlerosterol	1.1	
beta-sitosterol	86.88	
sitostanol	0.75	
delta-5-avenasterol	6.06	
delta-5,24-stigmastadienol	0.56	
delta-7-stigmastenol	0.27	$\leq 0.5$
delta-7-avenasterol	0.68	
app. beta-sitosterol	95.37	$\geq 93.0$
<b>TOTAL STEROLS</b>	<b>1265</b>	<b><math>\geq 1000</math> mg/kg</b>

Erythrodiol + uvaol, 1.51 % (limit EU  $\leq 4.5$  %)

<b>OLIVE OIL COMPANY:</b>	<b>CODE SAMPLE:</b>
-	EVOO_2_L
Date of sampling	March 2017 (first sampling)
Olive typology <i>(geographic origin of the olives, specify here if the olives were collected from: i. a single member of EU ii. EU iii. a third country outside EU)</i>	Italy
Olive typology <i>(olive variety/varieties, PDO, PGI)</i>	cultivar Biancolilla
Olive typology <i>(sanitary state of olives, time and storage conditions of olives before milling)</i>	well-preserved olives with medium ripening degree
Mill location <i>(location of the mill in which the oil was obtained)</i>	Sicily, Italy
Technology parameters <i>(press/2-phases/3-phases, capacity of the production line)</i>	3 phases
Date of production of the oil	2015-2016
Date of starting of oil storage	December 2015
Type of tank/bottles <i>(material, insulation, inert gas)</i>	steel tank with inert gas
Technology parameters <i>(filtered, not filtered)</i>	filtered
Volume of tank / oil mass & volume of the sampled oil	60000 kg & 9.5 L
Oil temperature <i>(at the moment of sampling)</i>	17.7 °C
Oil quality grade <i>(extra virgin, virgin, lampante); please specify here also the defect and its median value (if found)</i>	EVOO

*-IOC Panel test (EU Reg. 1227/2016)*

Fruity: 2.9; Bitter: 3.8; Pungent: 3.8

*-Determination of the fatty acids composition, as fatty acid methyl esters (EEC Reg. 2568/1991)*

<b>Fatty acids</b>	<b>%</b>	<b>limit EU %</b>
C14:0	0.01	≤ 0.03
C16:0	13.43	7.50-20.00
C16:1	1.07	0.30-3.50
C17:0	0.17	≤ 0.40
C17:1	0.33	≤ 0.60
C18:0	2.61	0.50-5.00
C18:1	70.56	55.00-83.00
C18:2	10.12	2.50-21.00
C20:0	0.45	≤ 0.60
C18:3	0.73	≤ 1.00
C20:1	0.33	≤ 0.50
C22:0	0.12	≤ 0.20
C22:1	n.d	
C24:0	0.07	≤ 0.20

*-Basic quality parameters (free acidity. peroxide values. UV spectrophotometric indexes), according to EEC Reg. 2568/1991 and following amendments*

		<b>limit EU</b>
Free acidity	0.19%	≤ 0.8
Peroxide value	4.2 meq O <sub>2</sub> /kg oil	≤ 20
K <sub>268</sub>	0.124	≤ 0.22
K <sub>232</sub>	1.59	≤ 2.50



*-Determination of the ethyl esters of fatty acids and waxes (EU Reg. 61/2011)\**

Fatty acid ethyl esters: 1.0 mg/kg (limit EU: ≤ 35 mg/kg)

Waxes (C<sub>42</sub> + C<sub>44</sub> + C<sub>46</sub>): 23.2 mg/kg (limit EU: ≤ 150 mg/kg)

*-Determination of sterols composition (EEC Reg. 2568/91)*

<b>Sterols</b>	<b>%</b>	<b>limit EU %</b>
cholesterol	0.1	≤ 0.5
brassicasterol	n.d	≤ 0.1
24-methylencholesterol	0.2	
campesterol	2.5	≤ 4.0
campestanol	0.1	
stigmasterol	0.7	< camp.
delta-7-campesterol	n.d	
delta-7-campesterol		
delta-5.23-		
stigmastadienol	n.d	
chlerosterol	1.0	
beta-sitosterol	78.7	
sitostanol	0.6	
delta-5-avenasterol	13.9	
delta-5.24-		
stigmastadienol	1.2	
delta-7-stigmastenol	0.4	≤ 0.5
delta-7-avenasterol	0.6	
app. beta-sitosterol	95.4	≥ 93.0
<b>TOTAL STEROLS</b>	<b>1142</b>	<b>≥ 1000 mg/kg</b>



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<b>OLIVE OIL COMPANY:</b>	<b>CODE SAMPLE:</b>
-	EVOO_H_2
Date of sampling	24/01/2018 (second sampling)
Olive typology <i>(geographic origin of the olives, specify here if the olives were collected from: i. a single member of EU ii. EU iii. a third country outside EU)</i>	Spain
Olive typology <i>(olive variety/varieties, PDO, PGI)</i>	cultivar Hojiblanca
Olive typology <i>(sanitary state of olives, time and storage conditions of olives before milling)</i>	Not available
Mill location <i>(location of the mill in which the oil was obtained)</i>	Sevilla, Spain
Technology parameters <i>(press/2-phases/3-phases, capacity of the production line)</i>	2 phases, 200 tonn/24 hours
Date of production of the oil	5-6/12/2017
Date of starting of oil storage	7/12/2017
Type of tank/bottles <i>(material, insulation, inert gas)</i>	Steel tank
Technology parameters <i>(filtered, not filtered)</i>	Not filtered
Volume of tank / oil mass & volume of the sampled oil	60000 L
Oil temperature <i>(at the moment of sampling)</i>	16 °C
Oil quality grade <i>(extra virgin, virgin, lampante); please specify here also the defect and its median value (if found)</i>	EVOO

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*-IOC Panel test (EU Reg. 1227/2016)*

Fruity: 4.8; Bitter: 5.7; Pungent: 4.9

*-Determination of the fatty acids composition, as fatty acid methyl esters (EEC Reg. 2568/1991)*

<b>Fatty acids</b>	<b>%</b>	<b>limit EU %</b>
C14:0	0.01	≤ 0.03
C16:0	10.88	7.50-20.00
C16:1	0.72	0.30-3.50
C17:0	0.14	≤ 0.40
C17:1	0.21	≤ 0.60
C18:0	2.96	0.50-5.00
C18:1	76.75	55.00-83.00
C18:2	7.02	2.50-21.00
C20:0	0.33	≤ 0.60
C18:3	0.63	≤ 1.00
C20:1	0.18	≤ 0.50
C22:0	0.07	≤ 0.20
C22:1	n.d	
C24:0	0.11	≤ 0.20

*-Basic quality parameters (free acidity, peroxide values, UV spectrophotometric indexes), according to EEC Reg. 2568/1991 and following amendments*

			<b>limit EU</b>
Free acidity	0.29%		≤ 0.8
Peroxide value	5 meq O <sub>2</sub> /kg oil		≤ 20
K <sub>270</sub>	0.134		≤ 0.22
K <sub>232</sub>	1.649		≤ 2.50



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*-Determination of the ethyl esters of fatty acids and waxes (EU Reg. 61/2011)*

Fatty acid ethyl esters: 0.78 mg/kg (limit EU:  $\leq 35$  mg/kg)

Waxes ( $C_{42} + C_{44} + C_{46}$ ): 41.6 mg/kg (limit EU:  $\leq 150$  mg/kg)

*-Determination of sterols composition (EEC Reg. 2568/91)*

<b>Sterols</b>	<b>%</b>	<b>limit EU %</b>
cholesterol	0.14	$\leq 0.5$
brassicasterol	n.d	$\leq 0.1$
campesterol	2.73	$\leq 4.0$
stigmasterol	0.45	$< \text{camp.}$
delta-7-stigmastenol	0.30	$\leq 0.5$
app. beta-sitosterol	95.47	$\geq 93.0$
<b>TOTAL STEROLS</b>	<b>1550</b>	<b><math>\geq 1000</math> mg/kg</b>

Erythrodiol + uvaol, 0.42 % (limit EU  $\leq 4.5$  %)



<b>OLIVE OIL COMPANY:</b>	<b>CODE SAMPLE:</b>
-	EVOO_L_2
Date of sampling	10/01/18 (second sampling)
Olive typology <i>(geographic origin of the olives, specify here if the olives were collected from: i. a single member of EU ii. EU iii. a third country outside EU)</i>	Greece
Olive typology <i>(olive variety/varieties, PDO, PGI)</i>	Koroneiki
Olive typology <i>(sanitary state of olives, time and storage conditions of olives before milling)</i>	Not available
Mill location <i>(location of the mill in which the oil was obtained)</i>	Not available
Technology parameters <i>(press/2-phases/3-phases, capacity of the production line)</i>	Not available
Date of production of the oil	Not available
Date of starting of oil storage	Not available
Type of tank/bottles <i>(material, insulation, inert gas)</i>	Steel tank with inert gas (N <sub>2</sub> )
Technology parameters <i>(filtered, not filtered)</i>	Filtered
Volume of tank / oil mass & volume of the sampled oil	Not available
Oil temperature <i>(at the moment of sampling)</i>	18-20 °C
Oil quality grade <i>(extra virgin, virgin, lampante); please specify here also the defect and its median value (if found)</i>	EVOO

*-IOC Panel test (EU Reg. 1227/2016)*

Fruity: 3.0; Bitter: 3.2; Pungent: 4.0

*-Determination of the fatty acids composition, as fatty acid methyl esters (EEC Reg. 2568/1991)*

<b>Fatty acids</b>	<b>%</b>	<b>limit EU %</b>
C14:0	0.01	≤ 0.03
C16:0	11.74	7.50-20.00
C16:1	0.92	0.30-3.50
C17:0	0.06	≤ 0.40
C17:1	0.09	≤ 0.60
C18:0	2.72	0.50-5.00
C18:1	76.55	55.00-83.00
C18:2	6.18	2.50-21.00
C20:0	0.47	≤ 0.60
C18:3	0.70	≤ 1.00
C20:1	0.34	≤ 0.50
C22:0	0.16	≤ 0.20
C24:0	n.d	≤ 0.20

*-Basic quality parameters (free acidity. peroxide values. UV spectrophotometric indexes), according to EEC Reg. 2568/1991 and following amendments.*

		<b>limit EU</b>
Free acidity	0.27%	≤ 0.8
Peroxide value	3.98 meq O <sub>2</sub> /kg oil	≤ 20
K <sub>270</sub>	0.129	≤ 0.22
K <sub>232</sub>	1.55	≤ 2.50

*-Determination of the ethyl esters of fatty acids and waxes (EU Reg. 61/2011)\**

Fatty acid ethyl esters: 0.7 mg/kg (limit EU: ≤ 35 mg/kg)

Waxes (C<sub>42</sub> + C<sub>44</sub> + C<sub>46</sub>): 51 mg/kg (limit EU: ≤ 150 mg/kg)

*-Determination of sterols composition (EEC Reg. 2568/91)*

<b>Sterols</b>	<b>%</b>	<b>limit EU %</b>
cholesterol	0.07	≤ 0.5
brassicasterol	0.01	≤ 0.1
campesterol	3.41	≤ 4.0
stigmasterol	0.62	< camp.
delta-7-stigmastenol	0.22	≤ 0.5
app. beta-sitosterol	94.81	≥ 93.0
<b>TOTAL STEROLS</b>	<b>1271.6</b>	<b>≥ 1000 mg/kg</b>

Erythrodiol + uvaol, 4.08 % (limit EU ≤ 4.5 %)