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COMMISSION REGULATION (EEC) No 183/93

of 29 January 1993

amending Regulation (EEC) No 2568/91 on the characteristics of olive oil and olive-residue oil and on the relevant methods of analysis

(OJ L 22, 30.1.1993, p. 58)

Corrected by:

►**C1** Corrigendum, OJ L 176, 20.7.1993, p. 26 (183/93)

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COMMISSION REGULATION (EEC) No 183/93

of 29 January 1993

amending Regulation (EEC) No 2568/91 on the characteristics of olive oil and olive-residue oil and on the relevant methods of analysis

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation 136/66/EEC of 22 September 1966 on the establishment of a common organization of the market in oils and fats ([1]), as last amended by Regulation (EEC) No 2046/92 ([2]), and in particular Article 35a thereof,

Whereas Commission Regulation (EEC) No 2568/91 ([3]), as last amended by Regulation (EEC) No 3288/92 ([4]), defines the characteristics of the various types of olive oil and olive-residue oil and the relevant methods of analysis; whereas Regulation (EEC) No 2568/91 also amends the additional notes 2, 3 and 4 to Chapter 15 of the combined nomenclature set out in Annex I to Council Regulation (EEC) No 2658/87 of 23 July 1987 on the tariff and statistical nomenclature and on the Common Customs Tariff ([5]), as last amended by Commission Regulation (EEC) No 2505/92 ([6]);

Whereas, in view of experience gained, the methods of analysis need to be adapted or set out with greater precision in some cases; whereas, it has also become apparent that errors have crept into Regulation (EEC) No 2568/91;

Whereas, on account of studies under way, the period during which the Member States may use tested, scientifically valid national analysis methods should be extended;

Whereas, because of developments in research, the characteristics of olive oil as defined in Regulation (EEC) No 2568/91, should be adjusted so as better to ensure the purity of the products marketed and to provide for the relevant method of analysis;

Whereas, in order to permit the introduction of the means needed to apply the new method, its entry into force should be deferred for some months;

Whereas Regulation (EEC) No 2568/91 should therefore be amended;

Whereas the measures provided for in this Regulation are in accordance with the opinion of the Management Committee for Oils and Fats,

HAS ADOPTED THIS REGULATION:

Article 1

Regulation (EEC) No 2568/91 is amended as follows:

1. in the first paragraph of Article 3 ‘31 December 1992’ is replaced by ‘28 February 1993’;

([1]) OJ No 172, 30. 9. 1966, p. 3025/66.
([2]) OJ No L 215, 30. 7. 1992, p. 1.
([3]) OJ No L 248, 5. 9. 1991, p. 1.
([4]) OJ No L 327, 13. 11. 1992, p. 28.
([5]) OJ No L 256, 7. 9. 1987, p. 1.
([6]) OJ No L 267, 14. 9. 1992, p. 1.

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2. Article 5 is replaced by the following:

'Article 5

Additional notes 2, 3 and 4 to Chapter 15 of the combined nomenclature set out in Annex I to Council Regulation (EEC) No 2658/87 (*) are replaced by the text set out in Annex XIV to this Regulation.

(*) OJ No L 256, 7. 9. 1987, p. 1.';

3. the Annexes are amended as set out in the Annex to this Regulation.

Article 2

This Regulation shall enter into force on the 21st day following its publication in the *Official Journal of the European Communities*.

However, point 10 of the Annex shall apply from 1 July 1993 to olive oil packaged from that date.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

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ANNEX

1. In the list of Annexes, the title of Annex IV is changed to: Determination of wax content by capillary column gas-liquid chromatography.

2. In the summary, the title of Annex XIII 'Proof that refining has taken place' is replaced by 'Neutralization and decolorization of olive oil in the laboratory.'

3. The first table in Annex I is replaced by the following table:

Type	'Acidity % meq	Peroxide value meq O_2/kg	Halogemated solvents mg/kg (1)	Waxes mg/kg	Saturated fatty acids in position 2 of triglyceride %	Erythiodiol + Uvaol %	Trilinolein %	Cholesterol %	Brassicasterol %	Campesterol %	Stigmasterol %	Betasitosterol % (2)	Delta-7-stigmasterol %	Total sterols mg/kg
1. Extra virgin olive oil	M 1,0	M 20	M 0,20	M 250	M 1,3	M 4,5	M 0,5	M 0,5	M 0,2	M 4,0	< Camp.	m 93,0	M 0,5	m 1 000
2. Virgin olive oil	M 2,0	M 20	M 0,20	M 250	M 1,3	M 4,5	M 0,5	M 0,5	M 0,2	M 4,0	< Camp.	m 93,0	M 0,5	m 1 000
3. Ordinary virgin olive oil	M 3,3	M 20	M 0,20	M 250	M 1,3	M 4,5	M 0,5	M 0,5	M 0,2	M 4,0	< Camp.	m 93,0	M 0,5	m 1 000
4. Virgin lampante olive oil	> 3,3	> 20	> 0,20	►C1 M 350 ◄	M 1,3	M 4,5	M 0,5	M 0,5	M 0,2	M 4,0	—	m 93,0	M 0,5	m 1 000
5. Refined olive oil	M 0,5	M 10	M 0,20	M 350	M 1,5	M 4,5	M 0,5	M 0,5	M 0,2	M 4,0	< Camp.	m 93,0	M 0,5	m 1 000
6. Olive oil	M 1,5	M 15	M 0,20	M 350	M 1,5	M 4,5	M 0,5	M 0,5	M 0,2	M 4,0	< Camp.	m 93,0	M 0,5	m 1 000
7. Crude olive residue oil	m 2,0	—	—	—	M 1,8	m 12	M 0,5	M 0,5	M 0,2	M 4,0	—	m 93,0	M 0,5	m 2 500
8. Refined olive residue oil	M 0,5	M 10	M 0,20	—	M 2,0	m 12	M 0,5	M 0,5	M 0,2	M 4,0	< Camp.	m 93,0	M 0,5	m 1 800
9. Olive residue oil	M 1,5	M 15	M 0,20	> 350	M 2,0	> 4,5	M 0,5	M 0,5	M 0,2	M 4,0	< Camp.	m 93,0	M 0,5	m 1 800

M = maximum, m = minimum.

(1) Overall upper limit for compounds detected by electron capture detector. For components detected individuella the upper limit is 0,10 mg/kg.

(2) Delta-5,23-stigmastadianol + clerosterol + sitosterol + sitostanol + delta-5-avamesterol + delta-5-24 stigmastadienol.

Notes: An oil is to be rejected if any one of its characteristics lies outside the limit laid down.'

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4. The following footnote is added to the second table in Annex I:

'For the purpose or establishing purity, where K_{270} exceeds the limit for the category concerned, it shall be determined again after passage over alumina.'

5. In Annex II, point 1.5*in fine,* the words 'of the two calculations' are replaced by 'of two calculations'.

6. In Annex IV, point 5.1.1, the words 'or seed oil' are deleted.

7. In Annex IV, point 5.2.2, the first two sentences are replaced by the following: 'A solution of 65:35 by volume of hexane and ethyl ether is introduced into the development chamber to an approximate depth of 1 cm. (*).

(*) In these special cases, a 95:5 by volume eluent mixture of benzene and acetone will have to be used to obtain good band separation'.

8. In Annex IV, ►**C1** point 5.2.5.2 and point 6. ◄, the number'100' is replaced by '1 000'and the words 'in square millimetres' are deleted.

9. In the Appendix to Annex IV, figure 1is replaced by the following:

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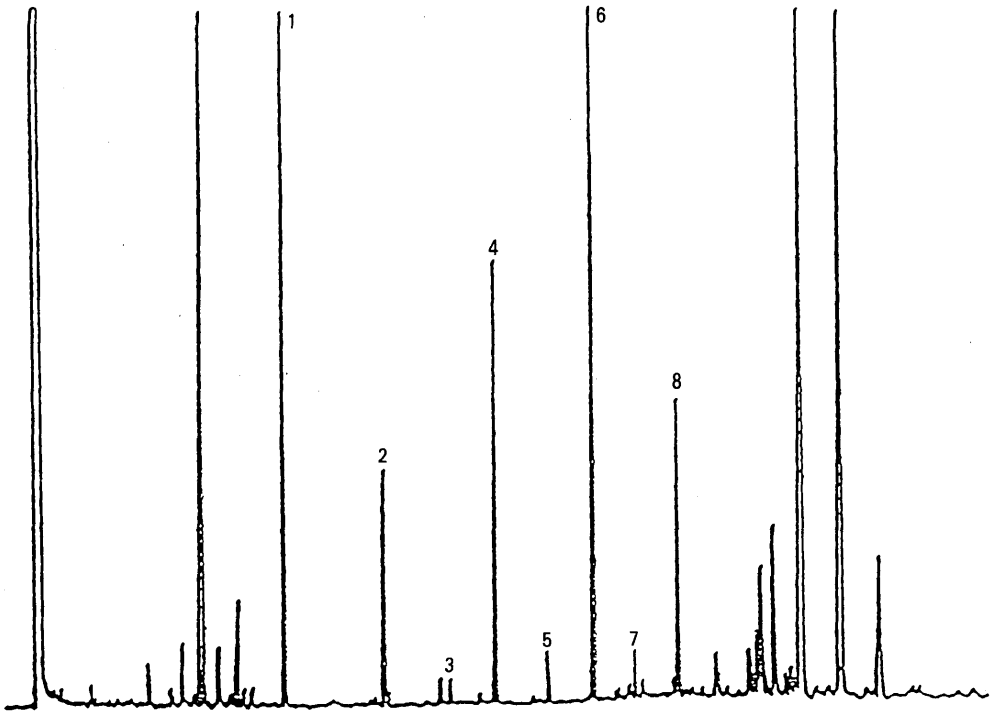

Figure 1 — Chromatogram of the alcoholic fraction of a virgin olive oil

1 = Eicosanol
2 = Decosanol
3 = Tricosanol
4 = Tetracosanol
5 = Pentacosanol
6 = Hexacosanol
7 = Heptacosanol
8 = Octacosanol

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10. Annex IV is replaced by the following text and diagram:

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'ANNEX IV

DETERMINATION OF WAX CONTENT BY CAPILLARY COLUMN GAS-LIQUID CHROMATOGRAPHY

1. SCOPE

This method describes a procedure for the determination of the wax content of certain fats and oils, under the test conditions.

It may be used in particular to distinguish between olive oil obtained by pressing and that obtained by extraction (olive-pomace oil).

2. PRINCIPLE

Addition of a suitable internal standard to the fat or oil, then fractionation by chromatography on a hydrated silica gel column. Recovery of the fraction eluted first under the test conditions (whose polarity is less than that of the triglycerides), then direct analysis by capillary column gas-liquid chromatography.

3. APPARATUS

3.1. 25-ml Erlenmeyer flask.

3.2. Glass column for chromatography, 15 mm internal diameter and 30-40 cm long.

3.3. Suitable gas-liquid chromatograph with a capillary column, equipped with a system for direct introduction into the column comprising the following:

3.3.1 Thermostat-controlled oven for the columns, capable of maintaining the desired temperature to within 1 °C.

3.3.2. Cold injector for direct introduction into the column.

3.3.3. Flame-ionization detector and converter-amplifier.

3.3.4. Recorder-integrator capable of working with the converter-amplifier (3.3.3.), rate of response below 1 second, with variable paper speed.

3.3.5. Capillary column, glass or fused silico, 10 to 15 m long 0,25 to 0,32 mm internal diameter, internally covered with SE-52 or SE-54 liquid, or equivalents, to a uniform thickness of 0.10 to 0,30 μm.

3.4. Microsyringe with facilities for on-column injection capacity 10 μl, equipped with a casehardened needle.

4. REAGENTS

4.1. Silica gel, 70/230 mesh, article 7754 Merck.

Place the gel in the oven at 500 °C for four hours. Allow to cool, then add 2 % water. Shake well the homogenize slurry. Keep in darkness for at least 12 hours prior to use.

4.2. n-hexane, for chromatography.

4.3. Ethyl ether, for chromatography.

4.4. n-heptane, for chromatography.

4.5. Standard solution of lauryl arachidate, at 0,1 % (m/v) in hexane (internal standard).

4.6. Carrier gas: hydrogen, pure, for gas-liquid chromatography.

4.7. Auxiliary gases:

— hydrogen, pure, for gas-liquid chromatography,

— air, pure, for gas-liquid chromatography.

5. PROCEDURE

5.1. Separation of the wax friction.

5.1.1. Preparation of the chromatographic column.

Suspend 15 g of silica gel hydrated at 2 % in anhydrous n-hexane and introduce into the column.

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Allow to settle spontaneously. Complete settling with the aid of an electric shaker to make the chromatographic band more homogeneous. Percolate 30-ml n-hexane to remove any impurities.

5.1.2. Column chromatography

Weigh exactly 500 mg of the sample into a 25-ml flask, and add a suitable amount of internal standard, depending on the assumed wax content, e.g. add 0,1 mg lauryl arachidate in the case of olive oil, and 0,25 to 0,5 mg in the case of olive-pomace oil.

Transfer the prepared sample to the chromatographic column, prepared according to 5.1., with the aid of two 2-ml portions of n-hexane.

Allow the solvent to flow to 1 mm above the upper level of the absorbent. Then start chromatographic elution; collect 140 ml of the n-hexane/ethyl ether mixture, at 99:1, at a flow of about 15 drops every 10 seconds (2,1 ml/minute).

Dry the resultant fraction in a rotary evaporator until almost all the solvent is eliminated. Remove the last 2 or 3 ml of solvent with the help of a weak current of nitrogen, then add 10 ml n-heptane.

5.2. Gas-liquid chromatographic analysis

5.2.1. Preliminary procedure, conditioning of column.

5.2.1.1. Fit the column to the gas-liquid chromatograph, connecting the inlet port to the on-column system and the outlet port to the detector.

Check the gas-liquid chromatography apparatus (operation of gas loops, detector and recorder efficiency, etc.).

5.2.1.2. If the columns is being used for the first time, it is advisable to condition it. Run a light flow of gas through the column, then switch on the gas-liquid chromatography apparatus. Gradually heat to a temperature at least 20 °C above the operating temperature (note). Maintain this temperature for at least two hours, then regulate the apparatus to the operating conditions (regulate gas flow, light flame, connect to electronic recorder, regulate oven temperature for column, regulate detector, etc.). Record the signal at a sensitivity at least twice as high as that required to perform the analysis. The base-line should be linear, with no peaks of any kind, and must have any deviation.

A negative rectilinear deviation indicates that the column connections are not correct; a positive deviation indicates that the column has not been properly conditioned.

Note: Keep the conditioning temperature at all times at least 20 °C below the maximum temperature specified for the eluent employed.

5.2.2. Choice of operating conditions.

5.2.2.1. The operating conditions are generally as follows:

— column temperature: 80 °C at first, rising by 30 °C/minute to 120 °C, then programmed to increase by 5 °C/minute up to 340 °C,
— detector temperature: 350 °C,
— linear speed of carrier gas: hydrogen, 20 to 35 cm/sec,
— instrument sensitivity: 4 to 16 times the minimum attenuation,
— Recorder sensitivity: 1 to 2 mV, from bottom of scale,
— paper speed: 30 cm/hour,
— amount injected: 0,5-1 µl solution.

These conditions may be modified to suit the characteristics of the column and the gas-liquid chromatographic apparatus (in order to obtain chromatgrams meeting the following conditions: retention time of C32 internal standard must be 25 ± 2 minutes and the most representative peak of the waxes must lie between 60 and 100 % from the bottom of the scale).

5.2.2.2. Determine the peak integration parameters in such a way as to obtain a correct evaluation of the peak areas considered.

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5.2.3. Performance of the analysis

5.2.3.1. Take up 1 µl of the solution with the aid of the 10 µl micry-syringe; draw back the piston until the needle is empty. Introduce the needle in the injection system and inject quickly after one to two seconds. After about five seconds, gently extract the needle.

5.2.3.2. Perform the recording until the waxes are completely eluted.

The base-line must always satisfy the required conditions (5.2.1.2).

5.2.4. Peak identification

Identify the peaks from the retention times, by comparing them with mixtures of waxes with known retention times, analysed under the same conditions.

Figure 1 gives a wax chromatogram of a virgin olive oil.

5.2.5. Quantitative analysis

5.2.5.1. Determine the areas of the peaks corresponding to the internal standard and the aliphatic esters from C40 to C46 with the aid of the integrator.

5.2.5.2. Determine the wax content of each of the esters, in mg/kg of fat, according to the formula:

$$\text{ester (mg/kg)} = \frac{A_x \cdot m_s \cdot 100}{A_s \cdot m}$$

where: A_x = area of the peak of each ester;

A_s = area of the lauryl arachidate peak;

m_s = mass of the lauryl arachidate added, in milligram;

m = mass of the sample taken for determination, in grams.

6. EXPRESSION OF THE RESULTS

Give the different wax contents, and the sum of those contents, in mg/kg of fat.

APPENDIX

Determination of linear gas speed

Inject 1 to 3 µl methane (propane) into the gas-liquid chromatographic apparatus, after adjusting it to the normal operating conditions. Measure the time the gas takes to run through the column, from the moment it is injected until the peak emerges (tM).

The linear speed in cm/sec. is given by the formula L/tM where L is the length of the column, in cm, and tM is the time measured in seconds.

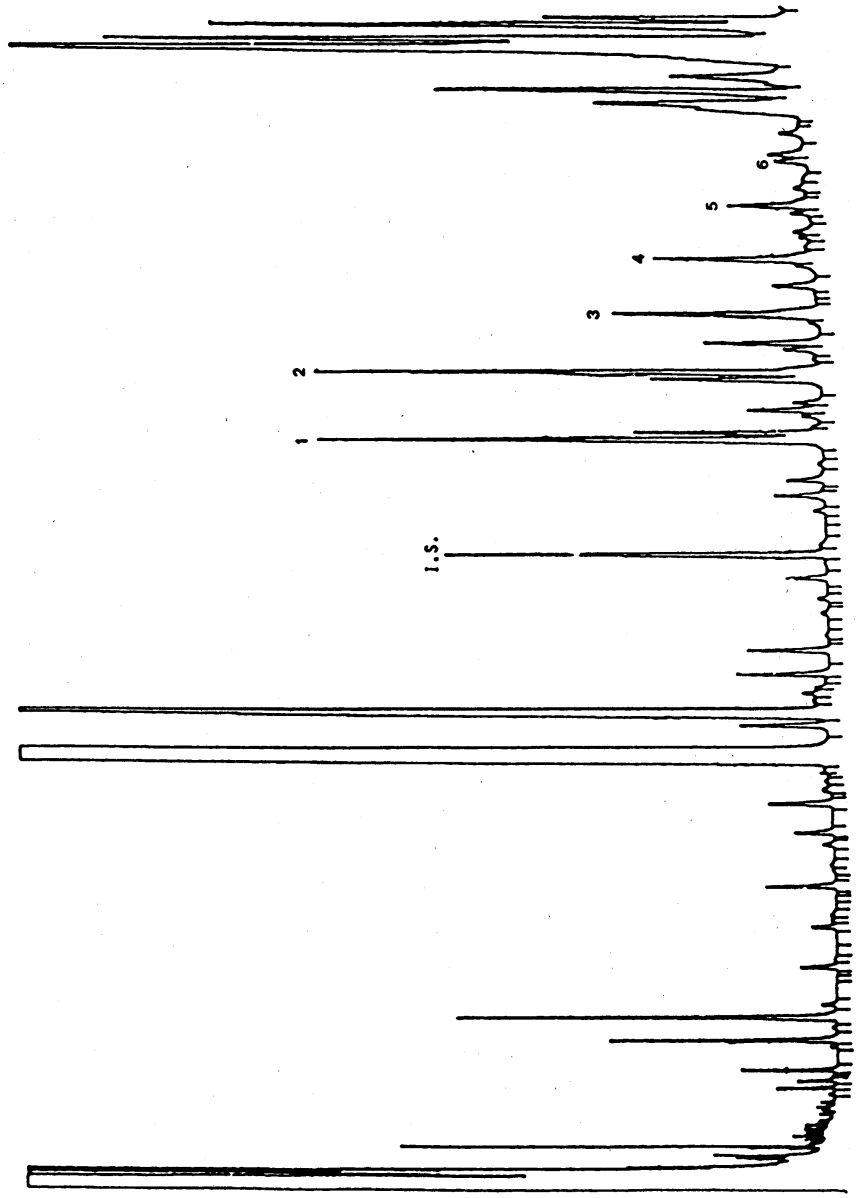

FIGURE 1: *Wax chromatogram of a virgin olive oil.*

I.S. = Internal standard Ester C32
1 = Esters C36,
2 = Esters C38,
3 = Esters C40,
4 = Esters C42,
5 = Esters C44,
6 = Esters C46, '

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11. In Annex V, point 4.11., ‘5 %’is replaced by ‘2 %’.

12. In Annex V, point 5.1.1., first subparagraph, ‘seed oils or’ is deleted.

13. In Annex V, point 5.1.1., third subparagraph, ‘animal or vegetable oils and fats’ is replaced by ‘oils’.

14. In Annex V, point 5.1.1.*in fine,* the following is added: ‘or betulinol will have to be used instead of cholestanol’.

15. In Annex V, point 5.4.5.2., ‘in square millimetres’ is deleted.

16. In Annex VI, point 6., ‘in square millimetres’ is deleted.

17. In Annex IX, point 3.4. is replaced by the following:

‘3.4. Chromatography column having an upper part 270 mm in length and a diameter of 35 mm and a lower part 270 mm in length and a diameter of approximately 10mm.’

18. In Annex IX, point 4.1., the second indentis deleted.

19. In Annex XIII, the title ‘Proof that refining has taken place’ is replaced by the following ‘Neutralization and decolorization of olive oil in the laboratory’.

20. Annex XIV is replaced by the following:

‘ANNEX XIV

ADDITIONAL NOTES 2, 3 AND 4 TO CHAPTER 15 OF THE COMBINED NOMENCLATURE

2. A. Nos 1509 and 1510 cover only oils derived solely from the treatment of lives, the analytical characteristics of the acidic and sterol composition of which are as follows:

Tabble I: Fatty acid composition as percentage of total fatty acids		Table II: Sterol composition as percentage of total sterols	
Myristic acid	M 0,1	Cholesterol	M 0,5
Linolenic acid	M 0,9	Brassicasterol	M 0,2
Arachidic	M 0,7	Campesterol	M 4,0
Eicosanoic acid	M 0,5	Stigmasterol (1)	< Campesterol
Behenic acid	M 0,3	Betasitosterol (2)	m 93,0
Lignoceric acid	M 0,5	Delta-7-stigmastzerol	M 0,5

m = minimum

M = Maximum

(1) Condition not valid for virgin lampante oil (subheading 1509 10 10) and for olive-residue oil (subheading 1510 00 10).

(2) Delta-5,23-Stigmastadienol + Chlerosterol + Beta-Sitosterol + Sitostanol + Delta-5-Avenasterol + Delta-5,24-Stigmastadienol.

Nos 1509 and 1510 do not cover chemically altered olive oil (in particular re-esterified olive oil) and mixtures of olive oil with other oils. The presence of re-esterified olive oil or other oils is ascertained using the methods set out in Annexes V, VIII, X A and X B to Regulation (EEC) No 2568/91.

B. Subheading 1509 10 covers only olive oils defined in Sections I and II belov obtained solely using mechanical or other physical means under conditions, and particularly thermal conditions, that do not lead to deterioration of the oil, and which have undergone no treatment other than washing, decantation, centrifugation or filtration. Oils derived from olives using solvents fall under heading 1510.

I. For the purposes of subheading 1509 10 10, “virgin lampante olive oil”, whatever its acidity, means olive oil with:

(a) an aliphatic alcohols content not exceeding 400 mg/kg;

(b) an erythrodical and uvaol content not exceeding 4,5 %;

(c) a content in saturated fatty acids at the 2-position in the triglycerides not exceeding 1,3 %

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and/or

(d) the sum of transoleic isomers lower than 0,10 % and the sum of translinoleic + translinolenic isomers lower than 0,10 %;

(e) one or more of the following characteristics:

(i) a periode number exceeding 20 meq 0_2/kg;

(ii) a content in volatile halogenated solvents exceeding 0,1 mg/kg for any one solvent;

(iii) a K_{270} (100) extinction coefficient higher than 0,250 and, after treatment of the oil with activated alumina, not higher than 0,11. In point of fact some oils having a free fatty acid content, expressed as oleic acid, of more than 3,3 g per 100 g may, after passage through activated alumina, in accordance with the method set out in Annex IX to Regulation (EEC) No 2568/91, may have a K_{270} extinction coefficient higher than 0,10. If so, after neutralization and decolorization in the laboratory, in accordance with the method set ou in Annex XIII to the aforementioned Regulation, they must have the following characteristics:

— aK_{270} extinction coefficient not higher than 1,20,

— an extinction coefficient variation (Delta K), in the 270 nm region, higher than 0,01 but not higher than 0,16, i.e.:

$$\text{Delta } K = K_m - 0{,}5\ (K_{m-4} + K_{m+4})$$

K_m = the extinction coefficient at the wavelength of the peak of the absorption curve in the 270 nm region,

K_{m-4} en K_{m+4} = the extinction coefficients at wavelengths 4 nm lower and higher than the K_m wavelength;

(iv) organoleptic organoleptic characteristics which include detectable defects exceeding the limits of acceptability and a panel test score lower than 3,5 in accordance with Annex XII to Regulation (EEC) No 2568/91.

II. For the purposes of subheading 1509 10 90, “virgin oil” means olive oil having the following characteristics:

(a) an acid content, expressed as oleic acid, not exceeding 3,3 g per 100 g;

(b) a peroxide number not exceeding 20 meq active 0_2/kg;

(c) an aliphatic alcohols content not exceeding 300 mg/kg;

(d) a content in volatile halogenated solvents not exceeding 0,2 mg/kg overall and not exceeding 0,1 mg/kg for each solvent;

(e) a K_{270} extinction coefficient not higher than 0,250 and, after treatment of the oil with activated alumina, not higher than 0,10;

(f) an extinction coefficient variation (Delta K), in the 270 nm region, not higher than 0,01;

(g) organoleptic characteristics which may include detectable defects within the limits of acceptability and a panel test score higher than 3,5 in accordance with Annex XII to Regulation (EEC) No 2568/91;

(h) an erythrodiol and uvaol content not exceeding 4,5 %;

(i) a content in saturated fatty acids at the 2-position in the triglycerides not exceeding;

(j) the sum of transoleic isomers lower than 0,03 % and the sum of translinoleic + translinolenic isomers lower than 0,03 %.

C. Subheading 1509 90 00 covers olive oil obtained by the treatment of olive oils falling within subheading 1509 10 10 or 1509 10 90, whether or not blended with virgin olive oil, having the following characteristics:

(a) an acid content, expressed as oleic acid, not exceeding 3,3 g per 100 g;

(b) an aliphatic alcohols content not exceeding 350 mg/kg;

(c) A K_{270} extinction coefficient (100) higher than 0,250 and not higher than 1,20 and, after treatment of the sample with activated alumina, higher than 0,10;

(d) an extinction coefficient variation (Delta K), in the 270 nm region, higher than 0,01 and not higher than 0,16;

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(e) an erythrodiol and uvaol content not exceeding 4,5 %;

(f) a content in saturated fatty acids at the 2-position in the triglycerides not exceeding 1,5 %;

(g) the sum of transoleic isomeres lower than 0,20 % and the sum of translinoleic + translinolenic isomeres lower than 0,30 %.

D. For the purposes of subheading 1510 00 10, "crude oils" means oils, particularly olive residue oils, with the following characteristics:

(a) an acid content, expressed as oleic acid, greater than 2 g per 100 g;

(b) an erythrodiol and uvaol content exceeding 12 %;

(c) a content in saturated fatty acids at the 2-position in the triglycerides not exceeding 1,8 %;

(d) the sum of transoleic isomers lower than 0,20 % and the sum of translinoleic + translinolenic isomers lower than 0,10 %.

E. Subheading 1510 00 90 covers oils obtained by the treatment of oils fallin within subheading 1510 00 10, whether or not blended with virgin olive oil, and oils not having the characteristics of the oils referred to in additional notes 2B, 2C and 2D. The oils falling within this subheading must have a content in saturated fatty acids at the 2-position in the triglycerides not exceeding 2 %, the sum of transoleic isomers lower than 0,4 % and the sum of translinoleic + translinolenic isomers lower than 0,35 %.

3. Subheadings 1522 00 31 and 1522 00 39 do not cover:

(a) residues resulting from the treatment of fatty substances containing oil having in iodine index, determined in accordance with the metod laid down in Annex XVI to Regulation (EEC) No 2568/91, lower than 70 or higher than 100;

(b) residues resulting from the treatment of fatty substances containing oil having an iodine index lower than 70 or higher than 100, of which the peadk area representing the retention volume of Beta-Sitosterol (*), determined in accordance with Annex V to Regulation (EEC) No 2568/91, is less than 93 % of the total sterol peak areas.

(*) Delta-5,23-Stigmastadienol + Chlerosterol + Beta-Sitosterol + Sitostanol + Delta-5-Avenasterol + Delta-5,24-Stigmastadienol.

4. The analytical methods for the determination of the characteristics of the products referred to above are those laid down in the Annexes to Regulation (EEC) No 2568/91.'