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PRECISION VALUES OF THE METHODS OF ANALYSIS ADOPTED BY THE INTERNATIONAL OLIVE COUNCIL

In compliance with the proposal of the group of chemical experts of the International Olive Council (IOC), the Executive Secretariat set up a electronic working group which was given the brief of reviewing expression of results and the precision values of the physico–chemical testing methods drawn up and adopted by the IOC and inserted in the trade standard for olive oils and olive-pomace oils..

The electronic working group drew up this document for approval at the 106th IOC session to be held in Madrid, Spain, on 21-24 November 2017.

The document reports the precision values for the following methods bearing the reference COI/T.20:

Reference	Method
COI/T.20/Doc. no 18/ Rev.3	Determination of the content of waxes, fatty acid methyl esters and fatty acid ethyl esters by capillary gas chromatography
COI/T.20/Doc. n° 20/ Rev.4	Determination of the difference between actual and theoretical content of triacylglycerols with ECN42
COI/T.20/Doc. n° 11/ Rev.3	Determination of stigmastadienes in vegetable oils
COI/T.20/Doc. n° 16/ Rev.2	Determination of sterenes in refined vegetable oils
COI/T.20/Doc. n° 26/ Rev.4	Determination of the sterol composition and content and alcoholic compounds by capillary gas chromatography
COI/T.20/Doc. n° 19/ Rev.5	Spectrophotometric analysis in the ultraviolet
COI/T.20/Doc. n° 23/ Rev.1	Determination of the percentage of 2-glyceryl monopalmitate
COI/T.20/Doc. n° 29/ Rev.1	Determination of biophenols in olive oils by HPLC
COI/T.20/Doc. n° 34/ Rev.1	Determination of free acidity, cold method
COI/T.20/Doc. n° 35/ Rev.1	Determination of the peroxide value
COI/T.20/Doc. n° 33/ Rev.1	Determination of fatty acid methyl esters by gas chromatography

The precision values for the following methods :

- COI/T.20/Doc. no. 16 – Determination of sterenes in refined vegetable oils;

- COI/T.20/Doc. no. 33 – Determination of fatty acid methyl esters by gas chromatography (relative solely to heptadecanoic acid and heptadecenoic acid)

have been calculated from the data for 2000–2017 supplied by the laboratories of many countries for earning entitlement under the IOC recognition scheme. The results underwent statistical analysis according to ISO 5725 "Accuracy (trueness and precision) of measurement methods and results" and with the aid of the AOAC Statistical Manual (W.J. Youden, E.H. Steiner). Outliers were detected by applying the Ranking, Cochran and Grubbs tests to the laboratory results for all the samples (replicates a and b).

The tables on the next pages report the following data for each parameter studied:.

n	number of laboratories which participated in the test
outliers	number of laboratories with outlying values
mean	mean of the accepted results
r	repeatability
S_r	repeatability standard deviation
RSD_r(%)	repeatability coefficient of variation (S_r x 100/ mean)
R	reproducibility
S_R	reproducibility standard deviation
RSD_R(%)	reproducibility coefficient of variation (S_R x 100/ mean)

Table	1
Analysis	Determination of the content of waxes, fatty acid methyl esters and fatty acid ethyl esters by capillary gas chromatography
Method	COI/T.20/Doc.n°18/Rev.3
Parameter	Waxes - Ring Test COI 1999
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil
B: virgin olive oil + refined sunflower oil
C: virgin olive oil + refined olive-pomace oil
D: virgin olive oil + refined soybean oil + refined sunflower oil
E: refined olive oil + refined olive-pomace oil + refined soybean oil + lampante virgin olive oil

	A	B	C	D	E
n	19	19	19	19	19
outliers	5	5	4	3	5
mean	120	123	222	174	346
r	9.5	12.6	10.5	12.2	14.9
S_r	3.4	4.5	3.8	4.7	5.3
RSD_r(%)	2.8	3.6	1.7	2.7	1.5
R	38.8	48.9	58.9	25.7	44.4
S_R	13.9	17.5	21.0	9.2	15.9
RSD_R(%)	11.5	14.2	9.5	5.3	4.6

Table	2
Analysis	Determination of the content of waxes, fatty acid methyl esters and fatty acid ethyl esters by capillary gas chromatography
Method	COI/T.20/Doc.n°18/Rev.3
Parameter	Waxes - Ring Test COI 2008
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive retail Italy

B: extra virgin olive retail Italy

C: extra virgin olive retail + refined

D: extra virgin olive oil + lampante

E: extra virgin olive oil + retail Germany

	A	B	C	D	E
n	20	18	19	18	18
outliers	2	1	0	0	0
mean	125	181	199	142	174
r	9.8	13.0	20.1	17.6	12.2
S_r	3.3	4.4	6.8	5.9	4.1
$RSD_r(\%)$	2.7	2.4	3.4	4.2	2.4
R	87.3	75.4	67.9	82.7	44.0
S_R	29.5	25.6	23.0	27.8	14.8
$RSD_R(\%)$	23.7	11.8	11.6	19.6	8.5

Table	3
Analysis	Determination of the content of waxes, fatty acid methyl esters and fatty acid ethyl esters by capillary gas chromatography
Method	COI/T.20/Doc.n°18/Rev.3
Parameter	FAEE (Ethyl C16+C18) - Ring Test COI 2010
Unit	mg/kg
Final result rounded to	no decimal

A: high quality extra virgin   year 2001

B: high quality extra virgin   year 1991

C: extra virgin supermarket   year 2010

D: extra virgin supermarket   year 2010

E: extra virgin supermarket   year 2010

	A	B	C	D	E
n	15	17	17	17	17
outliers	1	2	1	2	2
mean	5	137	276	96	28
r	2.14	5.36	7.6	6.66	2.66
S_r	0.76	1.91	2.71	2.38	0.95
$RSD_r(\%)$	14.8	1.4	1.0	2.5	3.4
R	6.71	38.82	95.91	29.23	15.50
S_R	2.40	13.86	34.25	10.44	5.54
$RSD_R(\%)$	46.5	10.1	12.4	10.9	19.7

Table	4
Analysis	Determination of the content of waxes, fatty acid methyl esters and fatty acid ethyl esters by capillary gas chromatography
Method	COI/T.20/Doc.n°18/Rev.3
Parameter	FAME (Methyl C16+C18) - Ring Test COI 2010
Unit	mg/kg
Final result rounded to	no decimal

A: high quality extra virgin   year 2001   D: extra virgin supermarket   year 2010
B: high quality extra virgin   year 1991   E: extra virgin supermarket   year 2010
C: extra virgin supermarket   year 2010

	A	B	C	D	E
n	15	17	17	17	17
outliers	2	2	1	1	3
mean	33	69	74	44	16
r	5.67	10.1	5.09	7.69	2.71
S_r	2.02	3.61	1.82	2.75	0.97
RSD_r(%)	6.1	5.2	2.5	6.2	6.1
R	13.38	26.85	29.48	18.44	10.52
S_R	4.78	9.59	10.53	6.58	3.76
RSD_R(%)	14.3	13.8	14.2	14.9	23.6

Table	5
Analysis	Determination of the content of waxes, fatty acid methyl esters and fatty acid ethyl esters by capillary gas chromatography
Method	COI/T.20/Doc.n°18/Rev.3
Parameter	FAAE (SUM Methyl + Ethyl) - Ring Test COI 2010
Unit	mg/kg
Final result rounded to	no decimal

A: high quality extra virgin   year 2001   D: extra virgin supermarket   year 2010
B: high quality extra virgin   year 1991   E: extra virgin supermarket   year 2010
C: extra virgin supermarket   year 2010

	A	B	C	D	E
n	15	17	17	17	17
outliers	2	1	2	2	2
mean	38	212	350	139	43
r	6.80	16.83	6.29	7.21	4.09
S_r	2.43	6.01	2.25	2.58	1.46
RSD_r(%)	6.3	2.8	0.6	1.9	3.4
R	17.91	77.26	112.95	38.47	14.12
S_R	6.39	27.59	40.34	13.74	5.04
RSD_R(%)	16.6	13.0	11.5	9.9	11.7

Table	6
Analysis	Determination of the content of waxes, fatty acid methyl esters and fatty acid ethyl esters by capillary gas chromatography
Method	COI/T.20/Doc.n°18/Rev.3
Parameter	RATIO (FAEE/FAME) - Ring Test COI 2010
Unit	-
Final result rounded to	1 decimal place

A: high quality extra virgin year 2001   D: extra virgin supermarket year 2010
B: high quality extra virgin year 1991   E: extra virgin supermarket year 2010
C: extra virgin supermarket   year 2010

	A	B	C	D	E
n	15	17	17	17	17
outliers	0	1	1	1	1
mean	0.2	2.0	3.8	2.2	1.8
r	0.08	0.21	0.30	0.35	0.42
S_r	0.03	0.08	0.11	0.13	0.15
RSD$_r$(%)	18.2	3.8	2.8	5.7	8.5
R	0.23	0.57	1.56	0.68	1.38
S_R	0.08	0.20	0.56	0.24	0.49
RSD$_R$(%)	51.5	10.1	14.7	11.0	28.2

Table	7
Analysis	Determination of the difference between actual and theoretical content of triacylglycerols with ECN42
Method	COI/T.20/Doc.n°20/Rev.4
Parameter	ΔECN42 determined with acetone and acetonitrile - Ring Test COI 1999
Unit	%
Final result rounded to	2 decimal place

A: extra virgin olive oil
B: virgin olive oil + refined sunflower oil
C: virgin olive oil + refined olive-pomace oil
D: virgin olive oil + refined soybean oil + refined sunflower oil
E: refined olive oil + refined olive-pomace oil + refined soybean oil + lampante virgin olive oil

	A	B	C	D	E
n	19	19	19	19	19
outliers	1	0	0	0	3
mean	0.04	1.66	0.04	0.18	0.82
r	0.08	0.12	0.09	0.11	0.11
S_r	0.02	0.04	0.03	0.04	0.04
RSD$_r$(%)	82.2(not sig.)	2.8	76.1(not sig.)	22.5	5.1
R	0.12	0.25	0.16	0.22	0.24
S_R	0.05	0.09	0.05	0.08	0.08
RSD$_R$(%)	127.6(not sig.)	5.4	132.2(not sig.)	46.2	10.9

Table	8
Analysis	Determination of the difference between actual and theoretical content of triacylglycerols with ECN42
Method	COI/T.20/Doc.n°20/Rev.4
Parameter	ΔECN42 determined with proprionitrile
Unit	%
Final result rounded to	2 decimal place

A: 70% virgin olive oil + 10% refined olive-pomace oil + 20% high oleic sunflower oil
B: 80% high campesterol virgin olive oil + 20% palm olein
C: 100% virgin olive oil
D: 70% virgin olive oil + 15% refined olive-pomace oil + 15% high oleic sunflower oil

	A	B	C	D
n	16	16	11	11
outliers	0	2	0	0
mean	1.07	0.10	0.06	0.84
r	0.05	0.02	0.06	0.06
S_r	0.02	0.01	0.02	0.02
RSD_r(%)	1.6	7.9	36.6	2.7
R	0.33	0.11	0.12	0.35
S_R	0.12	0.04	0.04	0.12
RSD_R(%)	11.2	36.8	78.6 (not sig.)	14.8

Table	9
Analysis	Determination of stigmastadienes in vegetable oils
Method	COI/T.20/Doc.n°11/Rev.3
Parameter	Stigmastadienes - Ring Test COI 1999
Unit	mg/kg
Final result rounded to	2 decimal place

A: extra virgin olive oil
B: virgin olive oil + refined sunflower oil
C: virgin olive oil + refined olive-pomace oil
D: virgin olive oil + refined soybean oil + refined sunflower oil
E: refined olive oil + refined olive-pomace oil + refined soybean oil + lampante virgin olive oil

	A	B	C	D	E
n	19	19	19	19	19
outliers	3	5	7	2	5
mean	0.01	0.80	9.49	0.22	7.55
r	0.01	0.08	0.39	0.05	0.48
S_r	0.00	0.03	0.14	0.01	0.17
RSD_r(%)	32.4 (not sig.)	3.7	1.5	8.4	2.3
R	0.03	0.15	1.66	0.06	1.59
S_R	0.01	0.05	0.59	0.03	0.57
RSD_R(%)	98.6 (not sig.)	6.7	6.3	11.5	7.6

Table	10
Analysis	Determination of sterenes in refined vegetable oils
Method	COI/T.20/Doc.n°16/Rev.2
Parameter	Sterenes - Results from recognition 2000-2006
Unit	mg/kg
Final result rounded to	1 decimal place

	A	B	C	D	E
n	31	31	31	31	31
outliers	4	4	5	4	4
mean	9.5	31.0	46.0	9.0	11.4
r	0.2	1.0	1.0	0.3	0.5
S_r	0.1	0.3	0.4	0.1	0.2
$RSD_r(\%)$	0.8	1.0	0.9	1.1	1.6
R	2.0	5.0	12.0	1.0	1.0
S_R	0.6	1.7	4.2	0.5	0.5
$RSD_R(\%)$	6.1	5.3	9.1	5.9	4.4

Table	11
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Total sterols - Ring Test COI 2016-1 – Separation by TLC
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	1	0	0	1	1
mean	1572	1742	1679	2830	3181
r	84.9	134.8	144.7	246.2	307.3
S_r	30.3	48.1	51.7	87.9	109.7
$RSD_r(\%)$	1.9	2.8	3.1	3.1	3.5
R	291.3	495.9	321.6	346.4	610.4
S_R	104.0	177.1	114.8	123.7	218.0
$RSD_R(\%)$	6.6	10.2	6.8	4.4	6.9

Table	12
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Total sterols - Ring Test COI 2016-1 – Separation by HPLC
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	1	1	1	1	0
mean	1583	1754	1730	2897	3216
r	74.0	93.5	95.0	59.01	181.9
S_r	264.4	33.4	33.9	21.1	65.0
RSD_r(%)	1.7	1.9	2.0	0.7	2.0
R	315.0	190.2	156.6	230.2	480.2
S_R	112.5	67.9	55.9	82.2	171.5
RSD_R(%)	7.1	3.9	3.2	2.8	5.3

Table	13
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Cholesterol - Ring Test COI 2016-1 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	1	0	0
mean	0.1	0.3	0.1	0.1	0.1
r	0.03	0.10	0.05	0.07	0.03
S_r	0.01	0.04	0.02	0.03	0.01
RSD_r(%)	8.3	13.6	14.2	20.2	8.6
R	0.06	0.28	0.09	0.14	0.07
S_R	0.02	0.10	0.03	0.05	0.02
RSD_R(%)	15.9	37.5	22.5	40.3	23.4

Table	14
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Cholesterol - Ring Test COI 2016-1 – Separation by HPLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	0	0	0
mean	0.1	0.3	0.1	0.1	0.1
r	0.03	0.10	0.07	0.03	0.04
S_r	0.01	0.04	0.02	0.01	0.01
$RSD_r(\%)$	7.6	15.0	17.7	9.3	10.8
R	0.06	0.24	0.09	0.11	0.06
S_R	0.02	0.08	0.03	0.04	0.02
$RSD_R(\%)$	16.5	34.3	23.2	35.4	16.7

Table	15
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Brassicasterol - Ring Test COI 2016-1 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	1	0	0	1	1
mean	0.0	0.0	0.0	0.0	0.1
r	0.02	0.03	0.03	0.02	0.02
S_r	0.01	0.01	0.01	0.01	0.01
$RSD_r(\%)$	68.1	23.6	39.3	25.3	14.7
R	0.03	0.11	0.09	0.07	0.15
S_R	0.01	0.04	0.03	0.03	0.05
$RSD_R(\%)$	103.7	90.5	105.3	94.5	90.7

Table	16
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Brassicasterol - Ring Test COI 2016-1 – Separation by HPLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	0	0	0
mean	0.1	0.3	0.1	0.1	0.1
r	0.03	0.10	0.07	0.03	0.04
S_r	0.01	0.04	0.02	0.01	0.01
$RSD_r(\%)$	7.6	15.0	17.7	9.3	10.8
R	0.06	0.24	0.09	0.11	0.06
S_R	0.02	0.08	0.03	0.04	0.02
$RSD_R(\%)$	16.5	34.3	23.2	35.4	16.7

Table	17
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Campesterol - Ring Test COI 2016-1 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	2	0	0	0	1
mean	3.1	3.2	3.9	8.3	3.1
r	0.22	0.15	0.26	0.18	0.15
S_r	0.08	0.06	0.09	0.06	0.05
$RSD_r(\%)$	2.6	1.7	2.4	0.8	1.7
R	0.25	0.39	0.45	0.78	0.27
S_R	0.09	0.13	0.16	0.28	0.10
$RSD_R(\%)$	2.9	4.3	4.1	3.4	3.1

Table	18
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Campesterol - Ring Test COI 2016-1 – Separation by HPLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	1	0	0	0
mean	3.0	3.3	3.9	8.4	3.2
r	0.12	0.13	0.18	0.22	0.11
S_r	0.04	0.05	0.06	0.09	0.04
RSD_r(%)	1.5	1.4	1.6	0.9	1.3
R	0.48	0.59	0.37	0.52	0.28
S_R	0.17	0.21	0.13	0.18	0.10
RSD_R(%)	5.7	6.5	3.4	2.2	3.2

Table	19
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Stigmasterol - Ring Test COI 2016-1 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	0	1	0
mean	1.1	2.4	2.0	7.2	1.3
r	0.07	0.16	0.25	0.12	0.09
S_r	0.02	0.06	0.09	0.04	0.03
RSD_r(%)	2.1	2.4	4.5	0.6	2.5
R	0.18	0.29	0.41	0.62	0.11
S_R	0.06	0.10	0.15	0.22	0.04
RSD_R(%)	5.9	4.3	7.4	3.1	3.0

Table	20
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Stigmasterol - Ring Test COI 2016-1 – Separation by HPLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	0	1	0
mean	1.1	2.4	2.0	7.2	1.3
r	0.07	0.14	0.08	0.21	0.16
S_r	0.03	0.05	0.03	0.08	0.06
$RSD_r(\%)$	2.3	2.1	1.5	1.1	4.4
R	0.15	0.22	0.11	0.45	0.19
S_R	0.06	0.08	0.04	0.16	0.07
$RSD_R(\%)$	5.1	3.3	2.0	2.2	5.3

Table	21
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Δ7 Stigmastenol - Ring Test COI 2016-1 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	0	0	1
mean	0.3	0.4	3.2	16.0	0.5
r	0.06	0.08	0.53	1.08	0.06
S_r	0.02	0.03	0.19	0.39	0.02
$RSD_r(\%)$	7.5	6.4	5.9	2.4	4.4
R	0.15	0.19	0.83	1.52	0.19
S_R	0.05	0.07	0.30	0.54	0.07
$RSD_R(\%)$	18.7	16.0	9.4	3.4	13.5

Table	22
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	$\Delta7$ Stigmastenol - Ring Test COI 2016-1 – Separation by HPLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	1	0	0	0	0
mean	0.32	0.46	3.22	16.09	0.52
r	0.10	0.12	0.38	0.75	0.08
S_r	0.037	0.041	0.13	0.267	0.029
$RSD_r(\%)$	11.4	9.0	4.2	1.7	5.6
R	0.13	0.24	0.75	1.95	0.16
S_R	0.045	0.087	0.269	0.696	0.058
$RSD_R(\%)$	14.2	18.8	8.3	4.3	11.0

Table	23
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Apparent β-sitosterol - Ring Test COI 2016-1 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	1	1	1
mean	94.4	92.6	89.0	61.1	94.0
r	0.45	0.37	1.43	1.43	0.33
S_r	0.16	0.13	0.51	0.51	0.12
$RSD_r(\%)$	0.17	0.14	0.57	0.84	0.13
R	0.76	1.31	1.79	4.00	0.63
S_R	0.27	0.47	0.63	1.43	0.23
$RSD_R(\%)$	0.29	0.51	0.72	2.34	0.24

Table	24
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Apparent β-sitosterol - Ring Test COI 2016-1– Separation by HPLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	15	15	15	15	14
outliers	0	0	1	1	1
mean	94.4	92.5	88.7	60.7	94.1
r	0.38	0.45	1.15	1.08	0.50
S_r	0.13	0.16	0.41	0.39	0.18
$RSD_r(\%)$	0.14	0.17	0.46	0.63	0.19
R	0.81	1.11	1.41	4.04	0.99
S_R	0.29	0.40	0.51	1.44	0.35
$RSD_R(\%)$	0.31	0.43	0.57	2.38	0.38

Table	25
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Erythrodiol + uvaol (% total sterols) - Ring Test COI 2016-1 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	1	0	1	0	0
mean	2.1	3.8	1.2	2.5	17.2
r	0.32	0.34	0.19	0.27	0.76
S_r	0.12	0.12	0.07	0.10	0.27
$RSD_r(\%)$	5.4	3.2	5.4	3.9	1.6
R	0.80	0.85	0.53	1.09	4.68
S_R	0.29	0.30	0.19	0.39	1.67
$RSD_R(\%)$	13.3	8.0	15.3	15.5	9.7

Table	26
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Erythrodiol + uvaol (% total sterols) - Ring Test COI 2016-1 – Separation by HPLC
Unit	%
Final result rounded to	1 decimal place

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	0	0	0
mean	2.2	3.8	1.4	2.0	17.2
r	0.40	0.32	0.24	0.16	0.73
S_r	0.14	0.11	0.09	0.06	0.26
RSD_r(%)	6.5	3.0	6.1	2.8	1.5
R	0.52	0.57	0.46	0.62	3.66
S_R	0.19	0.20	0.17	0.22	1.31
RSD_R(%)	8.4	5.3	11.8	10.9	7.6

Table	27
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Erythrodiol absolute - Ring Test COI 2016-1 – Separation by TLC
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	1	1	1	1	1
mean	31	61	17	52	598
r	2.8	4.4	1.5	4.0	71.0
S_r	1.0	1.6	0.5	1.4	25.3
RSD_r(%)	3.3	2.6	3.1	2.7	4.2
R	6.0	21.0	10.2	9.4	148.3
S_R	2.1	2.6	3.6	3.3	53.0
RSD_R(%)	7.0	12.4	20.8	6.5	8.9

Table	28
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Erythrodiol absolute - Ring Test COI 2016-1 – Separation by HPLC
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	1	1	0	0	1
mean	32	60	18	51	605
r	3.0	8.8	2.5	5.6	36.5
S_r	1.1	3.1	0.9	2.0	13.0
$RSD_r(\%)$	3.3	5.2	5.1	4.0	2.2
R	7.3	23.1	5.6	5.8	152.5
S_R	2.6	8.2	2.0	2.0	54.5
$RSD_R(\%)$	8.1	13.7	11.4	4.1	9.0

Table	29
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Uvaol absolute - Ring Test COI 2016-1 – Separation by TLC
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	2	1	1	1	2
mean	3.0	8	4	20	65
r	0.83	1.5	3.3	3.5	12.5
S_r	0.30	0.55	1.2	1.2	4.5
$RSD_r(\%)$	10.1	6.8	27.8	6.2	6.8
R	4.6	9.0	4.1	3.5	23.1
S_R	1.6	3.2	1.5	1.2	8.3
$RSD_R(\%)$	55.6	40.0	34.0	6.2	12.7

Table	30
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Uvaol absolute -
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	1	1	0	1
mean	5	8	5	20.0	65
r	1.6	1.3	0.80	2.8	6.5
S_r	0.55	0.48	0.29	1.0	2.3
$RSD_r(\%)$	10.2	5.7	5.7	5.2	3.6
R	4.2	6.8	3.4	3.5	15.5
S_R	1.5	2.4	1.2	1.3	5.5
$RSD_R(\%)$	27.3	28.6	24.0	6.4	8.5

Table	31
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Total aliphatic alcohols (C22 + C24 + C26 + C28) - Ring Test COI 2016-1– Separation by TLC
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	1	0	0	0
mean	143	420	62	78	1512
r	5.5	23	4.5	5.1	70
S_r	1.9	8.2	1.6	1.8	24.9
$RSD_r(\%)$	1.4	2.0	2.6	2.3	1.7
R	25	67	10	10	95
S_R	8.9	23.8	3.7	3.6	34.7
$RSD_R(\%)$	6.2	5.7	6.1	4.7	2.3

Table	32
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Total aliphatic alcohols (C22 + C24 + C26 + C28) - Ring Test COI 2016-1 – Separation by HPLC
Unit	mg/kg
Final result rounded to	no decimal

A: extra virgin olive oil from picual variety
B: lampante olive oil
C: olive oil + 10% sunflower oil
D: high oleic sunflower + 50 mg/kg erythrodiol + 20 mg/kg uvaol
E: virgin olive oil + refined olive pomace oil

	A	B	C	D	E
n	14	14	14	14	14
outliers	0	0	0	0	1
mean	139	423	62	78	1495
r	6.9	15	6.2	5.9	46
S_r	2.5	5.3	2.2	2.1	16.2
RSD_r(%)	1.8	1.3	3.6	2.7	1.1
R	23	36	9.0	11	86
S_R	8.4	12.9	3.2	3.8	30.8
RSD_R(%)	6.0	3.1	5.2	4.9	2.1

Table	33
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Erythrodiol + uvaol (% total sterols) - Ring Test COI 2016-2 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: Lampante olive oil
B: refined olive oil (from sample 1)
C: Desterolysed high oleic sunflower oil + 3.13% / 49.26 mg/kg of standard erythrodiol
D: Pomace Olive oil (traded)
E: Pomace Olive oil (traded)

	A	B	C	D	E
n	17	17	17	17	17
outliers	1	3	3	2	3
mean	3.3	4.3	3.5	22.8	22.7
r	0.30	0.70	0.20	1.5	1.5
S_r	0.10	0.20	0.10	0.50	0.50
RSD_r(%)	6.5	3.0	6.1	2.8	1.5
R	3.1	5.7	2.3	2.3	2.4
S_R	2.6	1.1	0.70	3.0	3.3
RSD_R(%)	0.9	0.4	0.2	1.1	1.2

Table	34
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Erythrodiol + Uvaol content - Ring Test COI 2016-2 – Separation by TLC
Unit	mg/kg
Final result rounded to	no decimal

A: Lampante olive oil
B: refined olive oil (from sample 1)
C: Desterolysed high oleic sunflower oil + 3.13% / 49.26 mg/kg of standard erythrodiol
D: Pomace Olive oil (traded)
E: Pomace Olive oil (traded)

	A	B	C	D	E
n	16	16	17	16	16
outliers	3	4	3	2	3
mean	59	50	52	772	745
r	6.5	6.7	4.7	40	100
S_r	2.3	2.4	1.7	14.2	35.6
RSD_r(%)	3.9	4.8	3.3	1.8	4.8
R	9.9	14	31	146	183
S_R	3.5	4.8	11.1	52.1	65.4
RSD_R(%)	6.0	9.8	21.5	6.8	8.8

Table	35
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Erythrodiol (% total sterols) - Ring Test COI 2016-2 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: Lampante olive oil
B: refined olive oil (from sample 1)
C: Desterolysed high oleic sunflower oil + 3,13% / 49,26 mg/kg of standard erythrodiol
D: Pomace Olive oil (traded)
E: Pomace Olive oil (traded)

	A	B	C	D	E
n	17	17	16	17	17
outliers	1	3	1	2	3
mean	3.1	3.9	3.4	18.8	18.7
r	0.30	0.60	0.20	1.2	1.5
S_r	0.10	0.20	0.10	0.40	0.50
RSD_r(%)	3.1	5.3	2.4	2.3	2.9
R	0.80	1.0	0.60	2.8	2.8
S_R	0.30	0.40	0.20	1.0	1.0
RSD_R(%)	8.9	9.1	6.6	5.3	5.3

Table	36
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Erythrodiol content - Ring Test COI 2016-2 – Separation by TLC
Unit	mg/kg
Final result rounded to	no decimal

A: Lampante olive oil
B: refined olive oil (from sample 1)
C: Desterolysed high oleic sunflower oil + 3.13% / 49.26 mg/kg of standard erythrodiol
D: Pomace Olive oil (traded)
E: Pomace Olive oil (traded)

	A	B	C	D	E
n	16	16	16	16	16
outliers	1	3	2	0	2
mean	53	46	48	638	635
r	5.1	13	4.1	41	78
S_r	1.8	4.6	1.5	14.5	27.7
$RSD_r(\%)$	3.4	9.9	3.1	2.3	4.4
R	13	16	12	125	130
S_R	4.7	5.7	4.1	44.6	46.4
$RSD_R(\%)$	8.8	12.2	8.6	7.0	7.3

Table	37
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Uvaol (% total sterols) - Ring Test COI 2016-2 – Separation by TLC
Unit	%
Final result rounded to	1 decimal place

A: Lampante olive oil
B: refined olive oil (from sample 1)
C: Desterolysed high oleic sunflower oil + 3,13% / 49,26 mg/kg of standard erythrodiol
D: Pomace Olive oil (traded)
E: Pomace Olive oil (traded)

	A	B	C	D	E
n	16	16	16	16	16
outliers	1	1	3	1	0
mean	0.4	0.4	0.1	4.0	4.1
r	0.10	0.30	0.10	0.40	0.30
S_r	0.00	0.10	0.00	0.10	0.10
$RSD_r(\%)$	11.4	23.3	35.8	3.4	2.4
R	0.70	0.20	0.10	0.60	0.70
S_R	0.20	0.10	0.00	0.20	0.30
$RSD_R(\%)$	55.4	22.5	86.9	5.5	6.2

Table	38
Analysis	Determination of the sterol content and alcoholic compounds
Method	COI/T.20/Doc.n°26/Rev.4
Parameter	Uvaol content - Ring Test COI 2016-2 – Separation by TLC
Unit	mg/kg
Final result rounded to	no decimal

A: Lampante olive oil
B: refined olive oil (from sample 1)
C: Desterolysed high oleic sunflower oil + 3,13% / 49,26 mg/kg of standard erythrodiol
D: Pomace Olive oil (traded)
E: Pomace Olive oil (traded)

	A	B	C	D	E
n	15	14	15	14	13
outliers	2	1	3	0	1
mean	8	5	1	136	138
r	2.5	2.6	1.0	12	22
S_r	0.90	0.90	0.30	4.4	8.0
RSD_r(%)	11.6	20.0	43.3	3.2	5.8
R	11	3.3	2.2	31	36
S_R	3.9	1.2	0.80	11.0	12.8
RSD_R(%)	51.6	25.8	99.0	8.1	9.2

Table	39
Analysis	Spectrophotometric analysis in the ultraviolet
Method	COI/T.20/Doc.n°19/Rev.5
Parameter	K270 using cyclohexane - Ring Test COI 2009
Unit	-
Final result rounded to	2 decimal place

	A	B	C	D	E
n	21	21	21	21	21
outliers	1	2	1	1	4
mean	0.13	0.43	1.12	0.45	0.59
r	0.014	0.023	0.029	0.033	0.018
S_r	0.005	0.008	0.010	0.012	0.006
RSD_r(%)	4.0	1.9	0.9	2.6	1.1
R	0.031	0.044	0.074	0.04	0.042
S_R	0.011	0.016	0.027	0.014	0.015
RSD_R(%)	8.5	3.7	2.4	3.2	2.5

Table	40
Analysis	Spectrophotometric analysis in the ultraviolet
Method	COI/T.20/Doc.n°19/Rev.5
Parameter	K268 using isooctane - Ring Test COI 2009
Unit	-
Final result rounded to	2 decimal place

	A	B	C	D	E
n	21	22	22	22	22
outliers	1	4	2	5	2
mean	0.12	0.43	1.14	0.45	0.60
r	0.014	0.014	0.043	0.018	0.018
S_r	0.005	0.005	0.016	0.007	0.007
$RSD_r(\%)$	4.0	1.2	1.4	1.5	1.1
R	0.028	0.045	0.083	0.038	0.094
S_R	0.010	0.016	0.030	0.013	0.034
$RSD_R(\%)$	8.0	3.8	2.6	3.0	5.6

Table	41
Analysis	Spectrophotometric analysis in the ultraviolet
Method	COI/T.20/Doc.n°19/Rev.5
Parameter	K232 using cyclohexane - Ring Test COI 2009
Unit	-
Final result rounded to	2 decimal place

	A	B	C	D	E
n	21	21	21	21	21
outliers	3	1	1	0	0
mean	1.76	2.12	3.83	3.86	2.79
r	0.070	0.060	0.119	0.113	0.093
S_r	0.025	0.0216	0.0423	0.0405	0.0332
$RSD_r(\%)$	1.4	1.0	1.1	1.1	1.2
R	0.138	0.204	0.424	0.386	0.279
S_R	0.049	0.073	0.151	0.138	0.100
$RSD_R(\%)$	2.8	3.4	4.0	3.6	3.6

Table	42
Analysis	Spectrophotometric analysis in the ultraviolet
Method	COI/T.20/Doc.n°19/Rev.5
Parameter	K232 using isooctane - Ring Test COI 2009
Unit	-
Final result rounded to	2 decimal place

	A	B	C	D	E
n	21	22	22	22	22
outliers	1	4	4	1	5
mean	1.76	2.10	3.81	3.85	2.82
r	0.072	0.035	0.043	0.101	0.054
S_r	0.026	0.013	0.016	0.036	0.019
RSD_r(%)	1.5	0.6	0.4	0.9	0.7
R	0.216	0.194	0.488	0.582	0.194
S_R	0.077	0.069	0.174	0.211	0.069
RSD_R(%)	4.4	3.3	4.6	5.5	2.5

Table	43
Analysis	Spectrophotometric analysis in the ultraviolet
Method	COI/T.20/Doc.n°19/Rev.5
Parameter	ΔK using cyclohexane - Ring Test COI 2009
Unit	-
Final result rounded to	2 decimal place

	A	B	C	D	E
n	20	21	21	21	21
outliers	1	1	2	1	3
Mean	-0.00	0.00	0.09	0.04	0.05
r	0.002	0.002	0.003	0.003	0.004
S_r	0.001	0.001	0.001	0.001	0.001
RSD_r(%)	28.9	21.6	1.1	2.9	2.9
R	0.008	0.004	0.012	0.007	0.011
S_R	0.003	0.001	0.004	0.003	0.004
RSD_R(%)	147.5	52.0	5.1	7.6	8.1

Table	44
Analysis	Spectrophotometric analysis in the ultraviolet
Method	COI/T.20/Doc.n°19/Rev.5
Parameter	ΔK using isooctane - Ring Test COI 2009
Unit	-
Final result rounded to	2 decimal place

	A	B	C	D	E
n	21	21	22	22	22
outliers	0	3	1	2	2
mean	-0.00	0.00	0.08	0.03	0.04
r	0.003	0.001	0.005	0.004	0.002
S_r	0.001	0.001	0.002	0.001	0.001
$RSD_r(\%)$	36.4	121.1	2.3	4.4	1.7
R	0.011	0.003	0.023	0.011	0.013
S_R	0.004	0.001	0.008	0.004	0.005
$RSD_R(\%)$	148.2	234.8	10.0	12.6	10.6

Table	45
Analysis	Determination of the percentage of 2-glyceryl monopalmitate
Method	COI/T.20/Doc.n°23/Rev.1
Parameter	2-glyceryl monopalmitate
Unit	%
Final result rounded to	1 decimal place

A: Extra virgin olive oil
B: Lampante virgin olive oil
C: Refined olive oil
D: Refined olive oil + re-esterified oil (90:10)
E: Refined olive oil + re-esterified oil (80:20)

	A	B	C	D	E
n	12	12	12	12	12
outliers	0	0	0	0	0
mean	0.5	0.8	0.9	1.8	2.8
r	0.11	0.11	0.17	0.10	0.26
S_r	0.04	0.04	0.06	0.04	0.09
$RSD_r(\%)$	8.9	5.4	6.8	2.0	3.3
R	0.14	0.27	0.26	0.56	0.86
S_R	0.05	0.10	0.09	0.20	0.31
$RSD_R(\%)$	11.1	12.7	10.2	11.1	10.9

Table	46
Analysis	Determination of biophenols in olive oils by HPLC
Method	COI/T.20/Doc.n°29/Rev.1
Parameter	Biophenols - Ring Test COI 2008
Unit	mg/kg
Final result rounded to	no decimal

A: Extra virgin olive oil (Italy)
B : Extra virgin olive oil (Spain)
C : Extra virgin olive oil (Tunisia)
D : Extra virgin olive oil (Slovenia)
E : Extra virgin olive oil (Greece)
R : Extra virgin olive oil (Italy)

	A	B	C	D	E	R
n	17	17	17	17	17	17
outliers	3	3	1	2	2	2
mean	694	573	153	343	297	301
r	29	36	18	24	22	17
S_r	10.4	12.7	6.4	8.7	7.7	6.2
$RSD_r(\%)$	1.5	2.2	4.2	2.5	2.6	2.1
R	100.8	83.7	59.6	62.7	77.0	32.2
S_R	36.0	29.9	21.3	22.4	27.5	11.5
$RSD_R(\%)$	5.2	5.2	14.0	6.5	9.3	3.8

Table	47
Analysis	Determination of free acidity, cold method
Method	COI/T.20/Doc.n°34/Rev.1
Parameter	Acidity - Ring Test COI 2014-2015
Unit	% of oleic acid
Final result rounded to	2 decimal place if ≤ 1 ; 1 decimal place if > 1

A: Crude olive pomace oil *E : Extra virgin olive oil (Mario Solinas 2014)*
B : Refined olive pomace oil *F : : 70% Lampante olive oil + 30% Grape Seed oil*
C : Refined olive oil *G : Extra virgin olive oil from late harvest*
D : Extra virgin olive oil (Mario Solinas 2011) *H : 90% Lampante olive oil + 10% palm olein*

	A	B	C	D	E	F	G	H
n	22	22	22	22	22	20	20	20
outliers	1	1	2	2	0	2	3	2
mean	6.3	0.11	0.07	0.13	0.15	1.4	0.50	0.69
r	0.144	0.019	0.018	0.011	0.021	0.015	0.018	0.022
S_r	0.052	0.007	0.006	0.004	0.007	0.005	0.006	0.008
$RSD_r(\%)$	0.8	6.1	9.3	3.2	4.8	0.4	1.3	1.1
R	0.535	0.074	0.043	0.053	0.100	0.121	0.074	0.085
S_R	0.191	0.027	0.015	0.019	0.036	0.043	0.026	0.030
$RSD_R(\%)$	3.0	24.2	22.7	14.7	23.3	3.1	5.3	4.4

Table	48
Analysis	Determination of the peroxide value
Method	COI/T.20/Doc.n°35/Rev.1
Parameter	Peroxide value - Ring Test COI 2016
Unit	$meqO_2/kg$
Final result rounded to	1 decimal place if ≤ 20 ; no decimal if > 20

A: 70% Lampante Olive Oil + 30% Grape Seed Oil

B : 90% Lampante olive oil + 10% palm olein

C : Extra virgin olive oil

D : Olive oil

E : 50% Extra virgin olive oil + 10% refined sunflower oil

F : Extra virgin olive oil (ripe fruitiness)

G : Extra virgin olive oil

	A	B	C	D	E	F	G
n	16	20	15	14	14	19	15
outliers	0	0	0	0	0	0	0
mean	11.7	24	7.8	2.8	4.9	14.3	8.2
r	0.26	1.31	0.41	0.27	0.19	0.52	0.33
S_r	0.09	0.47	0.15	0.1	0.07	0.18	0.12
$RSD_r(\%)$	0.8	1.9	1.9	3.4	1.4	1.3	1.4
R	1.86	4.00	1.55	1.09	1.19	3.18	2.81
S_R	0.66	1.43	0.55	0.39	0.43	1.14	1.0
$RSD_R(\%)$	5.7	5.9	7.1	13.8	8.6	8.0	12.3

Table	49
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Myristic acid C14:0 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil

B: Virgin olive oil

C: Lampante olive oil

D: Olive oil

E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	0	1	1	3
mean	0.01	0.01	0.01	0.01	0.02
r	0.005	0.007	0.012	0.011	0.006
S_r	0.002	0.003	0.004	0.004	0.002
$RSD_r(\%)$	20	20	36	38	11
R	0.011	0.017	0.017	0.013	0.016
S_R	0.004	0.006	0.006	0.005	0.006
$RSD_R(\%)$	45	47	52	42	32

Table	50
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Palmitic acid C16:0 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	2	3	1	0	0
mean	7.96	10.32	10.35	10.51	9.67
r	0.12	0.18	0.42	0.29	0.38
S_r	0.04	0.06	0.15	0.1	0.14
RSD_r(%)	0.5	0.6	1.5	1.000	1.4
R	0.68	0.44	0.93	1.3	1.3
S_R	0.24	0.16	0.33	0.46	0.45
RSD_R(%)	3.0	1.5	3.2	4.4	4.7

Table	51
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Palmitoleic acid C16:1- Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	2	0	1	1
mean	0.50	0.68	0.74	0.91	0.64
r	0.041	0.027	0.074	0.034	0.040
S_r	0.014	0.01	0.026	0.012	0.014
RSD_r(%)	2.9	1.4	3.6	1.3	2.3
R	0.966	0.077	0.132	0.123	0.128
S_R	0.034	0.027	0.047	0.44	0.046
RSD_R(%)	6.8	4.1	6.4	4.9	7.2

Table	52
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Heptadecanoic acid C17:0 - from 2000-2006 recognition data
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	25	25	25	25	25
outliers	1	1	1	2	2
mean	0.18	0.06	0.11	0.14	0.12
r	0.013	0.011	0.010	0.009	0.009
S_r	0.005	0.004	0.004	0.003	0.003
RSD_r(%)	2.7	6.9	3.1	2.3	2.7
R	0.020	0.021	0.024	0.021	0.027
S_R	0.007	0.007	0.009	0.008	0.010
RSD_R(%)	4.1	12.6	7.7	5.2	7.8

Table	53
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Heptadecenoic acid C17:1 - from 2000-2006 recognition data
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	29	29	29	29	29
outliers	3	2	2	3	2
mean	0.26	0.09	0.24	0.22	0.19
r	0.010	0.010	0.014	0.013	0.012
S_r	0.004	0.004	0.005	0.005	0.004
RSD_r(%)	1.4	3.8	2.0	2.2	2.2
R	0.031	0.027	0.041	0.030	0.031
S_R	0.011	0.010	0.015	0.011	0.011
RSD_R(%)	4.2	10.6	6.1	4.9	5.8

Table	54
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Stearic acid C18:0 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	2	0	0	0	1
mean	2.88	2.49	2.62	3.49	3.12
r	0.089	0.034	0.084	0.094	0.107
S_r	0.032	0.012	0.030	0.034	0.038
$RSD_r(\%)$	1.1	0.5	1.1	1.0	1.2
R	0.171	0.259	0.246	0.367	0.328
S_R	0.061	0.092	0.088	0.131	0.117
$RSD_R(\%)$	2.1	3.7	3.4	3.8	3.8

Table	55
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Oleic acid C18:1 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	0	1	1	0
mean	79.42	74.55	75.55	76.14	75.8
r	0.42	0.30	0.39	0.23	0.46
S_r	0.15	0.11	0.14	0.08	0.16
$RSD_r(\%)$	0.2	0.2	0.2	0.1	0.2
R	1.37	1.26	1.26	1.33	1.80
S_R	0.49	0.45	0.45	0.47	0.64
$RSD_R(\%)$	0.6	0.6	0.6	0.6	0.9

Table	56
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Linoleic acid C18:2 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
N	15	15	15	15	15
Outliers	2	1	0	1	0
Mean	7.33	9.66	8.52	7.18	8.75
r	0.07	0.08	0.17	0.12	0.13
S_r	0.02	0.03	0.06	0.04	0.05
$RSD_r(\%)$	0.3	0.3	0.7	0.6	0.6
R	0.34	0.52	0.50	0.45	0.59
S_R	0.12	0.19	0.18	0.16	0.21
$RSD_R(\%)$	1.7	1.9	2.1	2.2	2.4

Table	57
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Linolenic acid C18:3 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	2	0	0	0	4
mean	0.73	0.90	0.86	0.74	0.75
r	0.036	0.049	0.029	0.039	0.055
S_r	0.013	0.017	0.010	0.014	0.020
$RSD_r(\%)$	1.8	1.9	1.2	1.9	2.6
R	0.08	0.1	0.101	0.079	0.115
S_R	0.029	0.041	0.036	0.028	0.041
$RSD_R(\%)$	3.9	4.6	4.2	3.8	5.4

Table	58
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Arachidic acid C20:0 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	1	0	0	1	0
mean	0.39	0.44	0.44	0.42	0.43
r	0.041	0.050	0.037	0.037	0.053
S_r	0.015	0.018	0.013	0.013	0.019
RSD_r(%)	3.8	4.0	3.0	3.1	4.4
R	0.080	0.089	0.086	0.117	0.102
S_R	0.029	0.032	0.031	0.042	0.036
RSD_R(%)	7.3	7.2	7.0	9.8	8.6

Table	59
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Eicosenoic acid C20:1 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	1	1	1	0	1
mean	0.37	0.39	0.37	0.28	0.30
r	0.026	0.032	0.036	0.047	0.073
S_r	0.009	0.011	0.013	0.017	0.026
RSD_r(%)	7.8	3.0	3.5	6.0	8.9
R	0.082	0.095	0.064	0.079	0.077
S_R	0.029	0.034	0.023	0.028	0.027
RSD_R(%)	7.9	8.7	6.2	10.0	9.3

Table	60
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Behenic acid C22:0 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	1	1	1	3
mean	0.11	0.14	0.14	0.12	0.19
r	0.022	0.036	0.039	0.045	0.036
S_r	0.008	0.013	0.014	0.016	0.013
RSD_r(%)	7.0	9.6	10.0	14.0	6.9
R	0.038	0.044	0.050	0.056	0.043
S_R	0.014	0.016	0.018	0.020	0.015
RSD_R(%)	12.0	12.0	13.0	17.0	8.3

Table	61
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	Lignoceric acid C24:0 - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	1	0	0	0	3
mean	0.04	0.06	0.06	0.05	0.08
r	0.017	0.015	0.033	0.033	0.040
S_r	0.006	0.005	0.012	0.012	0.014
RSD_r(%)	15.0	8.9	20.0	24.0	19.0
R	0.055	0.073	0.072	0.054	0.04
S_R	0.020	0.026	0.026	0.019	0.014
RSD_R(%)	49.0	42.0	45.0	39.0	19.0

Table	62
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	C18:1 trans - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	1	1	1	1	2
mean	0.01	0.01	0.01	0.01	0.12
r	0.011	0.013	0.008	0.013	0.044
S_r	0.004	0.005	0.003	0.005	0.016
$RSD_r(\%)$	38.0	46.0	27.0	45.0	13.2
R	0.027	0.028	0.030	0.032	0.157
S_R	0.010	0.010	0.011	0.011	0.056
$RSD_R(\%)$	96.0	86.0	100.0	89.0	48.0

Table	63
Analysis	Determination of fatty acid methyl esters by gas chromatography
Method	COI/T.20/Doc. n° 33/ Rev.1
Parameter	C18:2 trans + C18:3 trans - Ring Test COI 2015
Unit	%
Final result rounded to	2 decimal place

A: Extra virgin olive oil
B: Virgin olive oil
C: Lampante olive oil
D: Olive oil
E: Crude olive pomace oil

	A	B	C	D	E
n	15	15	15	15	15
outliers	3	3	4	2	3
mean	0.01	0.01	0.01	0.01	0.03
r	0.013	0.014	0.006	0.029	0.017
S_r	0.005	0.005	0.002	0.010	0.006
$RSD_r(\%)$	84.0	50.0	28.0	115.0	24.0
R	0.019	0.022	0.018	0.032	0.059
S_R	0.007	0.008	0.006	0.012	0.021
$RSD_R(\%)$	123.0	79.0	81.0	130.0	83.0

3. **References**

ISO 5725 – Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions.

ISO 5725 - Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

ISO 5725 – Accuracy (trueness and precision) of measurement methods and results – Part 5: Alternative methods for the determination of the precision of a standard measurement method.

ISO 5725 – Accuracy (trueness and precision) of measurement methods and results – Part 6: Use in practice of accuracy values.

AOAC - Statistical Manual of the Association of Official Analytical Chemists. W.J. Youden. E.H. Steiner

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