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REGULATION (EEC) No 618/72 OF THE COMMISSION

of 29 March 1972

on the characteristics of olive oil and of certain products containing olive oil

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community;

Having regard to Council Regulation No 136/66/EEC[1] of 22 September 1966 on the establishment of a common organization of the market in oils and fats, as last amended by Regulation (EEC) No 2727/71,[2] and in particular Article 18 (3) thereof;

Having regard to Council Regulation (EEC) No 443/72[3] of 29 February 1972 on the levies on refined olive oil and on certain products containing olive oil, and in particular Article 8 thereof;

Having regard to Council Regulation No 162/66/EEC[4] of 27 October 1966 on trade in oils and fats between the Community and Greece, and in particular Articles 4 (2), 5 (3) and 9 thereof;

Whereas the export refunds on olive oil may be fixed at different levels for the various types of oil; whereas these oils should therefore be distinguished according to their chemical characteristics;

Whereas Regulation (EEC) No 443/72 provides for different levies for olive oil obtained by refining virgin olive oil and for refined olive-residue oil; whereas these oils should be distinguished according to their chemical characteristics;

Whereas the tariff nomenclature resulting from application of this Regulation is incorporated in the Common Customs Tariff;

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

1. Only oil obtained exclusively from the processing of olives, excluding re-esterified olive oil and any admixtures of olive oil with other types of oil, shall be considered as olive oil within the meaning of subheading No 15.07 A of the Common Customs Tariff.

2. Oils which have the characteristics described in Annex I shall be classified under subheadings Nos 15.07 A I (a) and 15.07 A I (b) of the Common Customs Tariff.

3. Products falling within heading No 15.17 other than those excluded by Annex II shall be classified under subheading No 15.17 A of the Common Customs Tariff.

4. For the purposes of granting the export refund, oils which have the characteristics described in Annex I shall be classified under subheadings Nos 15.07 A II (a) and 15.07 A II (b) as specified in Article 2 of Regulation (EEC) No 616/72.[5]

Article 2

The wording of Annex III shall be substituted for the Additional Note to Chapter 15 of the Common Customs Tariff.

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[1] OJ No 172, 30.9.1966, p. 3025/66.
[2] OJ No L 282, 23.12.1971, p. 8.
[3] OJ No L 54, 3.3.1972, p. 3.
[4] OJ No 197, 29.10.1966, p. 3393/66.
[5] OJ No L 78, 31.3.1972, p. 1.

Article 3

This Regulation shall enter into force on 1 April 1972.

Commission Regulation No 177/66/EEC[1] of 7 November 1966 on the distinction between various refined olive oils is hereby repealed.

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

Done at Brussels, 29 March 1972.

For the Commission

The President

S. L. MANSHOLT

[1] OJ No 203, 8.11.1966, p. 3491/66.

ANNEX I

Characteristics of the olive oil falling within subheadings Nos 15.07 A I (a), 15.07 A I (b) and 15.07 A II (a) and (b)

1. Olive oil having the following characteristics falls within subheading No 15.07 A I (a):

   (a) a free fatty acid content expressed as oleic acid of not more than 3%;

   (b) an extinction coefficient $E_{1\,cm}^{1\%}$ at 270 nm (absorbence of a solution of 1 g of oil in 100 ml of iso octane in a 1 cm cell at a wavelength of 270 nm), exceeding 0·25 but not exceeding 1·10 and, after treatment of the sample of oil on activated alumina, exceeding 0·11;

   (c) a variation in the extinction coefficient at approximately 270 nm, exceeding 0·01 and not exceeding 0·16.

   That variation is expressed by the following equation:

   $$\triangle K = K_m - 0{\cdot}5\,[(K_m - 4) + (K_m + 4)]$$

   Where K_m is the extinction coefficient for the wavelength of the maximum of the absorption curve at approximately 270 nm.

   and $K_m - 4$ and $K_m + 4$ are the extinction coefficients for wavelengths 4 nm less or greater than that of K_m.

   (d) absence of positive reaction by olive-residue oil.

2. Olive oil falls within subheading No 15.07 A I (b):

   (a) if it has the characteristics referred to under point 1 (a) to (c) and a positive reaction by olive-residue oil;

   (b) if it has the characteristics referred to under point 1 (a) and an extinction coefficient $E_{1\,cm}^{1\%}$ at 270 nm exceeding 1·10 and not exceeding 2·00 and a variation in the extinction coefficient at approximately 270 nm not exceeding 0·20.

3. Olive oil having the following characteristics falls within subheading No 15.07 A II (a):

   (a) an extinction coefficient $E_{1\,cm}^{1\%}$ at 270 nm, after treatment of the sample of oil on activated alumina, not exceeding 0·11.

   In exceptional cases certain oils of high acidity, after being passed over activated alumina, may have an extinction coefficient $E_{1\,cm}^{1\%}$ at 270 nm exceeding 0·11. In such cases, after neutralization and decolourization in the laboratory, they must have the characteristics of the oils referred to in point 1;

   (b) a variation in the extinction coefficient at approximately 270 nm not exceeding 0·01;

   (c) absence of positive reaction by olive-residue oil.

4. Olive oil having the following characteristics falls within subheading No 15.07 A II (b):

   (a) a free fatty acid content expressed as oleic acid of more than 3%; and

   (b) a positive reaction by olive-residue oil or, after neutralization and decolourization in the laboratory, the characteristics of the oils referred to under point 2 (b).

METHOD OF ANALYSIS

I. TREATMENT OF SAMPLE BY ACTIVATED ALUMINA

1. Place 30 g of basic alumina, obtained by the process described in paragraph 2, in a chromatographic column about 35 mm in diameter and 450 mm in length, having a draining tube about 10 mm in diameter.

Shake down the alumina mechanically, keeping the column vertical and letting it fall gently several times on a wooden surface. Add 100 ml of a 10% solution of oil in hexane.

Collect the eluate and evaporate the solvent in a vacuum at a temperature below 25 °C.

Determination of the extinction coefficient at 270 nm must be carried out immediately on the oil thus obtained.

2. Basic alumina of Brockmann activity I (0% of water) is obtained by heating pellets of basic alumina (for chromatography) of between 30 μm and 130 μm (average 80 μm) for 3 hours at 380—400 °C. To 100 g of this product add 5 ml of distilled water to obtain basic alumina of a Brockmann activity of between II and III inclusive. Stir frequently; then leave for one night in a hermetically sealed container.

Check on the activity of the alumina

Place 30 g of basic alumina (obtained by the process referred to above) in a chromatography column about 35 mm in diameter and 450 mm in length. Pass through this column, under the conditions specified by the method, a mixture of 95% of virgin olive oil with a E^{1}_{1} at 270 nm extinction coefficient less than 0·18 and 5% of groundnut oil treated during the refining process with decolourizing clays and having a E^{1}_{1} at 270 nm extinction coefficient of not less than 4. If the extinction coefficient of the mixture is greater than 0·11, the alumina is acceptable. If elution of the trienes conjugated on this alumina has not occurred, a more hydrated alumina should be used, after a check that it satisfies the requirements of the preceding test.

II. REACTION OF OLIVE RESIDUE OIL

1. **Apparatus**

— Round-bottomed flask, 100 ml, with a reflux condenser;

— Pipette, 5 ml, graduated in tenths;

— Heating system allowing a temperature of about 80 °C to be reached;

— Thermometer reading from 15 to 60 °C.

2. **Reagents**

— Aqueous alcoholic solution of potassium hydroxide (42·5 of KOH dissolved in 72 ml of distilled water, made up to 500 ml with 95 ° ethyl alcohol);

— Solution of ethyl alcohol, titrated 70 °:

— Aqueous solution of acetic acid 1 + 2 (by volume), adjusted so that 1·5 ml exactly neutralizes 5 ml of the hydro-alcoholic solution of potassium hydroxide in the presence of phenolpthalein.

3. **Preparation of the sample**

The oil is freed from water by decantation and filtration through paper effected at a temperature slightly higher than the fusion point of certain solid constituents which otherwise might separate from the fluid oil or fat.

4. **Procedure**

Place in the flask about 1 ml of oil prepared as indicated in paragraph 3. Add 5 ml of aqueous alcoholic solution of potassium hydroxide. Fit the reflux condenser and boil for 10 minutes, shaking from time to time. Allow to cool to ambient temperature. Add 1·5 ml of aqueous solution of acetic acid and 50 ml of ethyl alcohol solution, previously brought to a temperature of 50 °C. Mix by stirring, introduce the thermometer and allow to cool, observing the appearance of the solution as soon as it reaches a temperature of 45 °C. If a flaky precipitate forms at a temperature higher than 40 °C, the reaction is positive. In the absence of a distinctive flaky precipitate, keep the liquid at ambient temperature, which must be between 20 ° and 22 °C inclusive, for at least 24 hours and if necessary for 48 hours. Observe the solution again: the formation of a flaky precipitate in suspension in the liquid also indicates that the reaction is positive.

III. NEUTRALIZATION AND DECOLOURIZATION OF THE OLIVE OIL IN THE LABORATORY

A. Neutralization of the oil

1. Apparatus

— Beaker, 300 ml, tall;

— Laboratory centrifuge with 100 ml tubes;

— Beaker, 250 ml;

— Round-bottomed flasks, 100 ml;

— Separating funnel, 1 litre.

2. Reagents

— aqueous solution of 12% sodium hydroxide;

— ethyl alcohol solution of 1% phenolphthalein;

— pure hexane, AR;

— pure propan — 2 — ol AR.

3. Procedure

(a) oils with a free fatty acid content, expressed as oleic acid, of less than 30%.

Place 50 g of crude oil in a tall 300 ml beaker and heat to 65 °C in a water bath. Add a quantity of 12% solution of sodium hydroxide corresponding to the free acid of the oil, with an excess of 5%, stirring gently all the time. Continue to stir for 5 minutes, keeping the temperature at 65 °C.

Transfer the mixture into 100 ml centrifuge tubes and separate the soapy paste by centrifugation. Pour the decanted oil into a 250 ml beaker and wash with 50—60 ml of boiling distilled water, removing the water by means of a siphon. Repeat the washings until all traces of residual soap are removed (disappearance of the pink colouring in the phenolphthalein).

Centrifuge the oil to eliminate any small quantities of residual water.

(b) Oils with a free fatty acid content expressed as oleic acid exceeding 30%.

In a 1 l separating funnel place 50 g of crude oil, 200 ml of hexane, 100 ml of propan — 2 — ol and a quantity of 12% solution of sodium hydroxide corresponding to the free acid of the oil, with an excess of 0·3%.

Stir vigorously for 1 minute. Add 100 ml of distilled water, stir again and allow to stand.

After separation of the layers, allow the lower layer containing soaps to drain off. Between the two layers (oily on top and aqueous underneath) an intermediary layer often forms made up of mucilages and insoluble substances which must also be eliminated.

Wash the hexane solution of neutral oil with repeated washings of 50–60 ml of a solution of propan — 2 — ol/distilled water 1:1 (v/v) until the pink colouring disappears from the phenolphthalein. Then remove the hexane by distillation in a vacuum (for example in a rotary evaporator).

B. Decolourization of neutralized oil

1. Apparatus

— Round-bottomed flask, 250 ml, with 3 ground glass necks for the insertion of:

(a) a thermometer graduated in degrees and allowing readings to be taken at 90 °C;

(b) a mechanical stirrer operating at 250—300 revolutions per minute, equipped to operate in a vacuum;

(c) a vacuum pump connection.

— Vacuum pump, with a manometer, capable of giving residual pressure of 15—30 millibars.

2. **Procedure**

Weigh about 100 g of neutralized oil in the 3-necked flask. Insert the thermometer and the stirrer, connect the vacuum pump and heat to 90 °C, stirring all the time. Maintain that temperature, continuing to stir, until the oil to be analysed is entirely free from water (about 30 minutes). Then break the vacuum and add 2 to 3 g of activated earth. Re-establish the vacuum until a residual pressure of 15—30 millibars is obtained and, maintaining a temperature of 90 °C, stir for 30 minutes at about 250 revolutions a minute.

Filter while still hot in a thermostatic oven (50—60 °C).

ANNEX II

Products falling within heading No 15.17 A

The following products do not fall within heading No 15.17 A:

(a) residues resulting from the treatment of fatty substances containing oil of which the iodine value determined by the Wijs method without a catalyst is lower than 70 or higher than 100;

(b) residues resulting from the treatment of fatty substances containing oil of which the iodine value is between 70 and 100 inclusive but of which the surface of the peak having the retention volume of betasitosterol, determined in accordance with the provisions of the following note, represents less than 93% of the total area of the peaks of sterols.

ANALYSIS OF THE STEROL FRACTION OF FATTY SUBSTANCES

Analysis by chromatography in the gaseous phase of sterols prepared by chromatography on thin layers from the unsaponifiable matter dried carefully in a water bath.

Apparatus

1. Apparatus for chromatography on a thin layer, including in particular four glass plates 20×20×0·4 cm, two 20×5×0·4 cm and 1 micro-syringe of 0·1 ml;
2. Beaker, 50 ml;
3. Porous filters, porosity 3, diameter 15 mm;
4. Round-bottomed flask, 100 ml;
5. Centrifuge tube with conical bottom, 10 ml, with a ground glass stopper;
6. Graduated pipettes, 1 ml;
7. Apparatus for chromatography in the gaseous phase, equipped with a flame ionization detector with a silver or glass injector or direct injection system on the column linked to a registering device;
8. Column for chromatography in the gaseous phase, in glass or stainless steel in U or spiral form, from 1 to 2 m in length and from 3 to 4 mm internal diameter — stationary phase of silicone gum (methyl type) stable to at least 300 °C, impregnated calcined diatomaceous earth washed with acids and silanized, of a granulometric measurement 80/100 or 100/120 mesh, at a rate of 2 to 4%;

   N. B. As certain types of stainless steel can cause erroneous results through deterioration of the sterols, glass is recommended.
9. Micro-syringe capable of holding up to 5 or 10 μl.

Reagents

1. Chloroform for chromatography;
2. Benzene for chromatography;
3. Silica gel (for example Kieselgel G);
4. Reference solution for plate chromatography, made up of 5% cholesterol in chloroform;
5. Acetone for chromatography;
6. 0·1% solution of sodium salt of 2'7'-dichlorofluorescein in absolute ethyl alcohol;
7. Solution for the sensitivity test; 1 mg of cholesterol in 1 ml of n-pentane;
8. Solution for testing the resolution of peaks: 0·9 mg of phytosterols of oil of colza and 0·1 mg of cholesterol in 1 ml of n-pentane. The sterols must be freshly prepared in accordance with the procedure described under point A of the Procedure;
9. Solution for the reference test: 1 mg of phytosterols of sunflower oil in 1 ml of n-pentane, freshly prepared as described under point A of the Procedure.

Preparation of plates for chromatography

Place on the layering apparatus one 20×5×0·4 cm plate, four 20×20×0·4 cm plates and one 20×5×0·4 cm plate, in that order.

In a 500 ml round-bottomed flask with a wide neck place 40 g of silica gel and about 80 ml of water. Stir with a glass rod and if necessary with a mechanical stirrer until a homogeneous suspension is reached.

Remove any gases by creating a vacuum, using a water-jet pump for at least one minute. Then spread the suspension on the layering apparatus in a layer 0·5 mm thick and cover the plates uniformly.

Leave the plates to dry in the air for about 15 minutes and then dry in an oven at 105 °C for 2 hours. After preparing the plates in this way, keep them in a dessicator in a vacuum.

PROCEDURE

A. Separation of the sterol fraction by chromatography on a thin layer

Fill the developing tank with benzene/acetone mixture 95:5 (v/v) to a height of about one cm; cover with the lid and leave for at least 3 hours for the liquid/vapour balance to establish itself. It is also recommended to fix bands of filter paper on the interior surfaces of the tank projecting down into the eluant. This will reduce the period of migration of the front of the liquid by about one-third and will provide a more uniform elution of the components.

Meanwhile prepare a 5% solution of unsaponifiable matter extracted by petroleum ether in chloroform. Take about 0·3 ml of this solution and using the 0·1 micro-syringe put it on the chromatographic plate at about 1·5 cm from the lower edge, in a continuous and uniform band, keeping the line from the beginning as thin as possible.

Using the usual technique, place a few µl of the reference solution containing cholesterol at one end of the plate in order to identify the Rf of the sterol fraction.

Place the plate in the developing tank prepared as indicated above. The ambient temperature must be about 20 °C.

Cover with the lid and develop until the front of the solvent reaches about 1 cm from the upper edge of the plate.

Remove the plate from the developing tank and allow the solvent to evaporate in a current of hot air.

Develop by spraying uniformly with the alcohol solution of sodium salt of the 2'7'-dichloro-fluorescein on the plate. By examining the plate by ultraviolet the position of the sterols can be determined through alignment with the cholesterol stain coming from the reference solution.

Collect by scraping the band of sterols with a metal spatula.

Place the separated silica gel in a 50 ml beaker with 15 ml of hot chloroform, stir, transfer the whole of the silica gel to the porous filter and filter it. Wash the filter three times, with 15 ml of hot chloroform each time, collecting the filtrate in a 100 ml round-bottomed flask.

Evaporate the chloroform solution to 4 to 5 ml and pour it into the centrifuge tube previously tared with a ground glass stopper. Evaporate the solution by gently heating in a current of nitrogen until dry and weigh the sterol fraction thus obtained.

Dissolve it in 2 ml of chloroform.

B. Chromatographic analysis of sterols in the gaseous phase

1. *Conditions of analysis by chromatography in the gaseous phase*

Temperature of the column: 220 to 250 °C.

Temperature of the injection system if it is heated separately: 20 to 40 °C above the temperature of the column. Nitrogen flow: 30 to 60 ml/min. Disconnect the detector and

condition the new columns under these conditions for from 16 to 24 hours. Connect the detector, light the flame and regulate the flow of hydrogen, oxygen or air so as to obtain a suitable flame height and sensitivity. Switch on the registering device and allow the paper to unroll at an appropriate speed; adjust the zero and the measuring range attenuator. If the base line is stable, the apparatus is ready for use.

2. *Sensitivity test*

Inject 3 to 5 μl of the sensitivity test solution (7). A peak of cholesterol must only appear on the chromatogram.

Regulate the measuring range attenuator so as to use approximately the whole scale of the registering device.

3. *Peak resolution test*

Inject 3 to 5 μl of the resolution test solution (8). Peaks will appear on the chromatogram for cholesterol, brassicasterol, campesterol and β-sitosterol. Measure the retention distances (distances from the injection point to the maximum height of the peak) of the peaks, d_{CH} for cholesterol, d_B for brassicasterol, d_C for campesterol and d_S for β-sitosterol and the widths of the base of the peaks (length of retention between the intersections with the base line of the tangents at the inflection points on the sides before and after the peak), w_{CH} for cholesterol and w_B for brassicasterol.

The resolution of peaks, expressed by the formula:

$$PR = 2\,(d_B - d_{CH}) \,/\, (w_B + w_{CH})$$

must equal at least 1.

Calculate the relative retention times (cholesterol = 1·00) for brassicasterol, campesterol and β-sitosterol.

4. *Reference test*

Inject 3 to 5 μl of the reference test solution (9). The campesterol, stigmasterol, β-sitosterol and Δ 7 stigmasterol peaks should appear on the chromatogram.

Measure the retention distances of the peaks, d_C for campesterol, d_{ST} for stigmasterol, d_S for β-sitosterol and d_{ST}-7 for Δ7 stigmasterol.

Calculate the relative retention times which are approximately:

Cholesterol	1·00 (about 15 minutes)
Brassicasterol	1·13—1·15
Campesterol	1·32—1·34
Stigmasterol	1·44—1·46
β-sitosterol	1·66—1·68
Δ 7 Stigmasterol	1·88—1·92

5. *Analysis*

Inject 3 to 5 μl of the solution to be analyzed and register the chromatogram.

C. **Expression of results**

In interpreting the composition of the sterol fraction analyzed, peaks having different retention times from those determined experimentally for the 6 sterols mentioned above, should be ignored.

The content % of β-sitosterol is given by the formula:

$$\frac{\text{Area of the } \beta\text{-sitosterol peak}}{\text{Sum of the areas of the six sterol peaks}} \times 100$$

ANNEX III

ADDITIONAL NOTES

1. For the purposes of heading No 15.07:

   A. Fixed vegetable oils, fluid or solid, obtained by pressing are considered as 'crude' if they have not undergone treatment other than:

   — decantation within the normal period of time;

   — centrifugation or filtration, provided that in order to separate the oil from its solid constituents only mechanical force, such as gravity, pressure or centrifugal force, has been employed (excluding any absorption filtering process and any physical or chemical process).

   B. Fixed vegetable oils, fluid or solid, obtained by extraction continue to be considered as 'crude' when they can not be distinguished by their colour, odour or taste or by any recognized special analytical properties, from vegetable oils and fats obtained by pressing.

   C. The expression 'crude oils' shall be taken to extend to de-gummed soya oil and cotton-seed oil from which the gossypol has been removed.

2. A. Only oil obtained exclusively from the processing of olives, excluding re-esterified olive oil and any admixtures of olive oil with other types of oil, is considered as olive oil within the meaning of subheading No 15.07 A.

   B. Olive oil with the following characteristics falls within subheading No 15.07 A I (a):

   (a) a free fatty acid content expressed as oleic acid of not more than 3%;

   (b) an extinction coefficient E^{1}_{1} at 270 nm (absorbence of a solution of 1 g of oil in 100 ml of iso octane in a 1 cm cell at a wavelength of 270 nm), higher than 0·25 and not exceeding 1·10 and, after treatment of the sample of oil on activated alumina, higher than 0·11;

   (c) a variation in the extinction coefficient at approximately 270 nm, exceeding 0·01 and not exceeding 0·16

   That variation is expressed in the following equation:

   $$\triangle K = K_m - 0{\cdot}5\,[(K_m - 4) + (K_m + 4)]$$

   where

   K_m represents the extinction coefficient for the wavelength of the maximum of the absorption curve at approximately 270 nm.

   and

   $K_m - 4$ and $K_m + 4$ describe the extinction coefficients for wavelenghts lower or higher by 4 nm than that of K;

   (d) absence of positive reaction of olive-residue oil.

   C. Olive oil falls within subheading No 15.07 A I (b) if it has:

   (a) either the characteristics referred to under point 2 B (a) to (c) and a positive reaction of olive-residue oil;

   (b) or the characteristics referred to under point 2 B (a) and an extinction coefficient E^{1}_{1} at 270 nm higher than 1·10 but not higher than 2·00, and a variation in the extinction coefficient at approximately 270 nm not exceeding 0·20.

   D. The expression 'virgin olive oil' is taken to mean natural olive oil obtained exclusively by mechanical processes, including pressing, and excludes any mixture with olive oil obtained in a different manner.

3. The following do not fall within subheading No 15.17 A:

   (a) residues from the treatment of fatty substances containing oil of which the iodine value, determined by the Wijs method, without a catalyst, is lower than 70 or higher than 100;

   (b) residues from the treatment of fatty substances containing oil of which the iodine value is between 70 and 100 inclusive but of which the surface of the peak, having the retention volume of β-sitosterol, determined in accordance with the provisions of Annex II to the Regulation referred to in Additional Note 4 beneath, represents less than 93% of the total area of the sterol peaks.

4. The methods of analysis for the determination of the characteristics of the products referred to above are those laid down in Annexes I and II respectively of Regulation (EEC) No 618/72.