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I

(Acts whose publication is obligatory)

# COUNCIL REGULATION (EEC) No 1055/77

of 17 May 1977

on the storage and movement of products bought in by an intervention agency

THE COUNCIL OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community, and in particular Article 43 thereof.

Having regard to the proposal from the Commission,

Having regard to the opinion of the European Parliament (1),

Whereas in some cases it is essential for an intervention agency to store products bought in pursuant to Community provisions, outside the territory of the Member State within whose jurisdiction it falls;

Whereas, however, in view of the additional costs that may thus be incurred and the problems connected with the disposal of such products, such storage should only be allowed subject to prior authorization from the Community;

Whereas, for the sake of administrative simplicity and in view of the need to avoid trade disturbances, suitable machinery should be instituted to ensure that transport and marketing operations in respect of the products in question proceed in a simple manner and in accordance with market requirements; whereas, moreover, the same rules may be applied in the case of the transport of products being transferred from one intervention agency to another;

Whereas the implementation of such a system requires a derogation to be made from the systems of amounts levied or granted in trade in agricultural products, and from the price system in cases where the intervention agency holding the product has to apply prices that are not valid in the territory of the Member State within whose jurisdiction it falls,

(1) OJ No C 259, 4. 11. 1976, p. 47.

HAS ADOPTED THIS REGULATION:

#### Article 1

- 1. Intervention agencies may not store products they have bought in pursuant to Community provisions outside the territory of the Member State within whose jurisdiction they fall unless they have obtained prior authorization in accordance with the procedure laid down in Article 4.
- 2. Authorization shall be granted if such storage is essential and taking into account the following factors:
- (a) storage possibilities and storage requirements in the Member State within whose jurisdiction the intervention agency falls and in other Member States;
- (b) any additional costs resulting from storage in the Member State within whose jurisdiction the intervention agency falls and from transportation.
- 3. Authorization for storage in a third country shall only be granted if, on the basis of the criteria set out in paragraph 2, storage in another Member State creates significant difficulties.
- 4. The information referred to in paragraph 2 (a) shall be drawn up after consulting all the Member States.

#### Article 2

The customs duties and other amounts to be granted or levied under the common agricultural policy shall not apply to products:

- transported following an authorization granted under Article 1, or
- transferred from one intervention agency to another.

#### Article 3

- 1. Any intervention agency which uses an authorization granted under Article 1 shall remain responsible for products stored outside the territory of the State within whose jurisdiction it falls.
- 2. If products held by an intervention agency outside the territory of the Member State within whose jurisdiction it falls are not brought back into that Member State, they shall be disposed of at the prices and subject to the conditions laid down or to be laid down for the place of storage.

#### Article 4

Rules for the application of this Regulation, and in particular the conditions of disposal, shall be adopted in accordance with the procedure laid down in Article 26 of Council Regulation (EEC) No 2727/75 of 29 October 1975 on the common organization of the market in cereals (1) or, where appropriate, in the corresponding Articles of other agricultural Regulations establishing similar procedures, if necessary with

derogations from the rules on trade, but only as far as is strictly necessary for implementation of this Regulation.

#### Article 5

The Economic Union of Belgium and Luxembourg shall be considered as a single Member State for the purposes of this Regulation.

#### Article 6

In accordance with the procedure provided for in Article 4, temporary measures may be adopted to facilitate transition to the system laid down in this Regulation.

#### Article 7

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

It shall apply from the first day of the third month following its entry into force.

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

Done at Brussels, 17 May 1977.

For the Council
The President
J. SILKIN

#### COUNCIL REGULATION (EEC) No 1056/77

of 17 May 1977

derogating from and amending for the fourth time Regulation (EEC) No 1163/76 on the granting of a conversion premium in the wine sector

THE COUNCIL OF THE EUROPEAN COMMUNITIES.

Having regard to the Treaty establishing the European Economic Community, and in particular Article 43 thereof,

Having regard to Council Regulation (EEC) No 1163/76 of 17 May 1976 on the granting of a conversion premium in the wine sector (1), as last amended by Regulation (EEC) No 530/77 (2), and in particular Article 4 (5) thereof,

Having regard to the proposal from the Commission,

Having regard to the opinion of the European Parliament (3),

Whereas Regulation (EEC) No 1163/76 provides for the granting of a conversion premium in the wine sector; whereas, under the first indent of Article 3 (2) (b) of that Regulation, the applicant must undertake to grub up or have grubbed up by 1 May 1977 the vines on the areas for which the premium has been requested for the 1976/77 wine year; whereas various operations covered by the said Regulation have been delayed by recent unfavourable weather conditions;

Whereas grubbing should therefore be allowed until 15 June 1977; whereas provision should be made in case such a derogation is also required in subsequent wine years;

Whereas Regulation (EEC) No 1163/76 provides that the premiums fixed for the 1976/77 wine year are to be reduced by 100 u.a./hectare for the 1977/78 wine year and by 200 u.a./hectare for the 1978/79 wine year; whereas it is to be foreseen that these reduced sums will be considered inadequate by producers; whereas there is in consequence a risk of considerable reduction in the conversion programme, meaning that the aims of that Regulation will not be fully achieved; whereas it is therefore desirable to retain the premiums for the 1977/78 and 1978/79 wine years at the level of the present wine year,

HAS ADOPTED THIS REGULATION:

#### Article 1

By way of derogation from Regulation (EEC) No 1163/76 for the 1976/77 wine year and as from 1 May 1977: for the purposes of granting the conversion premium, the grubbing of vines shall take place before 16 June 1977 for the 1976/77 wine year.

#### Article 2

Article 4 of Regulation (EEC) No 1163/76 is replaced by the following text:

'Article 4

- The amount of the premium shall be fixed:
- (a) at 1 500 u.a./ha for areas under vines of average productivity which are kept in normal conditions and do not yet show any signs of deterioration due to age;
- (b) at 1 000 u.a./ha for areas under vines of low productivity or areas under vines which are less than two years old;
- (c) at 2 000 u.a./ha for areas under vines grown as a specialized crop using an upright cultivation method resulting in considerable vegetative growth in a horizontal plane.
- For the grant of the premium the mixed cultivation areas shall be expressed as specialized cultivation areas by using the customary conversion factor for the wine-growing area concerned.
- Where the area to be converted for which the premium is requested is planted with a mixture of several vine varieties, some of which do not qualify for the premium, the premium shall be granted:
- for the entire area, provided that the varieties referred to in Article 2 (1) (a) cover more than 70 % of the area in question,
- for the part of the area actually planted with the varieties referred to in Article 2 (1) (a) where those varieties cover 70 % or less of the area in question.

<sup>(1)</sup> OJ No L 135, 24. 5. 1976, p. 34.

<sup>(</sup>²) OJ No L 69, 16. 3. 1977, p. 34. (³) Opinion given on 13 May 1977 (not yet published in the Official Journal).

- 4. The premium shall be paid in full not more than six months after the applicant has supplied proof that grubbing-up has in fact taken place.
- 5. The Council, acting by a qualified majority on a proposal from the Commission, may decide to alter:
- the amount of the premium,
- the dates set out in Article 3 (1),
- the deadline for grubbing up.
- 6. Detailed rules for implementing this Article shall be adopted in accordance with the procedure laid down in Article 7 of Regulation No 24.'

# Article 3

The 78 million units of account referred to in Article 8 of Regulation (EEC) No 1163/76 are replaced by 84 million units of account.

# Article 4

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

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

Done at Brussels, 17 May 1977.

For the Council

The President

J. SILKIN

#### COUNCIL REGULATION (EEC) No 1057/77

of 17 May 1977

amending Regulation (EEC) No 350/77 laying down certain interim measures for the conservation and management of fishery resources

THE COUNCIL OF THE EUROPEAN COMMUNITIES.

Having regard to the Treaty establishing the European Economic Community, and in particular Article 103 thereof,

Having regard to the Act of Accession, and in particular Article 102 thereof,

Having regard to the proposal from the Commission,

Whereas all the latest scientific estimates of the North Sea herring stock for 1977 are not yet available; whereas, therefore, the prohibition on herring fishing in the North Sea should be extended to 30 June 1977 on the understanding that the Council will have to adopt before 1 July 1977 the measures to be applied for the remaining part of 1977;

Whereas, however, an exception to this prohibition should be made for the Netherlands as a result of the particular situation of that country;

Whereas the prohibition on herring fishing in the North Sea involves the risk that the fishing effort will be transferred to other zones; whereas herring fishing in the West of Scotland zone should therefore be regulated,

HAS ADOPTED THIS REGULATION:

# Article 1

The text of paragraphs 1 and 2 of Article 1 of Council Regulation (EEC) No 350/77 of 18 February 1977 laying down certain interim measures for the conserva-

tion and management of fishery resources (1), as amended by Regulation (EEC) No 879/77 (2), shall be replaced by the following:

'1. Fishing for North Sea herring shall be prohibited from 28 February 1977 until 30 June 1977 in sub-area IV and division VII-d defined by the International Council for the Exploration of the Sea.

However, the Kingdom of the Netherlands shall be authorized to catch up to 1 500 tonnes of North Sea herring in June by direct fishing.

2. The Council, acting by a qualified majority on a proposal from the Commission, shall lay down, before 1 July 1977, the measures to be applied for the remaining part of 1977.

These measures shall be determined taking account of the results of the latest available scientific evidence concerning the North Sea herring stock for 1977.'

#### Article 2

Regulation (EEC) No 350/77 shall be supplemented by the following Article 2a:

'Article 2a

Herring fishing shall be prohibited in division VI-a defined by the International Council for the Exploration of the Sea (West of Scotland including Donegal Bay) until 30 June 1977.'

#### Article 3

This Regulation shall enter into force on 1 June 1977.

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

Done at Brussels, 17 May 1977.

For the Council

The President

J. SILKIN

<sup>(</sup>¹) OJ No L 48, 19. 2. 1977, p. 28. (²) OJ No L 106, 29. 4. 1977, p. 30.

# **COMMISSION REGULATION (EEC) No 1058/77**

of 18 May 1977

on the characteristics of olive oil and of certain products containing olive oil and amending the Common Customs Tariff nomenclature as regards 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 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 1707/73 (2), and in particular Articles 13 (4) and 18 (3) thereof.

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

Having regard to Council Regulation (EEC) No 443/72 of 29 February 1972 on the levies on refined olive oil and on certain products containing olive oil (4), and in particular Article 8 thereof,

Whereas at present all virgin olive oils fall within subheading 15.07 A II a) of the Common Customs Tariff; whereas, therefore, a single levy is fixed for those products; whereas, so that the import levy may achieve its objective, it should be fixed according to the various types of virgin oil and for that purpose the types of olive oil should be distinguished in accordance with their physical and chemical characteristics; whereas, consequently, the nomenclature of the Common Customs Tariff should be amended;

Whereas the tariff nomenclature resulting from Regulation No 136/66/EEC is incorporated in the Common Customs Tariff annexed to Regulation (EEC) No 950/68 (5), as last amended by Regulation (EEC) No 874/77 (6);

Whereas to ensure the proper functioning of the system of levies applicable to imports to olive-oil cakes, a single method should be laid down for determining their oil content;

Whereas Commission Regulation (EEC) No 618/72 of 29 March 1972 on the characteristics of olive oil and of certain products containing olive oil (7) has been

amended on several occasions, in particularly by Regulation (EEC) No 3366/75(8); whereas in view of the new amendments to be made thereto, it should be repealed and all its provisions incorporated in a new Regulation;

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

For the purposes of subheading 15.07 A of the Common Customs Tariff, 'olive oil' means oil obtained solely from the processing of olives, excluding re-esterified olive oil and mixtures of olive oil with other types of oil.

Tests for the presence of re-esterified olive oil or of other types of oil shall be carried out by means of the methods set out in Annexes VII and VIII respectively.

- Oils which have the characteristics described in paragraphs 1, 2 and 3 of Annex I shall be classified under subheadings 15.07 A I a), 15.07 A I b) and 15.07 A I c) of the Common Customs Tariff. The said characteristics shall be determined in accordance with the methods laid down in Annexes IV, V and VI.
- Oils which have the characteristics described in paragraph 4 of Annex I shall be classified under subheading 15.07 A II a) of the Common Customs Tariff.
- Products falling within heading No 15.17 which have the characteristics described in Annex II shall not be classified under subheading 15.17 A of the Common Customs Tariff.

# Article 2

The olive oil content of olive-oil cake and other residues resulting from the extraction of olive oil falling within subheading 23.04 A shall be determined in accordance with the method set out in Annex IX.

<sup>(</sup>¹) OJ No 172, 30. 9. 1966, p. 3025/66. (²) OJ No L 175, 29. 6. 1973, p. 5.

<sup>(3)</sup> OJ No 197, 29. 10. 1966, p. 3393/66.

<sup>(\*)</sup> OJ No L 54, 3. 3. 1972, p. 3. (5) OJ No L 172, 22. 7. 1968, p. 1. (6) OJ No L 106, 29. 4. 1977, p. 20.

<sup>(&</sup>lt;sup>7</sup>) OJ No L 78, 31. 3. 1972, p. 5.

<sup>(8)</sup> OJ No L 333, 30. 12. 1975, p. 13.

2. The olive oil content referred to in paragraph 1 shall be expressed in terms of its weight as a percentage of dry matter.

# Article 3

The Common Customs Tariff annexed to Regulation (EEC) No 950/68 is hereby amended as follows:

- 1. In Additional Note 1 to Chapter 15, '15.07' is replaced by 'subheading 15.07 D'.
- 2. Additional Notes 2, 3 and 4 to Chapter 15 are replaced by Additional Notes 2, 3 and 4 in Annex III to this Regulation.
- 3. Subheading 15.07 A is amended to read as follows:

		Rate of duty			
Heading No	Description	Autonomous % or levy (L)	Conventional %		
1	2	3	4		
15.07	A. Olive oil:				
	I. Untreated:		1.		
	a) Virgin olive oil	20 (L)	•		
	b) Virgin lampante olive oil	20 (L)			
	c) Other	20 (L)			
	II. Other:				
	a) Obtained by processing oils				
	falling within subheading	•			
	15.07 A I a) or 15.07 A I b), whether or not blended with				
	virgin olive oil	20 (L)			
	b) Other	20 (L)			

# Article 4

Any reference in a Community instrument to subheadings 15.07 A II or 15.07 A I a) and b) of the Common Customs Tariff shall be construed, according to the characteristics of the product concerned, as a reference to subheadings 15.07 A I a), b) and c) or 15.07 A II a) and b) respectively of the Common Customs Tariff.

# Article 5

Commission Regulation (EEC) No 618/72 is hereby repealed.

# Article 6

This Regulation shall enter into force on the 43rd day following its publication in the Official Journal of the European Communities.

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

Done at Brussels, 18 May 1977.

For the Commission
Finn GUNDELACH
Vice-President

# **ANNEXES**

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#### ANNEX I

#### CHARACTERISTICS OF OLIVE OILS

- 1. For the purposes of subheading 15.07 A I a) of the Common Customs Tariff, 'virgin olive oil' means natural olive oil obtained exclusively by mechanical processes, including pressure, but does not include mixtures with olive oil obtained otherwise, having the following characteristics:
  - (a) a free fatty acid content, expressed as oleic acid, not greater than 3 %;
  - (b) a K<sub>270</sub> extinction coefficient (absorption under a thickness of 1 cm of solution of 1 g of oil per 100 ml in iso-octane (2,2,4-trimethylpentane) at a wavelength of 270 nm) not higher than 0·25 and, after treatment of the sample of oil with activated alumina, not higher than 0·11;
  - (c) an extinction coefficient variation, in the 270 nm region, not higher than 0.01.

This variation is defined by:

$$\Delta K = K_m - 0.5 (K_{m-4} + K_{m+4})$$

where  $K_m$  is the extinction coefficient at the wavelength of the maximum of the absorption curve in the 270 nm region and

 $K_{m-4}$  and  $K_{m+4}$  are the extinction coefficients at wavelengths 4 nm lower and higher, respectively than that of  $K_m$ ;

- (d) negative Bellier and modified Vizern reactions, determined by the methods specified in Annex V, Sections A and B;
- (e) negative soap test carried out according to the method described in Annex VI.
- 2. For the purposes of subheading 15.07 A I b) of the Common Customs Tariff, 'virgin lampante oil', whatever its acidity, means olive oil having the following characteristics:
  - (a) a  $K_{270}$  extinction coefficient higher than 0.25 and, after treatment of the sample with activated alumina, not higher than 0.11.

Some oils having a free fatty acid content, expressed as oleic acid, of more than 3 % may have, after passage through activated alumina, a  $K_{270}$  extinction coefficient higher than 0·11. If so, after neutralization and decolorization in the laboratory by the method specified in Annex IV, they must have the following characteristics:

- a K<sub>270</sub> extinction coefficient not higher than 1·10,
- an extinction coefficient variation, in the 270 nm region, higher than 0.01 but not higher than 0.16:
- (b) negative Bellier and modified Vizern reactions determined by the methods specified in Annex V, Sections A and B;
- (c) a negative soap test carried out according to the method described in Annex VI.
- 3. Subheading 15.07 A I c) of the Common Customs Tariff covers oils, especially oils from 'olive residues', having the following characteristics:
  - (a) a free fatty acid content, expressed as oleic acid, higher than 3 %;
  - (b) positive Bellier and modified Vizern reactions, determined by the methods described in Annex V, Sections A and B;
  - (c) a negative soap test, carried out by the method described in Annex VI.
- 4. Subheading 15.07 A II a) of the Common Customs Tariff covers olive oil obtained by the treatment of olive oils falling within subheading 15.07 A I a) or 15.07 A I b), whether or not blended with virgin olive oil, having the following characteristics:
  - (a) a free fatty acid content, expressed as oleic acid, not exceeding 3 %;
  - (b) a positive soap test carried out by the method described in Annex VI,

or:

- a K<sub>270</sub> extinction coefficient higher than 0·25 but not higher than 1·10 and, after treatment of the sample of oil with activated alumina, higher than 0·11, and
- an extinction coefficient variation, in the 270 nm region, higher than 0.01 but not higher than 0.16.

This variation is defined by:

$$\Delta K = K_m - 0.5 (K_{m-4} + K_{m+4})$$

where  $K_m$  is the extinction coefficient at the wavelength of maximum absorption on the absorption curve in the 270 nm region, and

 $K_{m-4}$  and  $K_{m+4}$  are the extinction coefficients at wavelengths 4 nm lower and higher, respectively than that of  $K_m$ ;

(c) negative Bellier and modified Vizern reactions, determined by the methods specified in Annex V, Sections A and B.

#### ANNEX II

#### PRODUCTS FALLING WITHIN SUBHEADING 15.17 A

Subheading 15.17 A does not cover:

- (a) residues resulting from the treatment of fatty substances containing oil having an iodine index, determined by the Wijs method without catalyst, lower than 70 or higher than 100;
- (b) residues resulting from the treatment of fatty substances containing oil having an iodine index not lower than 70 or not higher than 100, of which the peak area representing the retention volume of  $\beta$ -sitosterol, determined in accordance with the provisions of Annex VIII, is less than 93 % of the total sterol peak areas.

# ANNEX III

# ADDITIONAL NOTES 2, 3 AND 4 TO CHAPTER 15 OF THE COMMON CUSTOMS TARIFF

- 2. A. For the purposes of subheading 15.07 A, 'olive oil' means oil derived solely from the treatment of olives, excluding re-esterified olive oil and mixtures of olive oil with other oils.
  - B. 'Untreated olive oil' means oil with characteristics as defined in Sections I, II and III below.
    - I. For the purposes of subheading 15.07 A I a), 'virgin olive oil' means natural olive oil obtained exclusively by mechanical processes, including pressure, but does not include mixtures with olive oil obtained otherwise, having the following characteristics:
      - (a) a free fatty acid content, expressed as oleic acid, not greater than 3 %;
      - (b) a K<sub>270</sub> extinction coefficient (absorption under a thickness of 1 cm of solution of 1 g of oil per 100 ml in iso-octane (2,2,4-trimethylpentane) at a wavelength of 270 nm) not higher than 0.25 and, after treatment of the sample of oil with activated alumina, not higher than 0.11;

(c) an extinction coefficient variation, in the 270 nm region, not higher than 0.01.

This variation is defined by:

$$\Delta K = K_m - 0.5 (K_{m-4} + K_{m+4})$$

where K<sub>m</sub> is the

is the extinction coefficient at the wavelength of the maximum of the absorption curve in the 270 nm region and

 $K_{m-4}$  and  $K_{m+4}$  are the extinction coefficients at wavelengths 4 nm lower and higher, respectively than that of  $K_m$ ;

- (d) negative Bellier and modified Vizern reactions;
- (e) a negative soap test.
- II. For the purposes of subheading 15.07 A I b), 'virgin lampante oil', whatever its acidity, means olive oil having the following characteristics:
  - (a) a K<sub>270</sub> extinction coefficient higher than 0·25 and, after treatment of the sample with activated alumina, not higher than 0·11. Some oils having a free fatty acid content, expressed as oleic acid, of more than 3 % may have, after passage through activated alumina, a K<sub>270</sub> extinction coefficient higher than 0·11. If so, after neutralization and decolorization in the laboratory, they must have the following characteristics:
    - a K<sub>270</sub> extinction coefficient not higher than 1·10;
    - an extinction coefficient variation, in the 270 nm region, higher than 0.01 but not higher than 0.16;
  - (b) negative Bellier and modified Vizern reactions;
  - (c) a negative soap test.
- III. Subheading 15.07 A I c) covers oils, especially oils from 'olive residues', having the following characteristics:
  - (a) a free fatty acid content, expressed as oleic acid, higher than 3 %;
  - (b) positive Bellier and/or modified Vizern reactions;
  - (c) a negative soap test.
- C. Subheading 15.07 A II a) covers olive oil obtained by the treatment of olive oils falling within subheading 15.07 A I a) or 15.07 A I b), whether or not blended with virgin olive oil, having the following characteristics:
  - (a) a free fatty acid content, expressed as oleic acid, not exceeding 3 %;
  - (b) a positive soap test, or a K<sub>270</sub> extinction coefficient higher than 0.25 but not higher than 1.10 and, after treatment of the sample of oil with activated alumina, higher than 0.11 and
    - an extinction coefficient variation, in the 270 nm region, higher than 0.01 but not higher than 0.16.

This variation is defined by:

$$\Delta K = K_m - 0.5 (K_{m-4} + K_{m+4})$$

where K<sub>m</sub> is the exti

is the extinction coefficient at the wavelength of maximum absorption curve in the 270 nm region and

 $K_{m-4}$  and  $K_{m+4}$  are the extinction coefficients at wavelengths 4 nm lower and higher, respectively than that of  $K_m$ ;

(c) negative Bellier and modified Vizern reactions.

- 3. Subheading 15.17 A does not cover:
  - (a) residues resulting from the treatment of fatty substances containing oil having an iodine index, determined by the Wijs method, without catalyst, lower than 70 or higher than 100;
  - (b) residues resulting from the treatment of fatty substances containing oil having an iodine index not lower than 70 or higher than 100, of which the peak area representing the retention volume of β-sitosterol, determined in accordance with the provisions in Annex VIII to the Regulation mentioned in Additional Note 4 below, is less than 93 % of the total sterol peak areas.
- 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 1058/77.

#### ANNEX IV

#### I. TREATMENT OF THE SAMPLE WITH ACTIVATED ALUMINA

1. Into a chromatography column approximately 35 mm in diameter and 450 mm in length, fitted with an outlet tube approximately 10 mm in diameter, place 30 g of basic alumina prepared in accordance with the process described in Section 2.

Tamp down the alumina mechanically by allowing the column, held in the vertical plane, to fall gently on a wooden surface, repeating the process as often as necessary. Into the column prepared in this way place 100 ml of a 10 % solution of the oil in hexane.

Collect the eluate and evaporate off the solvent under vacuum at a temperature not exceeding 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 % moisture) is prepared by heating basic alumina (chromatography grade) of particle size in the range 30 to 130 μm (average 80 μm) for three hours at 380 to 400 °C. Add 5 ml of distilled water to 100 g of this product to obtain basic alumina with Brockmann activity II-III. Shake frequently and allow to stand overnight in a hermetically sealed container.

#### Alumina activity test

Into a chromatography column approximately 35 mm in diameter and 450 mm in length place 30 g of basic alumina (prepared in the manner described above). Pass a mixture of 95 % of olive oil having a K<sub>270</sub> extinction coefficient not exceeding 0·18 and 5 % of peanut oil treated with bleaching clay during its refining and having a K<sub>270</sub> extinction coefficient not exceeding four through this column under the conditions specified by the method. If the mixture has an extinction coefficient exceeding 0·11, the alumina is acceptable. If the conjugated trienes remain uncluted by the alumina, it is necessary to use an alumina with a higher degree of hydration, after ascertaining whether it complies with the requirements of the foregoing test.

# II. NEUTRALIZATION AND DECOLORIZATION 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 % phenolphtalein,
- pure hexane, AR,
- pure propan-2-ol of 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  $65\,^{\circ}$ 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 five minutes, keeping the temperature at  $65\,^{\circ}$ 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 to 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 (disapperance of the pink colouring in the phenolphtalein).

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 litre 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 one 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.

#### B. DECOLORIZATION OF NEUTRALIZED OIL

# 1. Apparatus

- round-bottomed flask, 250 ml, with three 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 to 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 to 30 millibars.

## 2. Procedure

Weigh about 100 g of neutralized oil in the three-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 analyzed 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 to 30 millibars is obtained and, maintaining a temperature of 90 °C, stir for 30 minutes at about 250 revolutions per minute.

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

#### ANNEX V

#### TEST FOR THE PRESENCE OF OIL FROM OLIVE RESIDUES IN OLIVE OILS

#### A. 'BELLIER' METHOD

#### 1. Apparatus

- round-bottomed flask, 100 ml, fitted with reflux condenser,
- pipette, 5 ml, graduated in tenths,
- heating system with which it is possible to attain a temperature of about 80 °C,
- thermometer graduated from 15 to 60 °C.

#### 2. Reagents

- aqueous alcoholic potassium hydroxide solution (42.5 g of KOH dissolved in 72 ml of distilled water, the volume then being brought up to 500 ml with 95° ethyl alcohol),
- ethyl alcohol solution of 70  $^{\circ}$  titre,
- solution of acetic acid in water, 1 + 2 (by volume), adjusted to strength so that 1.5 ml exactly neutralize 5 ml of the aqueous alcoholic potassium hydroxide solution in the presence of phenolphtalein.

#### 3. Sample preparation

Remove any moisture from the oil by decantation and filtration through paper, both operations being carried out at a temperature slightly higher than the melting point of any solid constituents which might have separated from the liquid.

#### 4. Procedure

Into the flask place about 1 ml of oil prepared as shown in Section 3. Add 5 ml of aqueous alcoholic potassium hydroxide solution. Fit the reflux condenser and bring to the boil for 10 minutes with occasional stirring. Allow to cool down to room temperature. Add 1.5 ml of diluted acetic acid and 50 ml of ethyl alcohol solution previously warmed to 50 °C. Mix by stirring, insert the thermometer and allow to cool, observing the appearance of the solution as soon as it has dropped to 45 °C. If a flocculent precipitate is formed at a temperature higher than 40 °C, the reaction is positive. If the characterizing flocculent precipitate does not materialize, keep the liquid at room temperature which must not be below 20 °C or above 22 °C, for at least 24 hours or, if necessary, for 48 hours. Observe the solution once again: the formation of a flocculent precipitate in suspension in the liquid also proves the reaction to be positive.

Reporting of results

The test for the presence of oil from olive residues (Bellier method) shall be reported as a positive or negative test.

# B. 'MODIFIED VIZERN' METHOD

The unsaponifiable matter of the oil to be analyzed is isolated and its behaviour in  $85\,^\circ$  alcohol studied under specified conditions.

#### 1. Apparatus

- round-bottomed alkali-resistant glass flask, 300 ml, fitted with reflux condenser,
- separating funnels, 500 or 1 000 ml,
- beakers, 300 and 250 ml,
- glass test-tube.

#### 2. Reagents

- alcoholic potassium hydroxide solution, 2N,
- petroleum ether,
- 50 % ethyl alcohol,
- 96° ethyl alcohol, checked by alcoholmeter,
- Hydrogen peroxide, 10 volume,
- 85° ethyl alcohol, checked by alcoholometer.

#### 3. Procedure

Into a 300 ml round-bottomed flask weigh approximately 5 g of the sample to be analyzed. Add 50 ml of 2N alcoholic potash solution. Fit the reflux condenser and bring to the boil gently. After an hour of heating, shake and cool to 30 to 35 °C; transfer to a separating funnel, using 50 ml of distilled water.

Rinse out the flask carefully with 50 ml of petroleum ether and repeat this operation several times. Transfer the petroleum ether into a separating funnel. Shake the contents vigorously for slightly more than a minute. After decantation, remove the aqueous layer by transferring it into a second separating funnel. Add another 50 ml of petroleum ether, shake the contents vigorously for just over a minute and allow to separate. Transfer the aqueous layer to a third separating funnel, adding another 50 ml of petroleum ether. Shake vigorously and allow to separate.

In a separating funnel, collect the ethereal extracts from the various extractions of unsaponifiable matter and wash at least three times with 50 % alcohol (50 ml each time) until the washing liquid is no longer alkaline to phenolphtalein.

Filter the unsaponifiable matter solution into a 300 ml round-bottomed flask, wash the filter with petroleum ether and remove the solvent by distillation. Add 10 ml of 96  $^{\circ}$  alcohol, warm moderately (to about 40  $^{\circ}$ C) and filter into a 100 ml beaker. Wash the 300 ml flask with 10 ml of 96  $^{\circ}$  alcohol and then filter into the beaker.

To the 100 ml beaker containing the alcoholic unsaponifiable matter extracts, add 5 ml of 10 volume hydrogen peroxide and heat on a water bath until completely evaporated. Add another 20 ml of 96  $^{\circ}$  alcohol and 5 ml of hydrogen peroxide and re-evaporate completely.

Withdraw the beaker from the water bath and add 20 ml of  $85\,^\circ$  alcohol. Warm gently on the water bath, being very careful not to lower the concentration by causing loss of alcohol. This is a very important factor which must not be overlooked.

Filter through paper, allow to cool and examine the behaviour of the solution at the end of an hour, and again at the end of four hours. If the solution is quite transparent at the end of an hour, the test is negative, i.e., there is no oil from olive residues. If the solution is cloudy at the end of an hour, repeat the observation four hours after that.

If the solution is still cloudy but contains no flocculence at the end of four hours, the test is still negative. On the other hand, if flocculence is observed, the test is positive and oil from olive residues is present.

Reporting of results

The test for the presence of oil from olive residues (modified Vizern method) shall be reported as a positive or a negative test.

Remarks

Olive oils give a transparent or, at the most, a milky solution throughout the test. Pure or blended oils from olive residues give rise to a characteristic flocculence which remains in suspension like small clouds and which finally precipitate after being allowed to stand for several hours.

#### ANNEX VI

#### SOAP TEST FOR DETECTION OF ALKALINITY

The principle of the method is the detection of alkali soaps through their action on bromophenol blue.

#### Reagents

- 0.1 % solution of bromophenol blue in 96 % (v/v) ethanol,
- freshly distilled acetone with a 2 % (v/v) water content.

This acetone with a 2 % water content must give a yellow or greenish yellow coloration in the presence of a few drops of the bromophenol blue solution.

#### **Apparatus**

— stoppered test-tube,  $150 \times 15$  mm.

#### Procedure

Into the stoppered test-tube pour 10 ml of acetone and one drop of bromophenol blue solution; the solution should turn yellow. If it does not, rinse the tube and its stopper with acetone until the blue coloration disappears. Pour 10 g of the oil into the tube, close it by means of its own stopper, shake and allow to settle. The upper acetone layer turning blue means the presence of soap.

#### Reporting of results

These shall be reported as positive or negative results.

#### ANNEX VII

# TEST FOR THE PRESENCE OF RE-ESTERIFIED OILS

The purpose of the method is to determine the composition of the fraction of fatty acids which are esterified at the 2-position ( $\beta$ - or internal position) of the glycerol in the oils or fats, as there is more palmitic acid in the  $\beta$ -position with re-esterified olive oils than with those which have not been re-esterified.

#### Principle

This method is based on the partial and specific hydrolysis of the triglycerides by pancreatic lipase with a preferential formation of 2-monoglycerides. This hydrolysis gives rise to a mixture containing diglycerides, 2-monoglycerides and some free fatty acids, in addition to unhydrolyzed triglycerides. This mixture is fractionated and the monoglycerides isolated by thin-layer chromatography. These monoglycerides are converted with methanol into methyl esters, which are analyzed by gas chromatography.

If the percentage of palmitic acid found at the 2-position of the triglycerides is higher than 2 %, the product analyzed is considered as containing added re-esterified oil.

# 1. Apparatus

- separating funnel, 500 ml,
- chromatography column of glass, 13 mm in diameter and 400 mm long, fitted with a sintered glass disc and a stopcock,
- wide-necked round-bottomed flask, 250 ml,
- round-bottomed flask, 100 ml,
- centrifuge tube with ground stopper, 10 ml,
- hypodermic syringe fitted with a fine needle, 1 ml,

- round-bottomed flask, 25 ml, with ground-jointed air-cooled condenser 1 m long,
- beaker, 50 ml,
- burette, 5 ml, graduated in 1/20 ml,
- thin-layer chromatography spreader with glass plates, 20 × 20 cm,
- microsyringe delivering 3 to 4 µl drops,
- thin-layer chromatography developing tank,
- rotary evaporator,
- oven controllable at 103 ± 2 °C,
- thermostat controllable between 30 and 45 °C to within ± 0.5 °C,
- vibrating electric shaker permitting vigorous shaking of the centrifuge tube,
- thin-layer chromatography spray,
- UV lamp for examining the chromatography plates,
- laboratory shaker of suitable design for the dispersion or mixing of heterogeneous substances,
- pH meter,
- paddle stirrer,
- stop-clock.

#### 2. Reagents

- aqueous sodium hydroxide solution, 12 % (m/v),
- solution of phenolphalein, 1 % (m/v), in ethanol, 95 % (v/v),
- peroxide-free diethyl ether,
- isopropyl or ethyl alcohol, analytical grade, 95% (v/v),
- neutral activated alumina, chromatography grade, of Brockmann I activity, activated recently for two hours at 206 °C, and stored in a dessicator,
- hexane, or if not available, petroleum ether (boiling range 30 to 50 °C), chromatography grade,
- formic acid, not less than 98 % (m/m),
- pancreatic lipase of suitable activity (Notes 1 and 2),
- buffer solution consisting of a 1M solution of tris-hydroxymethylaminomethane in water adjusted to pH 8 with 6N hydrochloric acid (potentiometric standard),
- aqueaus sodium cholate (enzyme grade) solution, 0·1 % (m/v),
- hydrogen chloride solution, 6N,
- developing solvent consisting of hexane (or, failing this, petroleum ether) mixed with diethyl ether and formic acid in the proportions of 70:30:1 (v/v/v),
- aqueous gum arabic solution, 10 % (m/v),
- aqueous calcium chloride (CaCl<sub>2</sub>) <sup>2</sup> solution, 22 % (m/v),
- alcoholic solution of 2',7'-dichlorofluorescein, 0.2 % (m/v), rendered slightly alkaline by the addition of 1N sodium hydroxide solution at the rate of one drop per 100 ml,
- powder silica with binder, thin-layer chromatography grade,
- aqueous sodium-cholate solution, 20 % (m/v),
- aqueous sodium hydroxide solution, 0:1N,
- neutralized vegetable oil.

# 3. Sample preparation

If the acidity of the sample is less than 3 %, direct neutralization with alumina as in Section 3.2.

If the acidity of the sample is higher than 3 %, alkali neutralization in the presence of solvent as in Section 3.1 followed by passage through alumina as in Section 3.2.

#### 3.1. Alkali neutralization in the presence of solvent

Into a 500 ml separating funnel pour about 10 g of raw oil, 100 ml of hexane or, failing this, petroleum ether, 50 ml of 95 % isopropyl or ethyl alcohol, a few drops of phenolphthalein solution and a sufficient quantity of 12 % sodium hydroxide to take up the free acidity of the oil and give  $0.3\,\%$  in excess. Shake vigorously for one minute, add 50 ml of distilled water, shake once more and allow to settle.

After separation, remove the lower layer containing the soaps. Discard any intermediate layers (mucilages or insoluble substances), wash the hexane solution of the neutralized oil with successive 25 or 30 ml portions of a 1:1 (v/v) solution of isopropyl or ethyl alcohol and distilled water until the pink phenolphtalein coloration disappears. Remove most of the hexane by vacuum distillation and take the oil to dryness at 30 to 40 °C under vacuum in a stream of pure nitrogen until solvent removal is complete.

#### 3.2. Passage through alumina

Prepare a suspension of 15 g of activated alumina in 50 ml of hexane, or petroleum ether, and pour it into the chromatography column of glass while stirring. Ensure that the alumina is evenly spread and allow the solvent to run down to 1 to 2 mm above the top of the absorbent. Into the column carefully pour a solution prepared about 15 minutes earlier by dissolving 5 g of oil in 25 ml of hexane, or petroleum ether, and collect in a 100 ml round-bottomed flask all the liquid issuing from the column.

Remove most of the solvent by vacuum distillation and then take the oil to dryness at 30 to 40 °C under vacuum in a stream of pure nitrogen until solvent removal is complete.

#### 4. Preparation of chromatography plates

Into wide-necked 250 ml round-bottomed flask place 30 g of prowdered silica with 60 ml of distilled water and stir until a fully homogeneous slurry is obtained. De-gas by keeping it under the vacuum of a water injector pump for one minute.

Spread the slurry in the usual way over the plates by means of the spreader, adjusting the layer thickness to 0.25 mm.

This quantity of slurry is sufficient for the preparation of five  $20 \times 20$  cm plates.

Allow the plates to air-dry for about 15 minutes and then dry them in the oven at 103  $\pm$  2 °C for two hours.

Store the plates so prepared in a dessicator.

#### Procedure

#### 5.1. Pancreatic lipase bydrolysis

Into a 10 ml centrifuge tube weigh about 0.1 g of prepared sample.

Add 20 mg of lipase and 2 ml of buffer solution. Stir carefully and with appropriate precautions then add 0.5 ml of 0.1 % sodium cholate solution and 0.2 ml of calcium chloride solution. Close the tube with its ground stopper, shake cautiously (avoid wetting the stopper) and place the tube immediately in a thermostat adjusted to  $40 \pm 0.5$  °C shaking for exactly one minute.

Remove the tube from the thermostat and shake it vigorously for exactly two minutes.

Cool immediately under running water and add 1 ml of 6N hydrogen chloride solution and 1 ml of diethyl ether. Stopper and shake vigorously. Allow to settle and sample the upper organic layer with a syringe.

## 5.2. Thin-layer chromatography separation of the monoglycerides

On a chromatography plate, about 1.5 cm from its bottom edge, deposit the extract in a continuous uniform line with the object of obtaining as fine a base line as possible.

Insert the plate in a well-saturated developing tank and develop with the developing solvent up to about 1 cm from the top edge. The development of the plate must take place at a temperature of about  $20\,^{\circ}$  C.

Air-dry the plate at the tank temperature and spray it with the 2', 7',-dichlorofluorescein solution. Identify the monoglycerides band ( $R_i$  = about 0·035) under UV light; remove them with a metal spatula (avoid removing any compounds remaining on the base line) and place the silica in the 25 ml round-bottomed methylation flask.

Convert the monoglycerides into their methyl esters by direct treatment of the previously collected silicy in accordance with the universal method for the preparation of the methyl esters of fatty acids mentioned in Section 7.3, then perform the gas chromatography of the esters in accordance with the method indicated in Section 7.4.

On the same sample determine the composition of the total fatty acids, comparison of which with that of the fatty acids at the 2-position is useful for the interpretation of the results obtained.

# 6. Reporting of results

Calculate the composition of the 2-position fatty acids as a percentage to one decimal place (Note 3).

#### 7. Notes

#### 7.1. Lipase activity test

In a suitable mixer prepare an oil emulsion by stirring a mixture of 165 ml of 10 % gum arabic solution, 15 g of crushed ice and 20 ml of an already neutralized oil for about 10 minutes.

Into a 50 ml beaker place 10 ml of this emulsion, 0.3 ml of 20 % sodium cholate solution and 20 ml of distilled water in succession.

Place the beaker in a thermostat controlled to  $37 \pm 0.5$  °C and insert into the beaker the electrodes of a pH pH meter and a paddle stirrer.

From a 5 ml burette add some 0·1N sodium hydroxide solution in drops until a pH of 8·5 is reached.

Add an appropriate volume of aqueous lipase powder suspension (see above). As soon as the pH meter indicates a pH of 8·3, start the stop-clock and add 0·1N sodium hydroxide solution at the necessary rate to maintain the pH at the value of 8·3. Note the volume of alkali solution consumed each minute.

Plot the data obtained in a system of coordinate axes, plotting times as abscissae and ml of alkali solution consumed in keeping the pH constant as ordinates. The resultant graph must be a straight line.

The lipase suspension referred to in the previous section is a 1 % suspension by weight in water. For each test take the necessary quantity of this suspension to ensure that about 1 ml of alkali solution is consumed in four to five minutes. This result is usually obtained with 1 to 5 mg of powder.

A lipase unit is defined as the quantity which liberates 10 μ -equivalents of acid per minute.

If A is the activity of the powder used, measured in lipase units per mg, then:

$$A = \frac{V \times 10}{m}$$

where: V = number of ml of 0.1N sodium hydroxide solution per minute, calculated from the graph and

m = mass of the test portion of powder, in mg.

The lipase used must have an activity of not less than 0.8 and not more than two units per mg.

#### 7.2. Lipase preparation

Lipases with a satisfactory lipase activity are available from trade sources. It is also possible to prepare it in the laboratory as follows:

Chill 5 kg of pig pancreas down to 0 °C, remove all surrounding fat and connective tissues and triturate in a mill with cutting blades until a fluid paste is obtained. Stir this paste in the cold state for four to six hours with 2.5 litres of anhydrous acetone, then centrifuge. Extract the residue a further three times with the same volume of acetone, twice with a 1:1 (v/v) mixture of acetone and diethyl ether and twice with diethyl ether.

Dry the residue for 48 hours under a vacuum to obtain a stable powder which must be stored in the refrigerator.

# 7.3. General method of preparing the methyl esters fatty acid

In accordance with the method set out in Section II of Annex VI to Commission Regulation (EEC) No 72/77 of 13 January 1977 amending Regulation (EEC) No 1470/68 on the drawing and reduction of samples and the determination of oil content, impurities and moisture in oil seeds (1).

#### 7.4. Gas chromatography of the methyl esters of the fatty acids

In accordance with the method set out in Section III of Annex VI to Commission Regulation (EEC) No 72/77.

#### ANNEX VIII

# TEST FOR THE PRESENCE OF OTHER OILS IN OLIVE OILS: ANALYSIS OF THE STEROL FRACTION OF THE OIL OR FAT

#### Principle

Gas chromatography of sterols prepared by means of thin-layer chromatography from unsaponifiable matter carefully dried.

#### Apparatus

- 1. Thin-layer chromatography apparatus, including in particular, four glass plates of size  $20 \times 20 \times 0.4$  cm, two of size  $20 \times 5 \times 0.4$  cm and one 0.1 ml microsyringe.
- 2. 50 ml beaker.
- 3. Porous filters, porosity 3, diameter 15 mm.
- 4. 100 ml round-bottomed flask.
- 5. 10 ml conical-bottomed centrifuge tube, fitted with ground glass stopper.
- 6. 1 ml graduated pipettes.
- 7. Gas chromatography apparatus equipped with a flame-ionization detector and a silver or glass injector or a system of direct injection into the column and coupled to a recorder.
- 8. A U-shaped or spiral glass or stainless steel gas chromatography column, 1 to 2 m long and 3 to 4 mm in internal diameter, with a stationary phase of silicone rubber (Methyl type) (1), stable up to at least 300 °C, impregnating a calcined diatomaceous earth to the extent of not less than 2 % and not more than 4 %, acid washed and silanized and of a particle size analysis of 80 to 100 mesh or 100 to 120 mesh.

Note: Glass is recommended, as some types of stainless steel can give rise to erroneous results by deteriorating the sterols.

9. 5 or 10 µl microsyringe.

## Reagents

- 1. Chloroform, chromatography grade.
- 2. Benzene, chromatography grade.
- 3. Heptane.
- 4. Silica gel (e.g., Kieselgel G).
- 5. Thin-layer chromatography reference solution composed of 5 % cholesterol in chloroform.
- 6. Acetone, chromatography grade.
- 7. 0.1 % absolute alcohol solution of 2',7'-dichlorofluorescein sodium salt.
- 8. Pyridine.
- 9. Hexamethyldisilazane.
- 10. Trimethylchlorosilane.
- 11. Sensitivity test solution composed of 1 mg of cholesterol per ml of n-pentane.
- 12. Peak-resolution test solution composed of 0.9 mg of colza oil phytosterols and 0.1 mg of cholesterol per ml of n-pentane. The sterols must be freshly prepared in accordance with the procedure described in Section B of the Procedure.
- 13. Reference test solution composed of 1 mg of sunflower seed oil phytosterols, freshly prepared as described in Section B of the Procedure, per ml of n-pentane.

# Preparation of the chromatography plates

On the spreader place one plate 20  $\times$  5  $\times$  0.4 cm, four plates 20  $\times$  20  $\times$  0.4 cm and one plate 20  $\times$  5  $\times$  0.4 cm, in that order.

<sup>(1)</sup> E.g., SE 30.

In a wide-necked 500 ml round-bottomed flask place 40 g of silica gel and 80 ml of water. Stir with a glass rod or, if available, a mechanical glass stirrer until a homogeneous suspension results. Remove any gas by creating a vacuum, using a water injector pump; for at least one minute. Then transfer the suspension to the spreader, adjust the thickness to 0.5 mm and coat the plates uniformly. Allow the plates to air-dry for about 15 minutes and then dry off in an oven at 105 °C for two hours. Store the plates so prepared in an evacuated dessicator.

#### **PROCEDURE**

#### A. Preparation of the unsaponifiable matter

#### Foreword

The unsaponifiable matter is defined as the substances soluble in the fat which are insoluble in water after saponification but soluble in the solvent used for the determination. It includes lipids of natural origin such as sterols, alcohols and hydrocarbons as well as any foreign organic matter which may be present that is not volatile at 100 °C (mineral oils). Light petroleum or diethyl ether is used as a solvent but in most cases the results will differ according to the solvent selected and, generally, the use of diethyl ether will give a higher result. In the case of olive oil, petroleum ether (light petroleum) has been adopted as the solvent to be used in view of the temperature conditions in which the majority of laboratory analyses are carried out.

## Light petroleum method

#### Apparatus

- 150 ml flask fitted with a reflux condenser,
- 500 ml separating funnels,
- oven regulated at 103 °C (± 2 °C).

#### Reagents

Approximately 2N KOH solution in ethanol (dissolve 120 g potassium hydroxide in 95 % v/v ethanol and make up to 1 litre). The reagent must not be darker in colour than straw yellow.

Light petroleum (B.p. 40 to 60  $^{\circ},$  bromine value 1), free from residue.

#### Procedure

Weigh about 5 g of fat to within 0.01 g into the flask.

Add 50 ml of approximately 2N ethanolic KOH solution. Attach the condenser. Boil gently for an hour.

Stop heating. Add 50 ml of distilled water through the top of the condenser and shake.

After cooling, transfer to a separating funnel and rinse the flask several times using 50 ml of light petroleum in all.

Shake vigorously for a minute.

Let it stand until there is complete separation of the two phases, and draw off the soap solution into a second separating funnel. If an emulsion should form, break it by adding small quantities of ethanol or concentrated potassium hydroxide solution.

Extract the soap solution twice more, using 50 ml light petroleum each time.

Combine the three ethereal extracts in one separating funnel, wash three times with 50 ml portions of 50 % (v/v) ethanol.

Pour off the petroleum extract quantitatively, if necessary in instalments through the top of the funnel into a tared 250 ml flask. Rinse the funnel with small quantities of light petroleum.

Remove the solvent by careful heating under vacuum and dry the residue under vacuum at a temperature not exceeding  $50\,^{\circ}$ C in order to avoid undesirable oxidative changes.

#### B. Separation of the sterol fraction by means of thin-layer chromatography

Into the development tank pour some of the 85:15 (v/v) mixture of n-heptane and acetone or 95:5 (v/v) mixture of benzene and acetone to a depth of about 1 cm; close with the lid and allow to stand for at least three hours so that liquid and vapour can reach equilibrium. It is also recommended that strips of filter paper dipping into the eluant be attached to the inside surface of the tank. The advantage this precaution offers is that it reduces by about a third the time taken for the front of the liquid to migrate and causes the compounds to elute more uniformly.

Meanwhile, prepare a 5 % solution in chloroform of the unsaponifiable matter extracted with petroleum ether. Take about 0·3 ml of this solution and deposit it, using a 0·1 ml microsyringe, in a continuous and uniform stripe on the chromatography plate at about 1·5 cm from the bottom edge in such a way as to give as thin a base line as possible. In accordance with standard practice, deposit a few  $\mu$ l of the reference solution containing cholesterol at one end of the plate in order to ascertain the  $R_t$  value of the sterol fraction.

Place the plate in the development tank prepared as described above. The room temperature must be about 20 °C. Close the tank with the lid and develop until the solvent front has reached a level about 1 cm from the top edge of the plate. Withdraw the plate from the tank and allow the solvent to evaporate in a stream of warm nitrogen.

Develop by spraying the plate uniformly and carefully with the alcoholic 2',7'-dichlorofluorescein sodium salt solution. By examination of the plate under ultraviolet light, the position of the sterols is determined by means of alignment on the spot of cholesterol derived from the reference solution. Collect the sterol band by scraping it away with a metal spatula. Place the separated silica gel in a 50 ml beaker with 15 ml of hot chloroform, shake and transfer the whole of the silica gel to a porous filter and filter.

Wash the filter three times, each time with a 15 ml portion of hot chloroform, collecting the filtrage in a 100 ml round-bottomed flask.

Evaporate the chloroform solution down to 4 to 5 ml and pour it into a previously tared centrifuge tube fitted with a ground stopper. Bring to dryness by evaporating the solvent off with gentle heating in a stream of nitrogen and weigh the resultant sterol fraction.

# C. Gas chromatography analysis of the sterols

# 1. Preparation of the trimethylsilylethers (TMS)

Into the centrifuge tube add for each mg of sterol 0.02 ml of silanization reagent composed of a 9:3:1 (v/v/v) mixture of pryridine, hexamethyldisilazine and trimethylchlorosilane, being careful to exclude every trace of moisture. Place the tube in a dessicator for about 30 minutes, insert the stopper and centrifuge for a few minutes. Sample the remaining solution for subsequent analysis.

### 2. Conditions for a gas chromatography analysis

Column temperature: 220 to 250 °C.

If heated separately, the injection system must be maintained at a temperature 20 to 40 °C above the column temperature. Nitrogen flowrate: 30 to 60 ml/min. Disconnect the detector and equilibrate any new column under these conditions for 16 to 24 hours. Reconnect the detector, light the flame and adjust the hydrogen, oxygen or air flowrates to give a suitable flame height and sensitivity. Switch on the recording apparatus and see that the paper unwinds at the right speed; adjust the zero and sensitivity control. When the base line is stable, the apparatus is ready for use.

#### 3. Sensitivity test

Take 5 ml of sensitivity test solution, evaporate off the solvent and treat it as shown in Section 1; inject 0.1 to 0.2  $\mu$ l of the TMS solution so prepared. The cholesterol peak must appear alone on the chromatogram.

Adjust the sensitivity control so as to use approximately the full scale of the recorder.

#### 4. Peak resolution test

Take 5 ml of so prepared solution (TMS). Inject 0·1 to 2 ml of resolution-test. Evaporate off the solvent and treat as described in Section 1.

Inject 0.1 to 0.2 µl of the TMS solution so prepared.

The cholesterol, brassiscasterol, campesterol and  $\beta$ -sitosterol peaks will appear on the chromatogram. Measure the retention distances (distances from the point of injection to the points of maximum peak height),  $d_{CH}$  in respect of cholesterol,  $d_B$  in respect of brassiscasterol,  $d_C$  in respect of campesterol and  $d_S$  in respect of  $\beta$ -sitosterol, and the widths of the peaks at their base (retention lengths between the points at which the tangents to the points of inflection located on the front side and rear side of the peak intersect the base line),  $\phi_{CH}$  in respect of cholesterol and  $\phi_B$  in respect of brassicasterol. Peak resolution, expressed by the formula:

$$PR = 2 \left( \frac{d_B - d_{CH}}{\omega_B + \omega_{CH}} \right)$$

must equal at least 1.

Calculate the relative retention times (cholesterol = 1.00) for brassicasterol, campesterol and  $\beta$ -sitosterol.

#### 5. Reference test

Take 5 ml of reference test solution, evaporate off the solvent, treat as described in Section 1 and inject 0.1 to 0.2  $\mu$ l of the TMS solution so prepared. The campesterol, stigmasterol,  $\beta$ -sitosterol and  $\Delta 7$ -stigmasterol peaks will appear on the chromatogram.

Measure the peak retention distances,  $d_C$  in respect of campesterol,  $d_{ST}$  in respect of stigmasterol,  $d_S$  in respect of  $\beta$ -sitosterol and  $d_{ST-7}$  in respect of  $\Delta 7$ -stigmasterol.

Calculate the relative retention times which are approximately:

cholesterol	1.0
brassicasterol	1.1
campesterol	1.3
stigmasterol	1.4
β-sitosterol	1.6 (1)
$\Delta$ 7-stigmastenol	1.8 (2)

## 6. Analysis

Inject 0·1 to 0·2  $\mu$ l of the sterol TMS solution to be analyzed and record the chromatogram.

# D. Reporting of results

In interpreting the composition of the sterol fraction analyzed, ignore any peaks having different retention times from those determined experimentally for the six sterols mentioned above.

The percentage  $\beta$ -sitosterol content is given by the formula:

Area of the β-sitosterol peak
Sum of the areas of the six sterol peaks  $\times$  100

The  $\beta$ -sitosterol content must not be less than 93 % of the total sterol percentage composition.

<sup>(</sup>¹) If other sterols, e.g., Δ5-avenasterol, have the same retention volume under these conditions as β-sitosterol, they shall be counted as though they were β-sitosterol.

<sup>(2)</sup> If other sterols have the same retention volume under these conditions as Δ7-stigmastenol; they shall be counted as though they were Δ7-stigmastenol.

#### ANNEX IX

#### OIL CONTENT OF OLIVE RESIDUE

#### Apparatus

- suitable extraction apparatus fitted with a 200 to 250 ml round-bottomed flask,
- electrically heated bath (e.g., sand bath, water bath) or hotplate,
- analytical balance,
- oven regulated to a maximum of 80 °C,
- electrically heated oven fitted with a thermostatic device regulated to 103  $\pm$  2 °C and one that can be swept with a stream of air or operated at reduced pressure,
- mechanical mill, easy to clean, and one that allows the olive residues to be ground without a rise in their temperature or any appreciable alteration in their content of moisture, volatile matter or substances extractable with hexane,
- extraction thimble and cotton wool or filter paper from which substances extractable with hexane have already been removed,
- dessicator,
- sieve with 1 mm diameter apertures,
- small particles of previously dried pumice stone.

#### Reagent

Normal hexane, technical grade, which must leave a residue of less than 0.002 g per 100 ml, on complete evaporation.

#### **PROCEDURE**

# Preparation of the test sample

If necessary, use the mechanical mill, which has previously been properly cleaned, to grind the laboratory sample in order to reduce it to particles that can pass completely through the sieve.

Use about one twentieth of the sample to complete the process of cleaning the mill, discard the ground material, grind the remainder and collect, mix carefully and analyze without delay.

#### Test portion

As soon as the grinding operation has been completed, weigh out about 10 g of the sample to the nearest 0.01 g for testing.

#### Preparation of the extraction thimble

Place the test portion in the thimble and plug with cotton wool. If a filter paper is used, envelope the test portion in it.

# Preliminary drying

If the olive residues are very moist (i.e., moisture and volatile matter content more than 10 %), carry out preliminary drying by placing the loaded thimble (or filter paper) in the oven heated for an appropriate time at not more than  $80\,^{\circ}\text{C}$  in order to reduce the moisture and volatile matter content to less than  $10\,^{\circ}\text{M}$ .

#### Preparation of the round-bottomed flask

Weigh to the nearest 1 mg the flask containing one or two particles of pumice stone, previously dried in the stove at  $103 \pm 2$  °C and then cooled in a dessicator for not less than one hour.

#### Initial extraction

Into the extraction apparatus insert the thimble (or filter paper) containing the test portion. Pour into the flask the requisite quantity of hexane. Fit the flask to the extraction apparatus and place the whole on the electrically heated bath. Adjust the rate of heating in such a way that the reflux rate is not less than three drops per second (moderate, not violent boiling). After four hours extraction, allow to cool. Remove the thimble from the extraction apparatus and place it in a stream of air in order to drive off most of the impregnating solvent.

#### Second extraction

Tip the contents of the thimble into the micro-grinder and grind as finely as possible. Return the ground mixture to the thimble without loss and place it back in the extraction apparatus.

Continue the extraction for a further two hours, using the same round-bottomed flask containing the initial extract.

The resultant solution in the extraction flask must be clear. If not, filter it through a filter paper and wash the original flask and the filter paper several times with hexane. Collect the filtrate and the washing solvent in a second round-bottomed flask which has been dried and tared to the nearest 1 mg.

#### Removal of solvent and weighing of extract

Remove the greater part of the solvent by distillation on an electrically heated bath. Remove the last traces of solvent by heating the flask in the oven at  $103 \pm 2$  °C for 20 minutes. Assist the elimination process either by blowing in air, or preferably an inert gas, at intervals or by using reduced pressure

Leave the flask in a dessicator to cool for at least one hour and weigh to the nearest 1 mg.

Heat again for 10 minutes under the same conditions, cool in a dessicator and reweigh.

The difference between the two weighings shall not exceed 10 mg. If it does, heat again for periods of 10 minutes followed by cooling and weighing until the weight difference is 10 mg or less. Note the last weight of the flask.

Carry out duplicate determinations on the test sample.

#### **EXPRESSION OF RESULTS**

#### Method of calculation and formula

(a) The extract expressed as a percentage by mass of the product as received is equal to:

$$S = m_1 \times \frac{100}{m_0}$$

where: S is the percentage by mass of extract of the product as received,

 $m_{\rm o}$  is the mass, in grams, of the test portion,

m<sub>1</sub> is the mass, in grams, of the extract after drying.

Take as the result the arithmetic mean of the duplicate determinations, providing the repeatability conditions are satisfied.

Express the result to the first decimal place.

(b) The extract is expressed on a dry matter basis by using the formula:

S 
$$\times \frac{100}{100 - U}$$
 = oil percentage of extract on a dry basis

where: S is the percentage of extract by means of the product as received (see (a)),
U is its moisture and volatile matter content.

#### Repeatability

The difference between the duplicate determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0.2 g of hexane extract per 100 g of sample.

If this condition is not satisfied, repeat the analysis on two other test portions. If in this case too the difference exceeds 0·2 g, take as the result the arithmetic mean of the four determinations.

#### COMMISSION REGULATION (EEC) No 1059/77

of 18 May 1977

re-establishing the levying of the customs duties on other kinds of leather, other, falling within subheading 41.05 B II, originating in Yugoslavia to which the preferential tariff arrangements set out in Council Regulation (EEC) No 3021/76 apply

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 3021/76 of 13 December 1976 establishing preferential tariffs in respect of certain products originating in developing countries (1), and in particular Article 4 (2) thereof,

Whereas Article 1 (3) and (4) of that Regulation provides that the customs duties may, for each category of products, be suspended up to a Community ceiling, expressed in units of account, which will be equal — with the exception of certain products the value of the ceilings for which is given in Annex A of the Regulation in question — to the sum arrived at by adding together the value of the products in question imported cif into the Community in 1974 and coming from countries and territories covered by those arrangements, but not including products coming from countries and territories already covered by various preferential tariff arrangements established by the Community, and 5 % of the value of 1974 cif imports coming from other countries and from countries and territories already covered by such arrangements; however the ceiling resulting from the sum of this addition may in no case exceed 172.5 % of that resulting from the substitution of the year 1971 for the year 1974 in the first term of the addition and of the year 1972 for the year 1974 in the second term of the addition;

Whereas, having regard to that ceiling, the amounts for products originating in any one of the countries or territories listed in Annex B to that Regulation should be within a maximum Community amount representing 50 % of that ceiling, with the exception of certain products for which the maximum amount is to be reduced to the percentage indicated in Annex A to that Regulation; whereas, for these products, this reduced percentage will be 20 %;

Whereas Article 2 (2) of that Regulation provides that the levying of customs duties may be re-established at any time in respect of imports of the products in question originating in any of the said countries or territo-

n the substitution of the year 1971 for in the first term of the addition and of the year 1974 in the second term of the year 1971 for following products, imported into the Community and originating in Yugoslavia:

Whereas, in respect of other kinds of leather, other, falling within subheading 41.05 B II, the ceiling, calculated as indicated above, should be 9 540 000 units of account, and therefore the maximum amount is 1 908 000 units of account; whereas on 11 May 1977, the amounts of imports into the Community of other kinds of leather, other, falling within subheading 41.05 B II, originating in Yugoslavia a country covered by preferential tariff arrangements, reached that maximum amount; whereas, bearing in mind the objectives of Regulation (EEC) No 3021/76 which provides that maximum amounts should not be exceeded, customs duties should be re-established in respect of the products in question in relation to

ries — with the exception of those listed in Annex C

of the same Regulation - once the relevant Commu-

nity amount has been reached;

HAS ADOPTED THIS REGULATION:

Yugoslavia,

#### Article 1

As from 27 May 1977, the levying of customs duties,

suspended in pursuance of Council Regulation (EEC)

CCT heading No	Description of goods
41.05	Other kinds of leather, except leather falling within heading No 41.06, 41.07 or 41.08:  B. Other:  II. Other

# Article 2

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

<sup>(1)</sup> OJ No L 349, 20. 12. 1976, p. 23.

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

Done at Brussels, 18 May 1977.

For the Commission
Étienne DAVIGNON

Member of the Commission

# **COMMISSION REGULATION (EEC) No 1060/77**

of 18 May 1977

re-establishing the levying of the customs duties on plaiting materials (other than products falling within heading No 46.01) bound together in parallel strands etc., falling within heading No 46.02, originating in South Korea to which the preferential tariff arrangements set out in Council Regulation (EEC)

No 3021/76 apply

THE COMMISSION OF THE EUROPEAN COMMUNITIES.

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 3021/76 of 13 December 1976 establishing preferential tariffs in respect of certain products originating in developing countries (1), and in particular Article 4 (2) thereof,

Whereas Article 1 (3) and (4) of that Regulation provides that the customs duties may, for each category of products, be suspended up to a Community ceiling, expressed in units of account, which will be equal — with the exception of certain products the value of the ceilings for which is given in Annex A of the Regulation in question — to the sum arrived at by adding together the value of the products in question imported cif into the Community in 1974 and coming from countries and territories covered by those arrangements, but not including products coming from countries and territories already covered by various preferential tariff arrangements established by the Community, and 5 % of the value of 1974 cif imports coming from other countries and from countries and territories already covered by such arrangements; however the ceiling resulting from the sum of this addition may in no case exceed 172.5 % of that resulting from the substitution of the year 1971 for the year 1974 in the first term of the addition and of the year 1972 for the year 1974 in the second term of the addition;

Whereas, having regard to that ceiling, the amounts for products originating in any one of the countries or territories listed in Annex B to that Regulation should be within a maximum Community amount representing 50 % of that ceiling, with the exception of certain products for which the maximum amount is to be reduced to the percentage indicated in Annex A to that Regulation; whereas, for these products, this reduced percentage will be 30 %;

Whereas Article 2 (2) of that Regulation provides that the levying of customs duties may be re-established at any time in respect of imports of the products in question originating in any of the said countries or territories — with the exception of those listed in Annex C of the same Regulation — once the relevant Community amount has been reached;

Whereas, in respect of plaiting materials (other than products falling within heading No 46.01) bound together in parallel strands etc., falling within heading No 46.02, the ceiling, calculated as indicated above, should be 4 823 000 units of account, and therefore the maximum amount is 1 446 900 units of account: whereas on 11 May 1977, the amounts of imports into the Community of plaiting materials (other than products falling within heading No 46.01) bound together in parallel strands etc., falling within heading No 46.02, originating in South Korea a country covered by preferential tariff arrangements, reached that maximum amount; whereas, bearing in mind the objectives of Regulation (EEC) No 3021/76 which provides that maximum amounts should not be exceeded, customs duties should be re-established in respect of the products in question in relation to South Korea,

HAS ADOPTED THIS REGULATION:

# Article 1

As from 27 May 1977, the levying of customs duties, suspended in pursuance of Council Regulation (EEC) No 3021/76, shall be re-established in respect of the following products, imported into the Community and originating in South Korea:

CCT heading No	Description of goods
46.02	Plaiting materials (other than products falling within heading No 46.01) bound together in parallel strands or woven in sheet form, including matting, mats and screens; straw envelopes for bottles

# Article 2

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

<sup>(1)</sup> OJ No L 349, 20. 12. 1976, p. 23.

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

Done at Brussels, 18 May 1977.

For the Commission
Étienne DAVIGNON

Member of the Commission

# COMMISSION REGULATION (EEC) No 1061/77

of 18 May 1977

re-establishing the levying of customs duties on terry towelling and similar terry fabrics of cotton, falling within heading No 55.08, originating in Yugoslavia to which the preferential tariff arrangements set out in Council Regulation (EEC)

No 3022/76 apply

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 3022/76 of 13 December 1976 opening, allocating and providing for the administration of Community tariff preferences for textile products originating in developing countries and territories (1), and in particular Article 4 thereof,

Whereas Article 2 (1) to (3) of that Regulation provides that customs duties may, for each category of products, be suspended up to a Community ceiling which is indicated, for each of the products listed in Annex B, under (a) in column 5; whereas, only the products originating in the countries and territories listed in Annex D of the Regulation in question, other than those specified under (b) in column 4 of Annex B, in respect of the corresponding products may be charged against that ceiling; whereas, having regard to that ceiling, the amounts for products originating in any one of the countries listed in Annex D should be within a maximum amount representing 50 % of that ceiling;

Whereas Article 3 (2) of that Regulation provides that the levying of customs duties may be re-established at any time in respect of imports of the products in question originating in any of the said countries and territories, with exception of countries listed in its Annex E, once the relevant Community maximum amount has been reached;

Whereas, in respect of terry towelling and similar terry fabrics of cotton, the ceiling, calculated as indicated above, should be 83 tonnes, and therefore the maximum amount is 42 tonnes; whereas on 16 May 1977 the amounts of imports into the Community of terry towelling and similar terry fabrics of cotton, originating in Yugoslavia a country covered by preferential tariff arrangements, reached that maximum amount; whereas, bearing in mind the objectives of Regulation (EEC) No 3022/76 which provides that maximum amounts should not be exceeded, customs duties should be re-established in respect of the products in question in relation to Yugoslavia,

#### HAS ADOPTED THIS REGULATION:

# Article 1

As from 27 May 1977, the levying of customs duties, suspended in pursuance of Council Regulation (EEC) No 3022/76, shall be re-established in respect of the following products, imported into the Community and originating in Yugoslavia:

CCT heading No	Description of goods
55.08	Terry towelling and similar terry fabrics of cotton

#### Article 2

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

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

Done at Brussels, 18 May 1977.

For the Commission
Étienne DAVIGNON
Member of the Commission

# COMMISSION REGULATION (EEC) No 1062/77

of 23 May 1977

# fixing the import levies on cereals and on wheat or rye flour groats and meal

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 2727/75 of 29 October 1975 on the common organization of the market in cereals (1), as last amended by Regulation (EEC) No 3138/76 (2), and in particular Article 13 (5) thereof,

Whereas the import levies on cereals, wheat and rye flour, and wheat groats and meal were fixed by Regulation (EEC) No 1882/76 (3) and subsequent amending Regulations;

Whereas it follows from applying the provisions contained in Regulation (EEC) No 1882/76 to the offer prices and today's quotations known to the Commission that the levies at present in force should be altered as shown in the Annex to this Regulation,

HAS ADOPTED THIS REGULATION:

#### Article 1

The import levies to be charged on products listed in Article 1 (a), (b) and (c) of Regulation (EEC) No 2727/75 are hereby fixed as shown in the table annexed to this Regulation.

#### Article 2

This Regulation shall enter into force on 24 May

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

Done at Brussels, 23 May 1977.

For the Commission Finn GUNDELACH Vice-President

<sup>(</sup>¹) OJ No L 281, 1. 11. 1975, p. 1. (²) OJ No L 354, 24. 12. 1976, p. 1. (³) OJ No L 206, 31. 7. 1976, p. 62.

**ANNEX** 

# to the Commission Regulation of 23 May 1977 fixing the import levies on cereals and on wheat or rye flour groats and meal

(n.a	

CCT heading Description of goods No		Levies		
10.01 A	Common wheat, and meslin	94.36		
10.01 B	Durum wheat	140.83 (1) (5)		
10.02	Rye	73.24 (6)		
10.03	Barley	56.19		
10.04	Oats	56.42		
10.05 B	Maize, other than hybrid maize for			
	sowing	67·33 (²) (³)		
10.07 A	Buckwheat	0		
10.07 B	Millet	74.25 (4)		
10.07 C	Grain sorghum	75.36 (4)		
10.07 D	Canary seed; other cereals	0 (5)		
11.01 A	Wheat or meslin flour	144-13		
11.01 B	Rye flour	114.54		
11.02 A I a)	Durum wheat groats and meal	228.81		
11.02 A·I b)	Common wheat groats and meal	154.13		

<sup>(1)</sup> Where durum wheat originating in Morocco is transported directly from that country to the Community, the levy is reduced by 0-50 u.a/tonne.

<sup>(2)</sup> Where maize originating in the ACP or OCT is imported into the French overseas departments, the levy is reduced by 6 u.a./tonne as provided for in Regulation (EEC) No 706/76.

<sup>(3)</sup> Where maize originating in the ACP or OCT is imported into the Community the levy is reduced by 1-50 u.a./tonne.

<sup>(4)</sup> Where millet and sorghum originating in the ACP or OCT is imported into the Community the levy is reduced by 50 %.

<sup>(5)</sup> Where durum wheat and canary seed produced in Turkey are transported directly from that country to the Community, the levy is reduced by 0.50 u.a./tonne.

<sup>(6)</sup> The import levy charged on rye produced in Turkey and transported directly from that country to the Community is laid down in Council Regulation (EEC) No 2754/75 and Commission Regulation (EEC) No 2622/71.

# COMMISSION REGULATION (EEC) No 1063/77

of 23 May 1977

#### fixing the premiums to be added to the import levies on cereals, flour and malt

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 2727/75 of 29 October 1975 on the common organization of the market in cereals (1), as last amended by Regulation (EEC) No 3138/76 (2), and in particular Article 15 (6) thereof,

Whereas the premiums to be added to the levies on cereals and malt were fixed by Regulation (EEC) No 1883/76 (3) and subsequent amending Regulations;

Whereas, on the basis of today's cif prices and cif forward delivery prices, the premiums at present in force, which are to be added to the levies, should be

altered as shown in the tables annexed to this Regulation.

#### HAS ADOPTED THIS REGULATION:

#### Article 1

The scale of the premiums to be added, pursuant to Article 15 of Regulation (EEC) No 2727/75, to the import levies fixed in advance in respect of cereals and malt is hereby fixed as shown in the tables annexed to this Regulation.

#### Article 2

This Regulation shall enter into force on 24 May 1977.

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

Done at Brussels, 23 May 1977.

For the Commission Finn GUNDELACH Vice-President

<sup>(1)</sup> OJ No L 281, 1. 11. 1975, p. 1.

<sup>(</sup>²) OJ No L 354, 24. 12. 1976, p. 1. (³) OJ No L 206, 31. 7. 1976, p. 64.

# ANNEX

# to the Commission Regulation of 23 May 1977 fixing the premiums to be added to the import levies on cereals, flour and malt

# A. Cereals and flour

(u.a/tonne)

CCT heading	Description of goods	Current	1st period	2nd period	3rd period
No		5	. 6	/	8
0.01 A	Common wheat, and meslin	0	1.00	1.00	1.00
0.01 B	Durum wheat	0	0	0	0
0.02	Rye	0	0	0	0
0.03	Barley	0	0	0	0.74
0.04	Oats	0	0.74	0.74	0.74
0.05 B	Maize, other than hybrid maize for sowing	0	0	0	0.37
0.07 A	Buckwheat	0	0	0	0
0.07 B	Millet	0	0	0	0
0.07 C	Grain sorghum	0	0	0	1-11
0.07 D	Other cereals	0	0	0	0
1.01 A	Wheat or meslin flour	0	1.40	1:40	1.40
		1	1	1	

# B. Malt

(u.a/tonne)

CCT heading No	Description of goods	Current - 5	lst period 6	2nd period 7	3rd period 8	4th period 9
11.07 A I (a)	Unroasted malt, obtained from wheat, in the form of flour	0	1.78	1.78	1.78	1.78
11.07 A I (b)	Unroasted malt, obtained from wheat, other than in the form of flour	0	1.33	1.33	1.33	1.33
11.07 A II (a)	Unroasted malt, other than that obtained from wheat, in the form of flour	0	0	0	1.32	1.32
11.07 A II (b)	Unroasted malt, other than that obtained from wheat, other than in the form of flour	0	0	0	0.98	0.98
11.07 B	Roasted malt	0	0	0	1.15	1.15

# COMMISSION REGULATION (EEC) No 1064/77

#### of 23 May 1977

# fixing the import levies on white sugar and raw sugar

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Regulation (EEC) No 3330/74 of 19 December 1974 on the common organization of the market in sugar (1), as last amended by Regulation (EEC) No 3138/76(2), and in particular Article 15 (7) thereof,

Whereas the import levies on white sugar and raw sugar were fixed by Regulation (EEC) No 1564/76 (3), as last amended by Regulation (EEC) No 1052/77 (4);

Whereas it follows from applying the rules and other provisions contained in Regulation (EEC) No 1564/76 to the information at present available to the Commission that the levies at present in force should be altered as shown in the Annex to this Regulation,

#### HAS ADOPTED THIS REGULATION:

#### Article 1

The levies referred to in Article 15 (1) of Regulation (EEC) No 3330/74 are, in respect of white sugar and standard quality raw sugar, hereby fixed as shown in the Annex to this Regulation.

#### Article 2

This Regulation shall enter into force on 24 May 1977.

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

Done at Brussels, 23 May 1977.

For the Commission Finn GUNDELACH Vice-President

# **ANNEX**

# to the Commission Regulation of 23 May 1977 fixing the import levies on white sugar and

		(u.a/100 kg)
CCT heading No	Description of goods	Levy
17.01	Beet sugar and cane sugar, solid: A. White sugar B. Raw sugar	20·53 17·23 (¹)

<sup>(1)</sup> Applicable to raw sugar with a yield of 92 %; if the yield is other than 92 %, the levy applicable is calculated in accordance with the provisions of Article 2 of Regulation (EEC) No 837/68.

OJ No L 359, 31. 12. 1974, p. 1.

<sup>(</sup>²) OJ No L 354, 24. 12. 1976, p. 1. (²) OJ No L 172, 1. 7. 1976, p. 31. (¹) OJ No L 125, 19. 5. 1977, p. 35.

# **CORRIGENDA**

Corrigendum to Commission Regulation (EEC) No 1008/77 of 13 May 1977 fixing the import levies on milk and milk products

(Official Journal of the European Communities No L 121 of 14 May 1977)

Annex, page 7, footnotes (9) c) and (10) b):

for: '9.70 u.a.',

read: '9.92 u.a.'.

Footnote (a) is deleted.