

Official Journal

of the European Communities

ISSN 0378-6978

L 327

Volume 22

24 December 1979

English edition

Legislation

Contents

I *Acts whose publication is obligatory*

.

II *Acts whose publication is not obligatory*

Council

79/1065/EEC:

- ★ Council Decision of 12 November 1979 on the conclusion of the International Olive Oil Agreement 1979 1
- International Olive Oil Agreement 1979 2
- ★ Information relating to the signature by the Community of the International Olive Oil Agreement 1979 16

Commission

79/1066/EEC:

- ★ First Commission Directive of 13 November 1979 laying down Community methods of analysis for testing coffee extracts and chicory extracts 17

79/1067/EEC:

- ★ First Commission Directive of 13 November 1979 laying down Community methods of analysis for testing certain partly or wholly dehydrated preserved milk for human consumption 29

Acts whose titles are printed in light type are those relating to day-to-day management of agricultural matters, and are generally valid for a limited period.
The titles of all other Acts are printed in bold type and preceded by an asterisk.

II

(Acts whose publication is not obligatory)

COUNCIL**COUNCIL DECISION**

of 12 November 1979

on the conclusion of the International Olive Oil Agreement 1979

(79/1065/EEC)

THE COUNCIL OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community, and in particular Article 113 thereof,

Having regard to the recommendation from the Commission,

Whereas the International Olive Oil Agreement 1979 is concerned in general with the promotion of international cooperation and contributes to the attainment of the objectives of the Community commercial and common agricultural policy,

The text of the Agreement is annexed to this Decision.

Article 2

The President of the Council is hereby authorized to designate the person empowered to sign the International Olive Oil Agreement 1979, in order to bind the Community.

Article 3

This Decision is published in the *Official Journal of the European Communities*.

Done at Brussels, 12 November 1979.

HAS DECIDED AS FOLLOWS:

Article 1

The International Olive Oil Agreement 1979, is hereby approved on behalf of the European Economic Community,

For the Council

The President

J. GIBBONS

INTERNATIONAL OLIVE OIL AGREEMENT 1979**PREAMBLE**

BEARING IN MIND that the olive tree:

- is a plant not only indispensable for the maintenance and conservation of soils, but one which makes it possible to develop land that will not yield other crops and which, even under non-intensive farming conditions, which account for the essential part of present production, responds favourably to any improvement in cultivation,
- is a perennial fruit tree that with appropriate techniques, which should be available to olive-growing countries and particularly to developing olive-growing countries, permits a return on what is invested in its cultivation,

EMPHASIZING that its cultivation governs the existence and level of living of millions of families which are wholly dependent on the measures taken to maintain and expand the consumption of its products, both in the producing countries themselves and in non-producing consumer countries,

MINDFUL that olive oil forms an essential basic commodity in the regions where olive-growing is established,

BEARING IN MIND that the essential feature of the olive oil market lies in irregular harvests and in the irregularity with which supplies reach the market, and that this results in fluctuations in the value of production, instability of prices and export earnings, and considerable differences in producers' incomes,

BEARING IN MIND that these circumstances give rise to special difficulties which may seriously harm the interests of producers and consumers and jeopardize general policies of economic expansion in countries in the regions where olive-growing is established,

EMPHASIZING in this connection the major importance of the products in question for the economies of many countries, particularly developing olive-growing countries,

MINDFUL that the measures to be taken in the light of the very particular characteristics of olive-growing and the olive oil market transcend the national sphere, and that international action is essential,

CONSIDERING the International Olive Oil Agreement, 1963, as extended and amended by the successive Protocols of 30 March 1967, 7 March 1969, 23 March 1973 and 7 April 1978, including the amendments which entered into force on 1 November 1971 by virtue of the provisions of Article 38 of the Agreement (all of which instruments are hereinafter referred to as 'the International Olive Oil Agreement, 1963'),

CONSIDERING that in principle that Agreement will expire on 31 December 1979,

BEING OF THE VIEW that it is essential to continue and develop the work undertaken within the framework of that Agreement, and that it is desirable to conclude a new agreement,

THE PARTIES TO THIS AGREEMENT HAVE AGREED AS FOLLOWS:

CHAPTER I

GENERAL OBJECTIVES

Article 1

The objectives of this Agreement, which take account of the provisions of resolution 93 (IV) of the United Nations Conference on Trade and Development, are as follows:

- (a) to foster international cooperation on problems relating to the world olive oil economy in general;
- (b) to foster research and development and the elaboration of all possible means of applying techniques relevant to the problems facing olive oil and the olive oil sector generally in the fields of production and processing, and conducive to the modernization of olive husbandry and the olive oil industry through technical and scientific planning, with a view to encouraging the transfer of technology, improving olive husbandry and the quality of the products obtained therefrom and reducing their cost of production, thus improving the position of olive oil in the total market for edible fluid vegetable oils;
- (c) to facilitate the study and application of measures for expanding the international olive oil trade, in order to increase the resources which producer countries, and especially developing producer countries, derive from their exports and to enable their economic growth and social development to be hastened, while taking consumer interests into account;
- (d) to facilitate the study and application of measures for balancing production and consumption through the introduction of the appropriate arrangements, including arrangements to expand consumption;
- (e) to lessen the drawbacks associated with fluctuations in the supplies available on the market, in order in particular:
 - (i) to prevent excessive fluctuations in prices, which must be at levels that are remunerative and just to producers and equitable to consumers;
 - (ii) to create conditions which allow production, consumption and international trade to expand harmoniously, having regard to the ways in which they are interrelated;
- (f) to forestall and, where appropriate, combat any practices of unfair competition in the international olive oil trade and ensure the delivery of merchandize which complies fully with the terms of the contracts that are concluded;

(g) to foster the coordination of production policies and marketing policies for olive oil and the organization of the olive oil market;

(h) to improve market access and reliability of supply, market structures, and marketing, distribution and transport systems;

(i) to improve procedures for information and consultation in order, among other things, to enhance the transparency of the olive oil market;

(j) to study and facilitate the application of the necessary measures for other products of the olive tree;

(k) to study the situation of the olive oil industry as far as the environment is concerned and, where appropriate, recommend suitable action in conformity with the recommendations of the United Nations Conference on the Human Environment, 1972 to abate any nuisances;

(l) to continue and extend the work done under the previous International Olive Oil Agreements.

CHAPTER II

MEMBERS

Article 2

Each Contracting Party shall constitute a single member of the Council.

CHAPTER III

DEFINITIONS

Article 3

1. For the purposes of this Agreement:

- (a) 'Council' means the International Olive Oil Council referred to in Article 27;

- (b) 'olive crop year' means the period from 1 November of each year to 31 October of the following year;
- (c) 'mainly producing member' means a member whose production of olive oil in the olive crop years 1972/73 to 1977/78 inclusive exceeded its imports for the calendar years 1973 to 1978 inclusive;
- (d) 'mainly importing member' means a member whose production of olive oil in the olive crop years 1972/73 to 1977/78 inclusive was less than its imports for the calendar years 1973 to 1978 inclusive, or for which no production of olive oil was recorded during those crop years;
- (e) 'member' means a Contracting Party to this Agreement.

2. Any reference in this Agreement to a 'government' or 'governments' shall be construed as including a reference to the European Economic Community (hereinafter referred to as 'the EEC') or any intergovernmental organization having responsibilities in respect of the negotiation, conclusion and application of international commodity agreements. Accordingly, any reference in this Agreement to 'signature' or to the 'deposit of instruments of ratification, acceptance or approval' or 'an instrument of accession' or to a 'notification of provisional application' by a government shall, in the case of the EEC, be construed as including signature or notification of provisional application on behalf of the EEC by its competent authority, and the deposit of the instrument required by the institutional procedures of the EEC to be deposited for the conclusion of an international agreement. It shall also, in the case of an intergovernmental organization having responsibilities in respect of the negotiation, conclusion and application of international commodity agreements, be construed as including signature or notification of provisional application on behalf of the intergovernmental organization concerned by its competent authority, and the deposit of the instrument required by its institutional procedures for the conclusion of an international agreement.

3. Notwithstanding the provisions of paragraph 1 (c) and (d) of this Article, the EEC shall be considered both as a 'mainly producing member' and as a 'mainly importing member'.

4. If an intergovernmental organization other than the EEC having responsibilities in respect of the negotiation, conclusion and application of international commodity agreements contemplates becoming a Contracting Party, the modalities of its participation in this Agreement shall be decided by agreement between the Council and that intergovernmental organization before commencement of

the procedure which the organization has to follow to become a Contracting Party.

CHAPTER IV

GENERAL OBLIGATIONS

Article 4

The members undertake not to adopt any measure that conflicts with their obligations under this Agreement and with the general objectives set forth in Article 1.

Article 5

Both the producing and consuming members undertake to adopt all appropriate measures to facilitate trade, encourage olive oil consumption and ensure the proper development of the international olive oil trade. To that end, they undertake to conform to the principles, rules and guidelines they have approved in the competent international forums. They also undertake to adopt measures to encourage the sale of olive oil at prices which are competitive at the consumer level, including measures for determining assistance and narrowing the price differential between olive oil and other edible vegetable oils, in order to encourage olive oil consumption.

Article 6

The members declare that, in order to raise the level of living of their populations, they will endeavour to maintain fair standards in working conditions throughout the olive-growing and olive oil industry and activities deriving from it.

Article 7

The members undertake to make available and furnish to the Council all the statistics, data and documentation which it needs to discharge its functions under this Agreement, and in particular any information it requires in order to establish the olive oil balance and acquire a knowledge of members' national olive oil policies.

CHAPTER V

TECHNICAL MEASURES

Article 8

1. In order to achieve the general objectives set forth in Article 1 with regard to technical improvements in olive cultivation and oil extraction, the Council shall foster and promote related activities and programmes.

2. It shall in particular:

- (a) collect technical information and circulate it to all members;
- (b) promote action to coordinate technical improvement activities among members, including action within the framework of interregional or regional planning;
- (c) assist national planning relating to technical improvements in olive cultivation and oil extraction and to research, research application, dissemination of know-how, experimentation and demonstration, in particular in the developing olive-growing countries;
- (d) undertake the necessary studies on the economic returns which can be expected from the application of research;
- (e) foster appropriate action to train high-level and specialized staff;
- (f) convene or foster the holding of international meetings;
- (g) encourage the transfer of technology to developing olive-growing countries from countries highly advanced in olive cultivation and oil extraction techniques;
- (h) promote bilateral or multilateral cooperation which can assist the Council in achieving the objectives of this Agreement.

Article 9

1. The Council shall, in support of measures to improve olive cultivation and oil extraction techniques, include in its administrative budget a special section of a maximum annual amount of US\$ 100 000; any sums not used under this section in any financial year may be carried over to subsequent financial years but may in no circumstances be transferred to other sections of the administrative budget.

2. The Council will in addition, as part of the development of international cooperation, endeavour to procure such essential financial and/or technical assistance as may be obtainable from the competent international, regional or national organizations, whether financial or of some other kind.

3. The provisions of paragraph 1 of this Article will be applicable, as the case may be, with the international financial assistance given to activities or projects submitted to the Council for improvements in olive cultivation and oil extraction techniques.

CHAPTER VI

DESIGNATIONS AND DEFINITIONS OF OLIVE OILS AND OLIVE-RESIDUE OILS

INDICATIONS OF SOURCE AND APPELLATIONS OF ORIGIN

Article 10

1. The designation 'olive oil' shall be restricted to oil obtained solely from the olive, to the exclusion of oil obtained by solvent or re-esterification processes and any mixture with oils of other kinds.

2. The members undertake to suppress in both internal and international trade, with the least possible delay and at the latest before the expiry of this Agreement, any use of the designation 'olive oil' alone or in combination with other words which is not in conformity with this Article.

3. In no case shall the designation 'olive oil' be used alone to refer to olive residue oils.

Article 11

1. The designations of olive oils and olive-residue oils of the different qualities are given below with the definition corresponding to each designation:

A. **Virgin olive oil:** oil which is obtained from the fruit of the olive tree solely by mechanical or other physical means under conditions, and particularly thermal conditions, that do not lead to deterioration of the oil, and which has not undergone any treatment other than washing, decantation, centrifugation and filtration, to the exclusion of oil obtained by solvent or re-esterification processes and any mixture with oils of other kinds. Virgin olive oil shall be classified and designated as follows:

- (a) *Virgin olive oil* ⁽¹⁾ fit for consumption as it is:

⁽¹⁾ It shall be permissible to use the term 'natural' for any virgin olive oil which is fit for consumption as it is.

- (i) **Virgin olive oil extra:** virgin olive oil of absolutely perfect flavour having a maximum acidity, in terms of oleic acid, of 1 gram per 100 grams;
 - (ii) **Virgin olive oil fine:** virgin olive oil complying with the conditions for virgin olive oil extra, except as regards acidity, which in terms of oleic acid shall not exceed 1.5 grams per 100 grams;
 - (iii) **Virgin olive oil semi-fine (or virgin olive oil ordinary):** virgin olive oil of good flavour having a maximum acidity, in terms of oleic acid, of 3 grams per 100 grams with a margin of tolerance of 10 % of the acidity indicated.
- (b) *Virgin olive oil not fit for consumption as it is:*
- Virgin olive oil lampante (lamp oil): off-flavour olive oil or olive oil having an acidity, in terms of oleic acid, of more than 3.3 grams per 100 grams.
- B. Refined olive oil:** oil obtained by refining virgin olive oil.
- C. Olive oil or pure olive oil:** oil consisting of a blend of virgin olive oil and refined olive oil.
- D. Olive-residue oil:** crude oil obtained by treating olive residues with solvent and intended for subsequent refining for human consumption, or for technical use. Olive-residue oil shall be classified and designated as follows:
- (a) *Refined olive-residue oil:* oil intended for food use and obtained by refining crude olive-residue oil. (Note: Blends of refined olive-residue oil and virgin olive oil — habitually destined for internal consumption in certain producing countries — shall be designated 'refined olive-residue oil and olive oil'. These blends shall not, under any circumstances, be termed simply 'olive oil', and it shall be obligatory for the packagings to bear the words 'refined olive-residue oil and olive oil'.)
 - (b) *Olive-residue oil for technical use:* all other crude olive-residue oils.
- The Council shall undertake and submit, before the end of the second year following the entry into force of this Agreement, a comprehensive study of the market for olive-residue oil, including blends of olive-residue oil and olive oil, with special reference to the repercussions of the marketing of these products on the olive oil economy as a whole.

2. Each of the above designations for the various qualities of olive oil and olive-residue oil shall comply with quality criteria laid down in conformity with the recommendations made under Article 28 (2), in regard to standards for the physical and chemical characteristics of olive oil and olive-residue oil.

3. The designations prescribed in paragraph 1 of this Article shall be compulsory in international trade and shall be used for each quality of olive oil and olive-residue oil; they shall appear in clearly legible characters on all packagings.

4. In the matter of quality criteria, as indicated in Article 28 (2), the Council will determine uniform standards applicable to the international trade. Until these standards are determined, reference shall be made to the standards customarily used in that trade, and in particular to such standards as are recommended by the Council within the framework of its activities.

Article 12

1. The members undertake to adopt at the earliest possible date, and at the latest before the expiry of this Agreement, all necessary measures in the form prescribed by their respective legislations to ensure the application of the principles and provisions laid down in Articles 11 and 13, and will endeavour to apply them in their internal trade.

2. They undertake in particular to prohibit and repress the use in their territories, for purposes of international trade, of such indications of source, appellations of origin and designations of olive oil and olive-residue oil as conflict with those principles. This undertaking shall apply to all inscriptions placed on packagings, invoices, consignment notes and commercial documents or used in advertising, and to trade marks, registered names and illustrations connected with the international marketing of olive oil and olive-residue oil, in so far as such inscriptions might constitute false statements or give rise to confusion as to the origin, source or quality of the olive oil or olive-residue oil concerned.

Article 13

1. Indications of source, when given, may only be applied to virgin olive oil produced and originating exclusively in the country, region or locality mentioned. Appellations of origin, when given, may only be applied to virgin olive oil extra produced and originating exclusively in the country, region or locality mentioned. Moreover, indications of

source and appellations of origin may not be used except in conformity with the conditions prescribed by the law of the country of origin.

2. Blends of virgin olive oil and refined olive oil may constitute types whose characteristics may be determined by agreement between buyers and sellers. Whatever their origin, these may only bear the indication of source of the exporting country. Nevertheless, when the oil has been packed and exported by the country supplying the virgin olive oil extra used in the blend, it may be identified by the appellation of origin of that virgin olive oil. Where use is made of the generic designation 'Riviera', well known in the international olive oil trade as a blend of virgin olive oil extra and refined olive oil, this designation shall in every case be followed by the word 'type'. The word 'type' must appear on all packagings in printed characters of the same size and appearance as the word 'Riviera'.

Article 14

1. Any dispute concerning indications of source and appellations of origin which arises from the interpretation of the provisions of the present chapter or from difficulties of application shall, if the dispute has not been settled by direct negotiation, be examined by the Council.

2. The Council shall endeavour to reconcile the dispute, after seeking the opinion of an advisory panel as provided for in Article 40 (1), and after consultation with the World Intellectual Property Organization, the International Federation of Olive Growers and a competent professional organization of a mainly importing member, and if necessary with the International Chamber of Commerce and the specialized international institutions for analytical chemistry; if this is unsuccessful, and after every means has been employed to reach agreement, the members concerned shall have the right of recourse in the final instance to the International Court of Justice.

CHAPTER VII

WORLD PUBLICITY TO PROMOTE OLIVE OIL CONSUMPTION

Publicity campaigns

Article 15

1. The members contributing to the publicity fund referred to in paragraph 3 of this Article undertake to conduct general olive oil publicity activities jointly, with a view to

maintaining and increasing world olive oil consumption, on the basis of the use of the designation 'olive oil' as defined in Article 10.

2. These activities shall take the form of educational and advertising campaigns and deal with the organoleptic and chemical characteristics of olive oil, and if necessary with its nutritive, therapeutic and other properties, but they shall not be concerned with any indication of quality, origin or source.

3. The resources of the publicity fund shall be used in the light of the following criteria:

- (a) the volume of consumption, with a view to maintaining and if possible expanding existing outlets;
- (b) the creation of new outlets for olive oil;
- (c) the return obtainable on the advertising expenditure.

Article 16

The general and specific publicity campaigns to be undertaken under Article 15 shall be decided on by the Council in the light of the resources made available to it for the purpose and of the following considerations:

- (a) priority shall be given to action in the mainly consuming countries and in countries in which the consumption of olive oil is likely to increase;
- (b) consultations with appropriate organizations and institutions.

Article 17

The Council shall administer the funds allocated for joint publicity purposes. It shall prepare an annual estimate of receipts and expenditure relating to this publicity as an annex to its budget.

Publicity fund

Article 18

1. The mainly producing members undertake to place at the disposal of the Council for each calendar year, for joint publicity purposes, a sum of money equivalent to US \$ 300 000 and payable in that currency. The Council may nevertheless decide what proportion of its contribution each member may be permitted to pay in other freely convertible currencies. The above sum of US \$ 300 000 may be increased by the Council, but not in excess of

US \$ 500 000, on condition, on the one hand, that no member's contribution may be increased without that member's consent and, on the other, that any alteration occurring in this connection in the coefficients referred to in paragraph 3 of this Article shall require a unanimous decision of the mainly producing members. The above sum of US \$ 300 000 may be reduced if the aggregate production of the members represents less than 80 % of the world production of olive oil in the reference period contemplated in Article 3 (1) (c) and (d). In this case, the sum of US \$ 300 000 shall be reduced to an amount proportionate to the share of world production represented by the aggregate production of the mainly producing members.

2. Mainly importing members may pay contributions to the publicity fund by special agreement with the Council. These contributions shall be added to the moneys constituting the fund, as determined by the application of paragraph 1 of this Article.

3. Subject to the provisions of paragraph 4 of this Article, the mainly producing members shall contribute to the publicity fund in proportion to their importance in the world olive oil economy, in accordance with a coefficient determined for each of them on the basis of the average production and average net exports or imports of olive oil of each of the members in the olive crop years and calendar years specified in Article 3 (1) (c) and (d), in the proportion of 20 % for production and 80 % for net exports or imports.

4. In the case of the EEC, net exports or imports of olive oil in the calendar years specified in Article 3 (1) (c) and (d) shall be determined after deduction of intra-EEC trade.

5. Contributions to the publicity fund shall be payable for the whole of a calendar year. The annual contribution of each mainly producing member shall become payable for the first time when it becomes a member provisionally or definitively, and subsequently on 1 January each year.

6. The provisions of Article 38 (5) shall apply as regards the collection of contributions to the publicity fund and in the event of delay in their payment.

7. On the expiry of this Agreement, and unless it is prolonged, extended or renewed, any funds not used for publicity shall be repaid to members proportionately to their total contributions for publicity during the periods of validity of the International Agreement on Olive Oil, 1956, the International Olive Oil Agreement, 1963 and this Agreement.

8. (a) In regard to all decisions relating to publicity, each mainly producing member shall have a number of votes proportionate to its contribution to the publicity fund under this Article. Any fraction of a vote

resulting from the application of the coefficient determined under paragraph 3 of this Article shall be counted as a whole vote.

(b) When, under paragraph 2 of this Article, a member concludes a special agreement with the Council to pay a contribution to the publicity fund, it shall acquire a number of votes proportionate to its contribution, on condition that the agreement in question covers the period which is to elapse before this Agreement expires.

(c) Decisions relating to publicity shall be taken by a majority of four-fifths of the votes cast by the members contributing to the publicity fund, which shall include the votes of a majority of the members so contributing that are entitled to vote.

Article 19

The technical execution of publicity campaigns may be entrusted by the Council to specialized bodies of its own choice which are representative of the olive-growing and olive oil industries, including the International Federation of Olive Growers.

Article 20

The Council may receive donations from governments or from other sources for the joint publicity in question. Such occasional resources shall be added to the moneys constituting the publicity fund, as determined by the application of Article 18.

CHAPTER VIII

ECONOMIC MEASURES

Article 21

1. Within the framework of the general objectives set forth in Article 1, and with a view to contributing towards the normalization of the olive oil market and correcting any imbalance between international supply and demand due to irregularity of harvests or to other factors, the Council shall at the beginning of each olive crop year make a detailed examination of olive oil balances and an over-all estimate of olive oil supplies and requirements, on the basis of the information furnished by each member

under Article 7 and any information communicated to the Council by governments of States not parties to this Agreement but interested in the international olive oil trade, and any other relevant statistical material available to the Council.

2. Not later than 31 May each year the Council shall, taking into account all the information available to it on that date, make a further examination of the market situation and a further over-all estimate of olive oil supplies and requirements, and may propose to the members such measures as it considers desirable.

3. An Economic Committee shall be set up and meet at regular intervals to exchange viewpoints on the world situation in the olive oil market, with the aim of finding solutions to problems liable to disrupt the international olive oil trade.

Article 22

1. The Council shall undertake studies with a view to making recommendations to the members that are designed to achieve a balance between production and consumption, and, more generally, normalization of the olive oil market over the long term through the application of appropriate measures, including measures to encourage the sale of olive oil at prices which are competitive at the consumer level, so as to narrow the price differential between olive oil and other edible vegetable oils, particularly through the granting of assistance.

2. With a view to such normalization, the Council shall also provide suitable solutions to the problems which may arise as regards the evolution of the international olive oil market, through appropriate modalities, account being taken of market imbalances resulting from fluctuations in production or from other causes.

Article 23

When the common fund which is envisaged in resolution 93 (IV) of the United Nations conference on trade and development, and whose fundamental elements are identified in resolution I (III) adopted on 19 March 1979 by the United Nations negotiating conference on a common fund, becomes operational under the integrated programme for commodities, the Council shall, in the light of those resolutions, consider measures through which it might take full advantage of any financial arrangements available under the said fund, and may make appropriate recommendations on the matter.

CHAPTER IX

OTHER PRODUCTS OF THE OLIVE TREE

Article 24

1. Within the framework of the general objectives set forth in Article 1, the Council shall request the close co-operation of all its members in the communication of the necessary statistical information on table olives and other products of the olive tree.

2. The Council shall at the beginning of each olive crop year make a detailed examination of quantitative and qualitative table olive balances, on the basis of the above information and any information transmitted to it by governments of States not parties to this Agreement but interested in the international trade in table olives, and any other statistical material available to the Council on the subject.

3. Not later than 31 May each year the Council shall, taking account of all the information available to it on that date, make a further examination of the market situation and an over-all estimate of table olive supplies and requirements, and may propose to the members such measures as it considers desirable.

Article 25

The Council will pursue the appropriate studies concerning:

- (a) the adoption and use of an international standard contract for table olive transactions;
- (b) arrangements with regard to conciliation and international arbitration for any disputes relating to international table olive transactions;
- (c) the adoption of uniform qualitative standards for table olives;
- (d) the biological value of table olives, with particular reference to their specific qualities and properties.

Article 26

1. The Council shall promote such market surveys as are thought conducive to stimulating the growth of table olive consumption. It will submit them to the members for the purposes which they consider appropriate.

2. In this connection, the Council will endeavour to obtain for all members, or for those of its members as may require it, such assistance of various kinds, including financial assistance, as international or other competent bodies may grant.

CHAPTER X

ADMINISTRATION

International Olive Oil Council

Article 27

The International Olive Oil Council shall administer this Agreement.

Functions of the Council

Article 28

1. Within the framework of its functions of administration under this Agreement, the Council shall:

- (a) exercise all such powers and perform or cause to be performed all such functions as are necessary for executing the express provisions of this Agreement and generally administering it;
- (b) promote all activities conducive to the harmonious expansion of the world olive oil economy by every means and encouragement in its power in the fields of production, consumption and international trade, having regard to the ways in which they are interrelated.

2. The Council shall examine ways and means of ensuring the expansion of international trade and an increase in olive oil consumption. In particular, it shall make appropriate recommendations to the members concerning:

- (a) the adoption and use of a standard international contract for transactions in olive oil and olive-residue oil;
- (b) the constitution and functioning of an international conciliation and arbitration office to deal with any disputes relating to transactions in olive oil and olive-residue oil;
- (c) the establishment of uniform standards for the physical and chemical characteristics of olive oil and olive-residue oil;
- (d) the establishment of uniform methods of analysis.

3. The Council shall take all appropriate measures to bring about the preparation of a code of standard fair trade practices for the international trade in olive oil and olive-residue oil, with particular reference to margins of tolerance.

4. The Council shall take any measures it considers useful for the suppression of unfair competition at the international level, including such competition by States which are not parties to this Agreement or by persons who are nationals of such States.

5. The Council may also undertake studies on the activities mentioned in paragraph 1 (b) of this Article. It is furthermore authorized to undertake or arrange for the undertaking of other work, including the collection of detailed information on special assistance in different forms to the olive-growing and olive oil industries, so as to enable it to formulate any recommendations and suggestions it may consider appropriate for achieving the general objectives laid down in Article 1. Any such studies and work shall, in particular, cover as many countries or groups of countries as possible and take into account the general social and economic conditions of the countries concerned.

6. The Council shall establish procedures under which the members shall inform it of the conclusions they reach after considering the recommendations and suggestions mentioned in this Article or arising from the application of this Agreement.

Article 29

1. The Council shall draw up rules of procedure consistent with the provisions of this Agreement. It shall keep up to date such records as it requires to perform its functions under this Agreement and such other records as it considers desirable. In the event of inconsistency between the rules of procedure it adopts and the provisions of this Agreement, the Agreement shall prevail.

2. The Council shall draw up, prepare and publish such reports, studies, charts, surveys or other documents as it considers useful and necessary.

3. The Council shall, at least once a year, publish a report on its activities and on the operation of this Agreement.

4. The Council may appoint such special committees as it deems useful for assisting it in the exercise of its functions under this Agreement.

5. The Council shall exercise all other functions that are necessary for the execution of the provisions of this Agreement.

Composition of the Council

Article 30

1. Each member shall have the right to vote. It shall have the right to be represented on the Council by a delegate and may appoint alternates. The delegate and alternates may be accompanied at the meetings of the Council by as many advisers as each member deems necessary.

2. The Council shall elect, from among the delegations of the members, a chairman who shall not have the right to vote and who shall hold office for one olive crop year. In the event of the chairman being a voting delegate, his right to vote shall be exercised by another member of his delegation. The chairman shall receive no remuneration.

3. The Council shall also elect a deputy chairman from among the delegations of the members. In the event of the deputy chairman being a voting delegate, he shall have the right to vote except when acting as chairman, in which case he shall transfer his right to vote to another member of his delegation. He shall hold office for one olive crop year and shall receive no remuneration.

Meetings of the Council

Article 31

1. The seat of the Council shall be at Madrid unless the Council decides otherwise. It shall hold its sessions there unless it decides exceptionally to hold a particular session elsewhere. If a member invites the Council to meet elsewhere than at its seat and the Council agrees to do so, that member shall bear the extra expenditure which this entails for the budget of the Council.

2. The Council shall meet at least twice a year, having regard in particular to the provisions of Article 21.

3. The Council may be convened at any time at the discretion of its chairman. The chairman shall also convene the Council if this is so requested:

(a) by five members;

(b) by one or more members holding at least 10 % of the total votes.

4. Notice of the sessions provided for in paragraph 2 of this Article shall be given at least 30 days before the date of the first meeting of each such session. Notice of the session provided for in paragraph 3 of this Article shall be given at least 15 days before the date of the first meeting of each such session.

Article 32

1. The quorum required for every meeting of the Council shall be the presence of the representative of a majority of the members holding at least two-thirds of the total votes.

2. If this quorum does not exist, the meeting shall be postponed for 24 hours and the quorum required shall be the

presence of the representatives of a majority of the members holding at least 50 % of the total votes.

Article 33

The Council may take decisions by an exchange of correspondence between the chairman and the members without holding a meeting, provided that no member objects. Any such decision shall be communicated as quickly as possible to all the members and be entered in the record of the following meeting of the Council.

Article 34

1. The number of votes allotted to each member shall be determined for the duration of this Agreement by the formula $n = p + i + 5$, but it may not exceed 450.

In this formula:

— n shall be the number of votes allotted to the member,

— p shall be the average annual olive oil production in thousands of tonnes in the olive crop years 1972/73 to 1977/78, any fraction of 1 000 tonnes above a whole number being disregarded,

— i shall be the average annual net imports of olive oil in thousands of tonnes in the calendar years 1973 to 1978, any fraction of 1 000 tonnes above a whole number being disregarded,

— 5 shall be the basic number of votes allotted to each member in each group of members.

2. Notwithstanding the provisions of paragraph 1 of this Article, since the EEC is considered under Article 3 (3) as both a mainly producing member and a mainly importing member, the number of votes allotted to it in each group of members shall be determined as follows:

— firstly, as a mainly producing member, by the formula $n = p + 5$,

— secondly, as a mainly importing member, by the formula $n = i + 5$, i being calculated after deducting intra-EEC trade,

but the number of votes allotted to the EEC in either group of members may not exceed 450.

Decisions of the Council*Article 35*

1. Unless otherwise provided in this Agreement, and subject to any arrangements under Article 47 (5), decisions of the Council shall be taken by a majority of four-fifths of the votes cast, including the votes of a majority of the members having the right to vote. The votes of abstaining members shall not be counted.

2. Any member may authorize the voting delegate of another member to represent its interests and to exercise its right to vote at one or more meetings of the Council. Evidence of such authorization acceptable to the Council shall be submitted to the Council.

3. The voting delegate of a member may not, in addition to the powers and the right to vote held by that member, represent the interests and exercise the right to vote of more than one other member.

Secretariat*Article 36*

1. The Council shall have a secretariat composed of a Director and such staff as are required for the work of the Council and its committees. The Council shall appoint the Director and determine his functions. The staff shall be appointed in accordance with regulations which are established by the Council and take account of the regulations applicable to the staffs of similar intergovernmental organizations; members of the staff shall not hold any office outside the organization or accept any other employment.

2. It shall be a condition of employment of the Director and the staff of the secretariat that they do not possess or shall cease to possess any direct or indirect commercial or financial interest in any of the various sectors of the olive-growing and olive oil industry.

3. The functions of the Director and of the members of the secretariat shall be exclusively international. In the discharge of their duties, they shall not seek or receive instructions from any government or from any authority other than the organization. They shall refrain from any action incompatible with their position as international officials.

4. The members shall respect the international character of the responsibilities of the members of the secretariat and shall not seek to influence them in the discharge of their duties.

CHAPTER XI**PRIVILEGES AND IMMUNITIES***Article 37*

1. In the territory of each member, and in so far as that member's legislation allows, the Council shall have the legal capacity necessary for the performance of the functions conferred upon it by this Agreement.

2. The government of the State in which the seat of the Council is situated shall, in so far as that State's legislation allows, exempt the funds of the Council and the salaries paid by the Council to its staff from taxation.

3. The Council, the Director and the staff of the secretariat shall enjoy the privileges, immunities and facilities set out in the Headquarters Agreement between the Council and the government of the State in which the seat of the Council is situated.

4. The Council may conclude with one or more members agreements to be approved by the Council concerning the privileges and immunities required to ensure the proper application of this Agreement.

CHAPTER XII**FINANCIAL PROVISIONS***Article 38*

1. Except for the expenses of the chairman, which shall be borne by the Council, the expenses of delegations to the Council shall be met by the members concerned. The contribution of each member to the administrative budget for each calendar year shall be proportionate to the number of votes which that member possesses when the budget for the year in question is approved.

2. At its first session the Council will approve an administrative budget for the first calendar year and assess the contribution to be paid by each member. Each year thereafter, at the autumn session, the Council shall approve its administrative budget for the following calendar year and assess the contribution to be paid by each member for the calendar year in question.

3. The initial contribution of any member which becomes a party to this Agreement after its entry into force shall be assessed by the Council on the basis of the number of votes allotted to that member and of the unexpired portion

of the year in question, but the assessments made upon the other members for that calendar year shall not be altered.

4. The contributions provided for in this article shall become payable upon their approval by the Council for the calendar year for which they are assessed. They shall be determined in US dollars and be payable in that currency or in the equivalent amount of another freely convertible currency.

5. If a member does not pay its contribution to the administrative budget in full within the six months commencing at the beginning of the financial year, the Director shall request it to make payment as quickly as possible. If the member in question does not settle its contribution within the three months following the end of that six-month period, its rights to vote at Council sessions and meetings of committees and to hold elective office in the Council and its committees shall be suspended until its contribution has been paid in full. It shall not however, unless the Council so decides by vote, be deprived of any of its other rights or be released from any of its obligations under this Agreement. It may not be relieved by vote from its financial obligations under the Agreement.

6. Any member whose participation in this Agreement ceases through its withdrawal or exclusion or for any other reason during the period of validity of the Agreement shall make the payments which it is due to make to the Council, and shall perform all the undertakings which it entered into before the date on which the cessation of its participation in this Agreement took effect. No such member may claim any share in the proceeds of liquidation of the assets of the Council upon the expiry of the Agreement.

7. After the spring session the Council shall publish a certified statement of its receipts and expenditure in the previous calendar year.

8. The Council shall, if dissolved, first take the necessary steps to settle its liabilities, to place its records in safe keeping and to dispose of any balance standing to its credit at the date of expiry of this Agreement.

CHAPTER XIII

COOPERATION WITH OTHER ORGANIZATIONS AND ADMISSION OF OBSERVERS

Article 39

1. The Council may make any arrangements that are appropriate for consultation or cooperation with the United Nations and its organs, in particular the United Nations Conference on trade and development (UNCTAD), with the

Food and Agriculture Organization of the United Nations (FAO) and with other specialized agencies of the United Nations and intergovernmental organizations where necessary. It may also make whatever arrangements it considers suitable for cooperating with governmental and non-governmental organizations and agencies. It may in addition invite any organization referred to in this Article to attend any of its meetings as an observer.

2. In view of the particular role of UNCTAD in international commodity trade, the Council shall keep UNCTAD suitably informed of its activities and programmes of work. The same shall apply as regards FAO.

3. The Council may also invite any member of the United Nations or of any of its specialized agencies or of the International Atomic Energy Agency that is not a party to this Agreement to attend any of its meetings as an observer.

CHAPTER XIV

DISPUTES AND COMPLAINTS

Article 40

1. Any dispute which concerns the interpretation or application of this Agreement, other than a dispute under Article 14, and which is not settled by negotiation shall, at the request of any member which is a party to the dispute, be referred to the Council for decision after it has sought an opinion, where appropriate, from an advisory panel, the composition of which shall be laid down in the Council's rules of procedure.

2. A substantiated opinion by the advisory panel shall be submitted to the Council, which shall in all circumstances settle the dispute after considering all the relevant facts.

3. Any complaint that a member has failed to fulfil its obligations under this Agreement shall, at the request of the member making the complaint, be referred to the Council, which shall take a decision on the matter after consulting the members concerned and, where appropriate, seeking an opinion from the advisory panel referred to in paragraph 1 of this Article.

4. A member may, by vote of the Council, be found in breach of this Agreement.

5. If the Council finds that a member has committed a breach of this Agreement, it may either impose sanctions on that member, ranging from a simple warning to the suspension of the member's right to vote until it has met its obligations, or exclude the member from participation in the Agreement.

CHAPTER XV

FINAL PROVISIONS

Participation in the Agreement

Article 41

1. The government of any State invited to the United Nations conference on olive oil, 1979 may become a party to this Agreement in accordance with its constitutional or institutional procedures:

- (a) by signing it; or
- (b) by ratifying, accepting or approving it, after having signed it subject to ratification, acceptance or approval; or
- (c) by acceding to it.

2. Every signatory government shall on signing this Agreement state whether, according to its constitutional or institutional procedures, its signature is or is not subject to ratification, acceptance or approval.

Signature

Article 42

This Agreement shall be open for signature at Madrid with the Government of Spain (hereinafter referred to as 'the depositary') from 1 July 1979 until and including 16 November 1979.

Ratification, acceptance or approval

Article 43

1. If ratification, acceptance or approval is required, the corresponding instrument shall be deposited with the depositary not later than 31 December 1979, but the Council may grant an extension or extensions of this time limit to any signatory government which has not deposited the instrument in question by that date.

2. Ratification, acceptance or approval shall take effect from the date on which the instrument in question is deposited or on which this Agreement enters into force, whichever is the later.

Accession

Article 44

1. The government of any non-signatory State may accede to this Agreement.

2. Accession shall be effected by the deposit of an instrument of accession with the depositary and shall take effect from the date on which such instrument is deposited or on which this Agreement enters into force, whichever is the later.

3. Any non-signatory government entitled to accede to this Agreement under paragraph 1 of this Article may notify the depositary that it undertakes to comply as rapidly as possible with the constitutional or institutional procedures required for its accession to this Agreement.

Notification of provisional application

Article 45

1. Any signatory government whose signature is subject to ratification, acceptance or approval or any non-signatory government which has made a notification under paragraph 3 of Article 44 may at any time notify the depositary that it will apply this Agreement provisionally either when it enters into force under Article 46 or, if it is already in force, on a date specified in the notification. If no date is specified in the notification of provisional application, such notification shall take effect from the date on which it is made or on which this Agreement enters into force, whichever is the later.

2. During the entire period for which this Agreement is in force either provisionally or definitively, a signatory government or a non-signatory government which has made a notification under paragraph 1 of this Article shall be a provisional member, with all the rights and obligations of a member, until the date on which its instrument of ratification, acceptance, approval or accession is deposited.

Entry into force

Article 46

1. This Agreement shall enter into force definitively on 1 January 1980, or on any date within the 12 months thereafter, between the governments which have signed it and, where their constitutional or institutional procedures so require, have ratified, accepted or approved it, or have acceded to it, if these governments include those of six countries together accounting for at least 60 % of world olive oil production in the reference period stipulated in Article 3 (1) (c) and (d). If this Agreement has not entered into force definitively in accordance with the above conditions, it shall enter into force definitively at any time after its provisional entry into force at which the requirements of the present paragraph as to number of governments and

percentage of world olive oil production are met by the deposit of instruments of ratification, acceptance, approval or accession.

2. This Agreement shall enter into force provisionally on 1 January 1980, or on any date within the 12 months thereafter, between the governments which have signed it and, where their constitutional or institutional procedures so require, have ratified, accepted or approved it, or have acceded to it or notified that they will apply it provisionally, if these governments include those of six countries together accounting for at least 60 % of world olive oil production in the reference period stipulated in Article 3 (1) (c) and (d).

3. If on 1 January 1980 this Agreement has not entered into force either provisionally or definitively in accordance with paragraphs 1 and 2 of this Article, but has received the number of signatures required for it to enter into force after ratification, acceptance or approval, the International Olive Oil Agreement, 1963 shall continue in force beyond 1 January 1980 until the date of the provisional or definitive entry into force of this Agreement, but the period of such prolongation shall not exceed 12 months.

4. If on 1 January 1980 this Agreement has not received the number of signatures required for it to enter into force after ratification, acceptance or approval, or if on 31 December 1980 it has not entered into force either provisionally or definitively in accordance with paragraphs 1 and 2 of this Article, the governments which have signed it and, where their constitutional or institutional procedures so require, have ratified, accepted or approved it, or acceded to it or notified that they will apply it provisionally, may decide by mutual agreement that this Agreement shall enter into force in whole or in part with regard to themselves, or may take whatever other decision they consider the circumstances require.

Amendment

Article 47

1. The Council may recommend to the members an amendment to this Agreement.

2. The Council shall prescribe the period within which each member shall notify the depositary whether or not it accepts the amendment.

3. If, on the date on which the period prescribed under paragraph 2 of this Article expires, the amendment has been accepted by members which together hold at least four-fifths of the total number of votes of the members having

the right to vote and include at least three-quarters of the members, it shall enter into force on that date or on such later date as the Council shall determine. If not, it shall be deemed withdrawn.

4. Any member on whose behalf no notification of acceptance of an amendment has been made by the date on which the amendment takes effect shall cease from that date to participate in this Agreement, unless such member satisfies the Council that it was unable to have the amendment accepted in time owing to difficulties in completing its constitutional or institutional procedures, and the Council decides to extend the period of acceptance for that member. The member in question shall not be bound by the amendment until it has notified the depositary of its acceptance of the amendment.

5. Any member which, during the period of validity of this Agreement, becomes a Member State of the EEC or of any other intergovernmental organization referred to in Article 3 (2) shall so notify the Council as soon as the decision on the matter has been taken, and in any event before the date on which its membership of the EEC or intergovernmental organization in question takes effect. The Council shall examine the question at its earliest succeeding session in order to negotiate with that member and the EEC or intergovernmental organization in question such appropriate adjustments as may ensue therefrom as regards the provisions of Article 18 (3), (4) and (8) (c), Article 34 and Article 35 (1). The Council may in such a case recommend an amendment pursuant to the provisions of this Article.

Withdrawal

Article 48

1. If any member considers that its interests are prejudiced, either by the fact that a signatory government whose signature is subject to ratification, acceptance or approval and which has not made a notification of provisional application of this Agreement fails to deposit its instrument of ratification, acceptance or approval, or else by the operation of the Agreement, it shall so inform the Council which shall consider the matter at its first session following the notification of the matter by the member in question. If, after the Council has examined the matter, the member concerned continues to consider that its interests are prejudiced, it may withdraw from this Agreement by giving written notice of withdrawal to the depositary.

2. Notwithstanding the provisions of paragraph 1 of this Article, any member may withdraw from this Agreement at any time after the Agreement has entered into force by giving written notice of withdrawal to the depositary.

3. Withdrawal under this Article shall take effect at the end of the calendar year in which the notification is made to the depositary.

Duration, prolongation, extension or renewal, and expiry

Article 49

1. This Agreement shall remain in force until 31 December 1984 unless it is prolonged or extended pursuant to paragraphs 2 or 4 of this Article.

2. Before the end of 1984 the Council may, by unanimous decision of the members, prolong this Agreement for a period not exceeding two calendar years. Such prolongation shall be notified by the Council to the depositary, and by the depositary to the Secretary-General of the United Nations.

3. Before the expiry of this Agreement on the date provided for in paragraph 1 of this Article or, if the Agreement is prolonged, on the date resulting from the provisions of paragraph 2 of this Article, the Council shall, at such time as it sees fit, make its recommendations to the members with regard to the extension or renewal of the Agreement.

4. If, before the expiry of this Agreement, a new agreement or a protocol for the extension of this Agreement has been negotiated and that new agreement or protocol has

received the number of signatures required for it to enter into force upon deposit of the instruments of ratification, acceptance or approval, or the requisite number of notifications of provisional application, and if that new agreement or protocol has not entered into force provisionally or definitively, this Agreement shall remain in force beyond its expiry date until the new agreement or protocol enters into force, but the period of such prolongation shall not exceed 12 months.

5. On the expiry of this Agreement and unless it is prolonged, extended or renewed, the operations for which the Council is responsible and the funds it administers shall be liquidated on terms to be established by the Council, having regard to the provisions of this Agreement. For the purposes of the application of these provisions and other conditions concerning liquidation, the Council shall continue in being as long as required and exercise the powers and functions given it under this Agreement to the full extent necessary for completing its tasks.

Authentic texts

Article 50

The texts of this Agreement in the Arabic, English, French, Italian and Spanish languages shall all be equally authentic; the originals shall be deposited with the Government of Spain.

Information relating to the signature by the Community of the International Olive Oil Agreement 1979

In accordance with the Council Decision of 12 November 1979, the Ambassador of Ireland in Madrid, duly designated to that end by the President of the Council, signed the International Olive Oil Agreement 1979 on 16 November 1979, for the purpose of binding the European Economic Community.

COMMISSION

FIRST COMMISSION DIRECTIVE

of 13 November 1979

laying down Community methods of analysis for testing coffee extracts and chicory extracts

(79/1066/EEC)

THE COMMISSION OF THE EUROPEAN
COMMUNITIES,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Having regard to the Treaty establishing the European Economic Community,

Member States shall take all measures necessary to ensure that the analyses necessary for the verification of the criteria set out in Annex I are carried out in accordance with the methods described in Annex II.

Article 2

Having regard to Council Directive 77/436/EEC of 27 June 1977 on the approximation of the laws of the Member States relating to coffee extracts and chicory extracts ⁽¹⁾, and in particular Article 8 thereof,

Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with this Directive within 18 months of its notification. They shall forthwith inform the Commission thereof.

Article 3

Whereas under Article 8 of Directive 77/436/EEC, the composition and characteristics of coffee extracts and chicory extract are required to be checked according to Community methods of analysis;

This Directive is addressed to the Member States.

Whereas a first series of methods for which studies are completed should now be adopted;

Done at Brussels, 13 November 1979.

For the Commission

Étienne DAVIGNON

Member of the Commission

Whereas the measures provided for in this Directive are in accordance with the opinion of the Standing Committee on Foodstuffs,

⁽¹⁾ OJ No L 172, 12. 7. 1977, p. 20.

ANNEX I**SCOPE OF THE FIRST COMMUNITY METHODS OF ANALYSIS DIRECTIVE FOR COFFEE
EXTRACTS AND CHICORY EXTRACTS**

- I. General provisions.
 - II. Determination of the caffeine content in decaffeinated coffee extracts using Annex II, method 1.
 - III. Determination of the dry matter content in dried coffee extract and dried chicory extract, soluble coffee and chicory, and instant coffee and chicory, using Annex II, method 2.
 - IV. Determination of the dry matter content in liquid coffee extract, liquid chicory extract, coffee extract paste and chicory extract paste using Annex II, method 3.
-

ANNEX II**METHODS OF ANALYSIS RELATING TO THE COMPOSITION OF COFFEE EXTRACTS AND CHICORY EXTRACTS****GENERAL PROVISIONS****1. PREPARATION OF THE ANALYSIS SAMPLE****1.1. General**

The mass of the sample presented to the laboratory for analysis shall be at least 50 g.

1.2. Preparation of the sample for analysis in the laboratory**1.2.1. *Mixing***

The sample for analysis shall always be mixed thoroughly before any test portion is weighed out.

1.2.1.1. Samples in powder or paste form shall be removed from the container, any lumps broken down, the sample mixed in an appropriate manner and placed in a suitable container.

1.2.1.2. Samples in liquid form shall be mixed by stirring.

1.3. Containers

The sample shall always be kept in an airtight and moisture-tight container.

2. REAGENTS**2.1. Water**

2.1.1. Wherever mention is made to water for solution, dilution or washing purposes, distilled water, or demineralized water of at least equivalent purity, shall be used.

2.1.2. Wherever reference is made to 'solution' or 'dilution' without further indication, 'solution in water' or 'dilution with water' is meant.

2.2. Chemicals

All chemicals used shall be of recognized analytical reagent quality except where otherwise specified.

3. EQUIPMENT**3.1. Lists of equipment**

The lists of equipment contain only those items with a specialized use and items with a particular specification.

3.2. Analytical balance

Analytical balance means a balance capable of weighing to at least 0.1 mg.

4. EXPRESSION OF RESULTS**4.1. Results**

The result stated in the analytical report is the mean value obtained from at least two determinations, the repeatability of which is satisfactory.

4.2. Calculation of percentage

Except where otherwise specified, the result shall be calculated as a percentage by mass of the

4.3. **Number of significant figures**

The result shall not contain more significant figures than are justified by the precision of the method of analysis used.

5. **TEST REPORT**

The test report shall identify the method of analysis used as well as the results obtained. In addition, it shall mention all details of procedure, not specified in the method of analysis, or which are optional, as well as any circumstances that may have influenced the results obtained. The test report shall give all the information necessary for the complete identification of the sample.

METHOD 1: DETERMINATION OF CAFFEINE CONTENT

1. **SCOPE AND FIELD OF APPLICATION**

This method determines the caffeine content of decaffeinated coffee extracts.

2. **DEFINITION**

Caffeine content: the content of caffeine as determined by the method specified.

3. **PRINCIPLE**

Caffeine is extracted from a test portion of the sample in an ammoniacal medium. It is then successively purified with diethyl ether on two chromatographic columns, the first in an alkaline medium and the second in an acid medium. The caffeine is then eluted from the column by chloroform and determined spectrophotometrically.

4. **REAGENTS**

4.1. Sulphuric acid, 2 M solution.

4.2. Sodium hydroxide, 2 M solution.

4.3. Celite 545 or equivalent.

4.4. Ammonia solution, approximately 4 M (prepare by adding 1 volume of concentrated ammonia solution, p_{20} ca. 0.9 g/ml to 2 volumes of water).

4.5. Diethyl ether, pure or repurified by chromatography on a column of basic aluminium oxide of activity grade 1.

Pass 800 ml of diethyl ether through a column filled with 100 g of aluminium oxide. The diethyl ether thus purified, shall be kept in dark bottles until used.

(Diethyl ether, recently distilled and free of peroxides, can be used instead of diethyl ether purified by chromatography.) Saturate the diethyl ether with water.

4.6. Caffeine (1,3,7-trimethyl-2,6-dihydroxypurine), pure, anhydrous ($C_8H_{10}N_4O_2$).

4.7. Chloroform, pure or repurified by chromatography according to the method specified in 4.5 and saturated with water.

5. **APPARATUS**

5.1. Chromatographic columns (see figure 1), approximately 250 mm long, 21 mm internal diameter (column I) and 17 mm internal diameter (column II), fitted with stopcocks.

5.2. Ultra-violet spectrophotometer: The spectrophotometer shall be accurate to within 0.004 absorbance unit within the range used.

5.3. Silica cells with 10 mm optical path length.

5.4. Usual laboratory equipment, including

- 5.4.1. Water bath, boiling.
- 5.4.2. One-mark volumetric flasks, 50 ml, 100 ml and 1 000 ml, complying with ISO 1042.
- 5.4.3. One-mark pipettes, 2 ml and 5 ml; complying with ISO 648.
- 5.4.4. Analytical balance.

6. PROCEDURE

6.1. Preparation of the test portion

Weigh, to the nearest 0.1 mg, about 0.5 g of dried coffee extract sample, between 0.5 and 0.7 g of coffee extract paste sample or between 0.8 and 3.2 g of liquid coffee extract sample. (These last two weights should be chosen to give test portions containing approximately 0.5 g of dried coffee extract.) Transfer sample to a 100 ml beaker add 5 ml of ammonia solution (4.4) and warm for two minutes on the boiling water bath (5.4.1). Add 6 g of Celite (4.3) and mix carefully.

6.2. Filling of the columns

6.2.1. Column I (alkaline column)

Layer A. Mix carefully, by kneading with a flexible spatula blade, 3 g of Celite (4.3) and 2 ml of the sodium hydroxide solution (4.2), until homogeneous (see note below). A slightly wet powder will be obtained. Transfer this powder, in small portions (of approximately 2 g), into a chromatographic column (5.1), the lower part of which is packed with a wad of cotton wool or glass wool. Tamp down the mixture after each addition, without excessive force, using a glass rod, one end of which is flattened to the internal diameter of the column, until a perfectly homogeneous and compact layer is obtained. A small wad of cotton wool or glass wool may be placed on the top of layer A.

Note: Column packing material may be prepared in bulk quantities in advance and stored in closed containers.

Layer B. Transfer the Celite-sample mixture (6.1) into the column upon layer A. Dry the sample beaker twice with portions of about 1 g of Celite (4.3) transferring this Celite into the column. Tamp down to obtain a homogeneous layer and place a wad of cotton wool or glass wool on the top of layer B.

6.2.2. Column II (acid column)

Place in a second column, the lower part of which is packed with a wad of cotton wool or glass wool, 3 g of Celite (4.3) and 3 ml of the sulphuric acid solution (4.1), carefully mixed as directed for layer A in 6.2.1 (see the note in that sub-clause). Place a wad of cotton wool or glass wool on the top of the layer to prevent damage to the top of the column packing.

6.3. Chromatography

Mount the columns one above the other so that the effluent from column I can drip directly into column II: Pass 150 ml of diethyl ether (4.5) through the two columns. Keep open the stopcock in column I. Adjust the stopcock of column II so that a quantity of supernatant liquid remains above the column packing. Remove column I. Pass 50 ml of diethyl ether (4.5) through column II, using the initial portion to wash the tip of column I and passing this portion also into column II. Discard the effluent from column II.

Note: Used diethyl ether may be recovered by shaking it with iron (II) sulphate.

Pass a stream of air through column II with the stopcock open (for example by using an inflated rubber blower), until no more diethyl ether drips from the column and the air flow from the stopcock carries only a faint smell of diethyl ether (see note below). Elute column II with 45 to 50 ml of chloroform (4.7). Collect the eluate in a 50 ml volumetric flask (5.4.2), make up to volume with chloroform (4.7) and mix carefully.

The flow rate of the diethyl ether and the chloroform under conditions of natural flow should be between 1.5 and 3 ml/min. If this rate is exceeded, channelling through the packing should be suspected.

Note: This step should be carried out in a well-ventilated fume-cupboard to prevent both the possibility of inhalation of solvent vapours and the possibility of an explosion.

6.4. Spectrophotometric measurement (see figure 2)

6.4.1. *Measurement of the test solution*

Avoiding error from chloroform evaporation, measure the absorbance of the solution of caffeine in chloroform (6.3) using silica cells (5.3), against chloroform (4.7) at 276 nm (absorption maximum). Measure also the absorbance at 246 nm (absorption minimum) and at 306 nm in order to verify the purity of the caffeine obtained.

If the absorbance at 276 nm exceeds 1.3, repeat the measurement on a diluted portion of the test solution. In this case, take into account the dilution factor; the appropriate factors of the formulae in 7.1 will have to be adjusted accordingly. If the absorbance measured at 276 nm is lower than 0.2, repeat the determination using a larger test portion.

6.4.2. *Preparation and measurement of a reference solution*

Prepare a reference solution of caffeine in the following manner:

Weigh, to the nearest 0.1 mg; 100 ± 20 mg of pure anhydrous caffeine (4.6). Place in a 1 000 ml volumetric flask (5.4.2), dissolve in chloroform, and make up to volume. With a volumetric pipette (5.4.3), take 5 ml of this solution and make up the volume to 50 ml with chloroform.

Measure the absorbance of this solution as described in 6.4.1. The corrected absorbance of the reference solution should be in the region of 0.4.

6.5. Number of determinations

Carry out at least two determinations on the same test sample.

6.6. Blank test

Carry out a blank test on the reagents following the procedure described above but omitting the test portion. Before using recovered reagents (see 4.5 and 4.7), repeat the blank test to verify their purity.

7. EXPRESSION OF RESULTS

7.1. Formulae and method of calculation

The caffeine content, in percent by mass of dry matter on the sample, is equal to:

$$\frac{5 \times 10^3 \times C \times A_1}{A_2 \times m \times p}$$

where:

C is the concentration of caffeine in the reference solution (6.4.2) in g/ml;

*A*₁ is the corrected absorbance of the purified extract (6.4.1) [i.e. absorbance at 276 nm – 0.5x (absorbance at 246 nm + absorbance at 306 nm)];

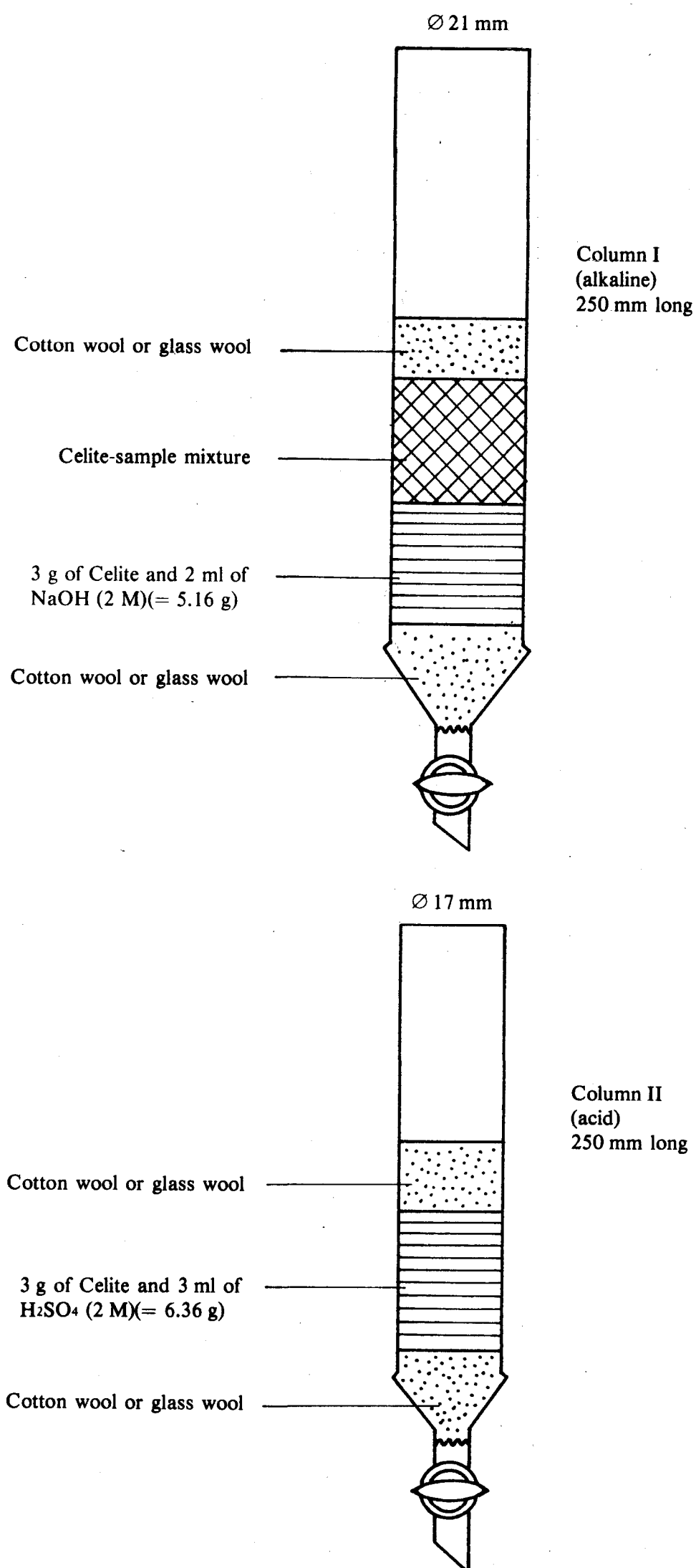
*A*₂ is the corrected absorbance of the caffeine reference solution (6.4.2) [i.e. absorbance at 276 nm – 0.5x (absorbance at 246 nm + absorbance at 306 nm)];

m is the mass in g of the test portion;

p is the dry matter content, expressed as a percentage by mass, of the sample, as determined by methods 2 or 3, Annex II.

7.2. Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.01 g caffeine per 100 g sample on a dry matter basis.

*Figure 1*

Chromatographic columns

Cells: silica with 10 mm optical path length
Solvent: chloroform
Blank: chloroform

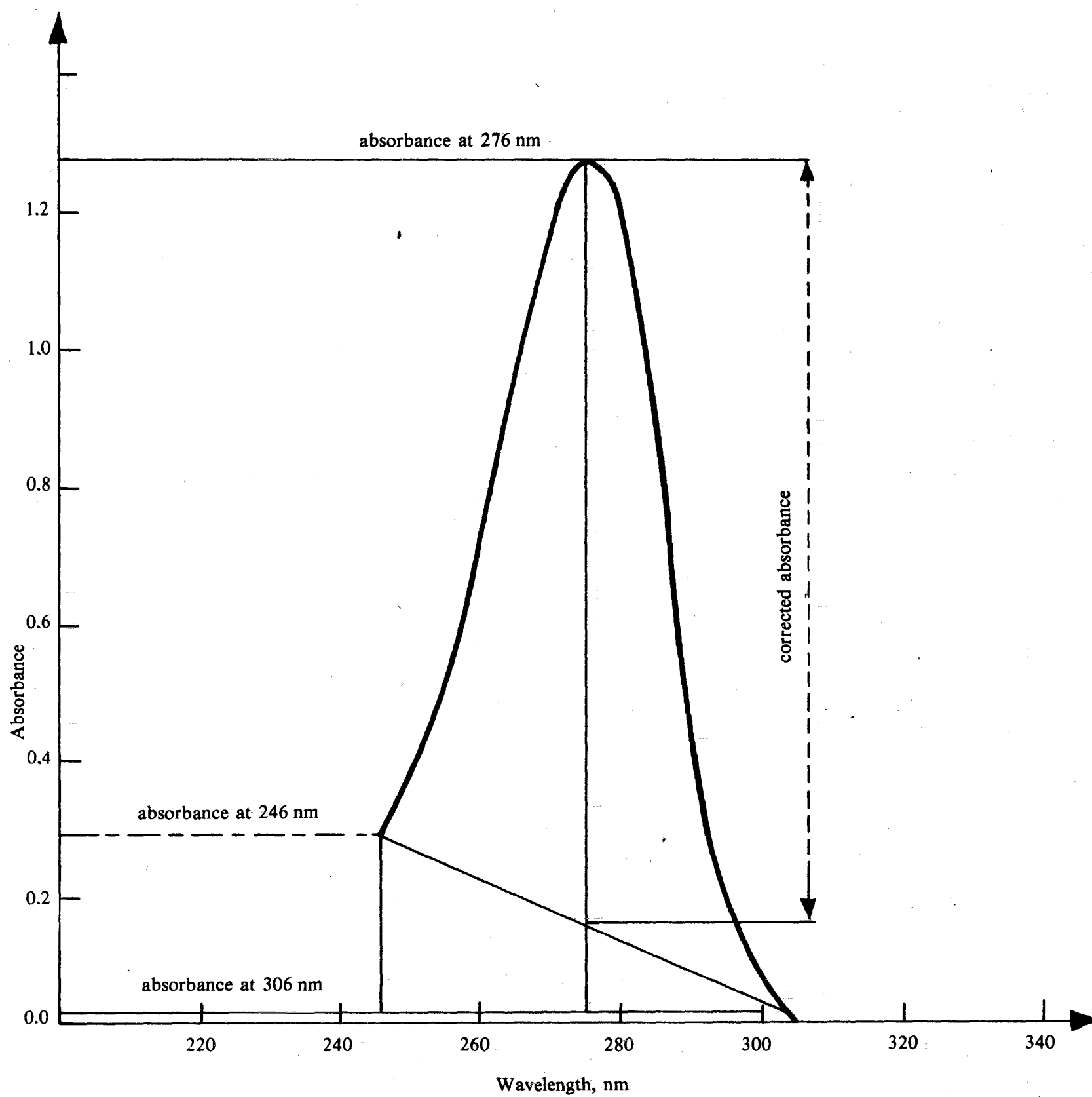


Figure 2
Spectrophotometric measurement

METHOD 2: DETERMINATION OF DRY MATTER CONTENT**1. SCOPE AND FIELD OF APPLICATION**

This method determines the dry matter content of:

- dried coffee extract, soluble coffee, instant coffee,
- dried chicory extract, soluble chicory, instant chicory.

2. DEFINITION

Dry matter content: the content of dry matter as determined by the method specified.

3. PRINCIPLE

The residual mass of a test portion is determined after drying for 16 hours in a vacuum oven at a temperature of 70 °C and a pressure of 5.0 kPa and calculated as a percentage by mass of the sample.

4. APPARATUS

- 4.1. Weighing dishes, flat bottomed, resistant to attack by the sample and condition of the test, approximate dimensions 50 mm diameter by 30 mm height with closely fitting lids. Aluminium and stainless steel dishes are suitable.
- 4.2. Vacuum oven, electrically heated, temperature controlled by thermostat at $70\text{ °C} \pm 1\text{ °C}$ throughout the volume of the oven equipped with a thermometer with a certificate of accuracy at 70 °C, indicating the temperature in the immediate vicinity of the shelf and a gauge indicating the internal pressure in kPa above zero pressure. This oven should have a uniform internal temperature distribution. The shelves shall be constructed and fitted so as to ensure good heat transfer to the dishes (4.1).
- 4.3. Drying oven, electrically heated, temperature controlled by thermostat at $102\text{ °C} \pm 2\text{ °C}$ throughout the volume of the oven.
- 4.4. Vacuum pump capable of evacuating the vacuum oven (4.2) to an internal pressure of 5.0 kPa or less.
- 4.5. Air drying train, consisting of two glass washing bottles filled with glycerol to form a bubble train and two glass drying towers filled with freshly activated silica gel with a moisture content indicator. The bubble train and drying trains are connected in series with the vacuum oven (4.2) with the drying towers between the oven and the bubble train.
- 4.6. Desiccator, containing freshly activated silica gel (or an equivalent desiccant) with a moisture content indicator.
- 4.7. Analytical balance.

5. PROCEDURE**5.1. Preparation of the dishes**

Place the clean dry empty dishes and lids (4.1) in a drying oven (4.3) set at $102\text{ °C} \pm 2\text{ °C}$ for one hour. Covers should be placed beside dishes in order to expose all surfaces for drying.

Remove dishes and lids from the oven and place in a desiccator (4.6). Allow to cool and weigh dish and matching lid to the nearest 0.1 mg (M_0).

5.2. Test portion

Remove the lid from the prepared dish (5.1). As rapidly as possible place approximately 3 g of sample in dish and spread uniformly over the bottom of the dish. Cover dish with its matching lid and weigh with its contents to the nearest 0.1 mg (M_2). If more than one weighing is to be made place covered dishes in desiccator until all samples have been weighed and are ready to be placed in oven.

- 5.3. Place the dish and its lid separately in the vacuum oven (4.2).
- 5.4. Close the oven and reduce the pressure slowly (at least 2 to 2.5 minutes), to 5.0 ± 0.1 kPa.
- 5.5. Allow dry air to enter the oven slowly through the drying towers and bubble train (4.5) at about one bubble per second as observed in the liquid in the bubble train.
- 5.6. Dry in the vacuum oven at $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 16 ± 0.5 hours maintaining the air stream.
- 5.7. At the end of the drying period allow the air to enter the oven slowly (two to three minutes) to prevent air turbulence that might cause some sample to be lost from dish.
Replace lid on matching dish and place covered dish in desiccator (4.6) and allow to cool to ambient temperature.
- 5.8. Weigh, to 0.1 mg, covered dish and contents (M_1).

6. EXPRESSION OF RESULTS

6.1. Formula and method of calculation

The content of dry matter calculated as a percentage by mass of the prepared sample, as given by:

$$\frac{M_1 - M_0}{M_2 - M_0} \times 100$$

where:

M_0 = mass of the dry prepared dish and lid;

M_1 = mass of the dish lid and test portion after drying;

M_2 = mass of the dish, lid and test portion before drying.

Take as the result the arithmetic mean of the results of two determinations provided that the evaluation concerning repeatability (6.2) is satisfied.

6.2. Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.06 g dry matter per 100 g of sample.

METHOD 3: DETERMINATION OF DRY MATTER CONTENT

1. SCOPE AND FIELD OF APPLICATION

This method determines the dry matter content of:

- liquid coffee extract,
- liquid chicory extract,
- coffee extract paste,
- chicory extract paste.

2. DEFINITION

Dry matter content: the content of dry matter as determined by the method specified.

3. PRINCIPLE

Test portions of the samples are mixed with sea sand and then dried for 16 hours in a vacuum oven at a temperature of 70°C and a pressure of 5 kPa. The residual mass is calculated as a percentage by mass of the sample.

4. REAGENTS

Sea sand, washed in acid and then water till acid free and then ignited.

5. APPARATUS

5.1. Weighing dishes, flat bottomed, resistant to attack by the sample and condition of the test, approximately 80 mm in diameter and with closely fitting lids.

5.2. Glass rods of such a length that they lie wholly in the weighing dishes (5.1) for example 50 to 75 mm long.

5.3. Vacuum oven, electrically heated, temperature controlled by thermostat at $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$ throughout the volume of the oven, equipped with a thermometer with a certificate of accuracy at 70°C , indicating the temperature in the immediate vicinity of the shelf and a gauge indicating the internal pressure in kPa above zero pressure.

This oven shall have a uniform internal temperature distribution. The shelves shall be constructed and fitted so as to ensure good heat transfer to the dishes (5.1).

5.4. Vacuum pump capable of evacuating the vacuum oven (5.3) to an internal pressure of 5.0 kPa or less.

5.5. Air drying train consisting of two glass washing bottles filled with glycerol to form a bubble train and two glass drying tubes filled with freshly activated silica gel with a moisture content indicator. The bubble train towers are connected in series with the vacuum oven (5.3) with the drying towers between the oven and the bubble train.

5.6. Desiccator containing freshly activated silica gel (or an equivalent desiccant) with a moisture content indicator.

5.7. Analytical balance.

5.8. Water bath boiling.

6. PROCEDURE

6.1. Preparation of weighing dish

Place 25 to 35 g of sea sand (4) in a weighing dish (5.1) together with a glass rod (5.2) and weigh. Place the dish with sea sand lid and rod in the vacuum oven (5.3).

The lid should be placed beside the dish in order to expose all surfaces for drying.

Remove the dish, with contents, and its lid from the oven and place in a desiccator (5.6).

Allow to cool and weigh, to the nearest 0.1 mg, dish, contents and matching lid.

Repeat till constant weight is obtained (M_0).

6.2. Test portion

Remove the lid from the prepared dish (6.1). Add (as rapidly as possible) a portion of the sample with a dry matter content which should correspond to 0.1 to 1 g.

Weigh, to 0.1 mg, dish, contents, including test portion, and lid (M_2).

6.3. Carefully mix the sea sand and the sample with the glass rod (5.2). If mixing is not carried out satisfactorily add a little water to aid the mixing. Re-heat on a water bath (5.8) stirring occasionally until a completely homogeneous sandy mixture is obtained. Should the mixture tend to become lumpy or crusty, stir or pound continuously so as to prevent any lumpiness.

6.4. Place the dish and its lid separately in the vacuum oven (5.3).

6.5. Close the oven and reduce the pressure slowly (at least 2 to 2.5 minutes) to 5.0 ± 0.1 kPa.

6.6. Allow dry air to enter the oven slowly through the drying towers and bubble train (5.5) at about one bubble per second as observed in the liquid of the bubble train.

- 6.7. Dry in the vacuum oven at $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 16 ± 0.5 hours maintaining the air stream.
- 6.8. At the end of the drying period allow the air to enter the oven slowly (two to three minutes) to prevent air turbulence that might cause some sample to be lost from the dish.
Replace lid on matching dish and place covered dish in desiccator (5.6) and allow to cool to ambient temperature.
- 6.9. Weigh, to 0.1 mg, covered dish and contents (M_1).

7. EXPRESSION OF RESULTS

7.1. Formula and method of calculation

The content of dry matter, calculated as a percentage by mass of the prepared sample, is given by:

$$\frac{M_1 - M_0}{M_2 - M_0} \times 100$$

where:

M_0 = the mass of the prepared dish, and lid;

M_1 = the mass of the dish, lid and test portion after drying;

M_2 = the mass of the dish, lid and test portion before drying.

Take as a result the arithmetic mean of the results of two determinations provided that the evaluation concerning repeatability (7.2) is satisfied.

7.2. Repeatability

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.06 g dry matter per 100 g of sample.

FIRST COMMISSION DIRECTIVE**of 13 November 1979****laying down Community methods of analysis for testing certain partly or wholly dehydrated preserved milk for human consumption****(79/1067/EEC)****THE COMMISSION OF THE EUROPEAN
COMMUNITIES,**

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 76/118/EEC of 18 December 1975 on the approximation of the laws of the Member States relating to certain partly or wholly dehydrated preserved milk for human consumption ⁽¹⁾, and in particular Article 11 thereof,

Whereas under Article 11 of Directive 76/118/EEC, the composition of certain partly or wholly dehydrated preserved milk is required to be verified by Community methods of analysis;

Whereas it is desirable to adopt an initial series of methods in respect of which studies have been completed;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Standing Committee of Foodstuffs,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Member States shall take all measures necessary to ensure that the analyses necessary for verification of the criteria set out in Annex I are carried out in accordance with the methods described in Annex II.

Article 2

Where alternative methods for a single determination are specified, the sample may be analysed by either method. The test report referred to in Annex II must state the method which has been employed.

Article 3

Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with this Directive within 18 months of its notification. They shall forthwith inform the Commission thereof.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 13 November 1979.

For the Commission

Étienne DAVIGNON

Member of the Commission

⁽¹⁾ OJ No L 24, 30. 1. 1976, p. 49.

ANNEX I**SCOPE OF THE FIRST COMMUNITY METHODS OF ANALYSIS FOR CERTAIN PARTLY OR WHOLLY DEHYDRATED PRESERVED MILK DIRECTIVE**

- I. General provisions**
- II. Determination of dry matter in:**
- unsweetened condensed high fat milk (using method 1, Annex II),
 - unsweetened condensed milk (using method 1, Annex II),
 - unsweetened condensed partly skimmed milk (using method 1, Annex II),
 - unsweetened condensed skimmed milk (using method 1, Annex II),
 - sweetened condensed milk (using method 1, Annex II),
 - sweetened condensed partly skimmed milk (using method 1, Annex II),
 - sweetened condensed skimmed milk (using method 1, Annex II).
- III. Determination of moisture in:**
- dried high fat milk or high fat milk powder (using method 2, Annex II),
 - dried whole milk or whole milk powder (using method 2, Annex II),
 - dried partly skimmed milk or partly skimmed-milk powder (using method 2, Annex II),
 - dried skimmed milk or skimmed-milk powder (using method 2, Annex II).
- IV. Determination of fat in:**
- unsweetened condensed high fat milk (using method 3, Annex II),
 - unsweetened condensed milk (using method 3, Annex II),
 - unsweetened condensed partly skimmed milk (using method 3, Annex II),
 - unsweetened condensed skimmed milk (using method 3, Annex II),
 - sweetened condensed milk (using method 3, Annex II),
 - sweetened condensed partly skimmed milk (using method 3, Annex II),
 - sweetened condensed skimmed milk (using method 3, Annex II),
 - dried high fat milk or high fat milk powder (using method 4, Annex II),
 - dried whole milk or whole milk powder (using method 4, Annex II),
 - dried partly skimmed milk or partly skimmed-milk powder (using method 4, Annex II),
 - dried skimmed milk or skimmed-milk powder (using method 4, Annex II).
- V. Determination of sucrose in:**
- sweetened condensed milk (using method 5, Annex II),
 - sweetened condensed partly skimmed milk (using method 5, Annex II),
 - sweetened condensed skimmed milk (using method 5, Annex II).
- VI. Determination of lactic acid and lactates in:**
- dried high fat milk or high fat milk powder (using method 6, Annex II),
 - dried whole milk or whole milk powder (using method 6, Annex II),
 - dried partly skimmed milk or partly skimmed-milk powder (using method 6, Annex II),
 - dried skimmed milk or skimmed-milk powder (using method 6, Annex II).
- VII. Determination of phosphatase activity in:**
- dried high fat milk or high fat milk powder (using method 7 or 8, Annex II),
 - dried whole milk or whole milk powder (using method 7 or 8, Annex II),
 - dried partly skimmed milk or partly skimmed-milk powder (using method 7 or 8, Annex II),
 - dried skimmed milk or skimmed-milk powder (using method 7 or 8, Annex II).

ANNEX II**METHODS OF ANALYSIS RELATING TO THE COMPOSITION OF CERTAIN PARTLY OR WHOLLY DEHYDRATED PRESERVED MILK PRODUCTS INTENDED FOR HUMAN CONSUMPTION****GENERAL PROVISIONS****1. PREPARATION OF THE SAMPLE FOR CHEMICAL ANALYSIS**

- 1.1. Unsweetened condensed high fat milk**
Unsweetened condensed milk
Unsweetened condensed partly skimmed milk
Unsweetened condensed skimmed milk

Shake and invert the closed can. Open the can and slowly pour the milk into a second container which can be closed hermetically, mixing by repeated transfer. Ensure that all remaining fat and milk adhering to the wall and the ends of the can are mixed in with the sample. Close the container. If the contents are not homogeneous warm the container in a waterbath at 40 °C. Shake vigorously every 15 minutes. After two hours, remove the container from the waterbath and let it cool to room temperature. Remove the lid and thoroughly mix the contents of the container with a spoon or spatula (if the fat has separated the sample should not be tested). Store in a cool place.

- 1.2. Sweetened condensed milk**
Sweetened condensed partly skimmed milk
Sweetened condensed skimmed milk

Cans: Warm the closed can in a waterbath at 30 to 40 °C for about 30 minutes. Open the can and thoroughly mix the contents with a spatula or a spoon by making upward, downward, and circular movements in order to obtain an intimate mixture of the top and bottom layers with all the contents. Ensure that the remaining milk adhering to the wall and end of the can is incorporated in the sample. As far as possible, pour the contents into a second container provided with an air-tight lid. Close the container and store in a cool place.

Tubes: Cut the end and pour the contents into a container provided with an air-tight lid. Next, cut the tube lengthwise. Scrape out all material adhering to the interior and mix it carefully with the rest of the contents. Store the container in a cool place.

- 1.3. Dried high fat milk or high fat milk powder**
Dried whole milk or whole milk powder
Dried partly skimmed milk or partly skimmed-milk powder
Dried skimmed milk or skimmed-milk powder

Transfer the milk powder to a clean, dry container (with air-tight lid) of a capacity of twice the volume of the powder. Close the container immediately and thoroughly mix the milk powder by repeatedly shaking and inverting the container. During the preparation of the sample exposure of the milk powder to the atmosphere should be avoided as far as possible to minimize absorption of moisture.

2. REAGENTS**2.1. Water**

- 2.1.1.** Wherever mention is made of water for dissolution, dilution or washing purposes, distilled water, or demineralized water of at least equivalent purity shall be used.

- 2.1.2.** Wherever reference is made to 'dissolution', 'solution' or 'dilution' without further indication, 'dissolution in water', 'solution in water' and 'dilution in water' is meant.

2.2. Chemicals

All chemicals used shall be of recognized analytical reagent quality except where otherwise specified.

3. EQUIPMENT**3.1. Lists of equipment**

The lists of equipment contain only those items with a specialized use and items with a particular specification.

3.2. Analytical balance

Analytical balance means a balance capable of weighing to at least 0.1 mg.

4. EXPRESSION OF RESULTS**4.1. Calculation of percentage**

Except where otherwise specified, the results shall be calculated as a percentage by mass of the sample as received by the laboratory.

4.2. Number of significant figures

The result shall not contain more significant figures than are justified by the precision of the method of analysis used.

5. TEST REPORT

The test report shall identify the method of analysis used as well as the results obtained. In addition, it shall mention all details of procedure not specified in the method of analysis, or which are optional, as well as any circumstances that may have influenced the results obtained.

The test report shall give all the information necessary for the complete identification of the sample.

METHOD 1: DETERMINATION OF DRY MATTER CONTENT

(oven 99 °C)

1. SCOPE AND FIELD OF APPLICATION

This method determines the dry matter content of:

- unsweetened condensed high fat milk,
- unsweetened condensed milk,
- unsweetened condensed partly skimmed milk,
- unsweetened condensed skimmed milk,
- sweetened condensed milk,
- sweetened condensed partly skimmed milk,
- sweetened condensed skimmed milk.

2. DEFINITION

The dry matter content of condensed milks: dry matter content as determined by the method specified.

3. PRINCIPLE

A known amount of the sample is diluted with water, mixed with sand and dried at a temperature of $99\text{ °C} \pm 1\text{ °C}$. The mass after drying is the mass of dry matter and is calculated as a percentage by mass of the sample.

4. REAGENTS

Quartz sand or sea sand, treated with hydrochloric acid (size of the grains: 0.18 to 0.5 mm, that is passing through a 500 micron sieve and retained by a 180 micron sieve). It should meet the following control test:

Heat about 25 g of sand for two hours in the drying oven (5.3) as described in 6.1. to 6.3. Add 5 ml of water, heat again in the oven for two hours, cool and reweigh. The difference between the two masses should not exceed 0.5 mg.

If necessary treat the sand with a 25 % hydrochloric acid solution for three days, mixing occasionally. Wash with water until the acid reaction disappears or the wash water is chloride free. Dry at 160 °C and re-test as above.

5. APPARATUS

- 5.1. Analytical balance.
- 5.2. Metal dishes, preferably of nickel, aluminium or stainless steel. The dishes must have lids which fit very well but which are readily removed. Suitable dimensions are: diameter 60 to 80 mm and depth about 25 mm.
- 5.3. Atmospheric-pressure drying oven, well ventilated, thermostatically controlled with temperature regulated at $99^{\circ}\text{C} \pm 1^{\circ}\text{C}$. The temperature should be uniform throughout the oven.
- 5.4. Desiccator, containing freshly activated silica gel with a water content indicator or an equivalent desiccant.
- 5.5. Glass rods, flattened at one end of such a length that they can fit inside the metal dishes (5.2).
- 5.6. Waterbath, boiling.

6. PROCEDURE

- 6.1. Place about 25 g sand (4) and a short glass rod (5.5) in the dish (5.2).
- 6.2. Without covering the dish and contents with the lid, place the dish, contents and lid in the oven (5.3) and heat for two hours.
- 6.3. Replace lid and transfer the dish to the desiccator (5.4). Allow to cool to room temperature and accurately weigh to the nearest 0.1 mg (M_0).
- 6.4. Tilt the sand to one side of the dish. Introduce into the clear space about 1.5 g of the sample of sweetened condensed milk and 3.0 g of unsweetened condensed milk. Replace the lid and accurately weigh to the nearest 0.1 mg (M_1).
- 6.5. Remove the lid, add 5 ml of water and, with the aid of the glass rod, mix the liquids and then the sand and the liquid portion. Leave the rod in the mixture.
- 6.6. Place the dish on a boiling waterbath (5.6) until the water has evaporated; this usually takes 20 minutes. Stir the mixture from time to time with the rod to keep the mass well aerated so that the mass when dry does not form a cake. Lay the rod inside the dish.
- 6.7. Place the dish and lid in the oven for one and a half hours.
- 6.8. Replace the lid, transfer the dish to the desiccator (5.4), allow to cool to room temperature and accurately weigh to the nearest 0.1 mg.
- 6.9. Replace the dish and lid in the oven, uncover the dish and heat it with its lid for a further hour.
- 6.10. Repeat process 6.8.
- 6.11. Repeat the described processes 6.9 and 6.10 until the difference in mass of two successive weighings is less than 0.5 mg or until the mass increases. If an increase in mass occurs use the lowest mass obtained in the calculation (7.1). Let the final weight recorded be M_2 g.

7. EXPRESSION OF RESULTS**7.1. Method of calculation**

The content of dry matter, calculated as a percentage by mass of the sample, is given by:

$$\frac{M_2 - M_0}{M_1 - M_0} \times 100$$

where:

M_0 = mass, in g of the dish, lid and sand after process 6.3;

M_1 = mass, in g of the dish, lid, sand and sample after process 6.4;

M_2 = mass, in g of the dish, lid, sand and dried sample after process 6.11.

7.2. Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.2 g of dry matter per 100 g of the product.

8. CALCULATION OF TOTAL MILK SOLIDS AND MILK SOLIDS NOT FAT**8.1. The total milk solids content of the sweetened condensed milk is given by:**

Total dry matter (obtained by method 1, Annex II) — sucrose (obtained by method 5, Annex II).

8.2. The milk solids not fat content of the sweetened condensed milks is given by:

Total dry matter (obtained by method 1, Annex II) — (sucrose content obtained by method 5, Annex II) and fat content (obtained by method 3, Annex II).

8.3. The milk solids not fat content of unsweetened condensed milks is given by:

Total dry matter (obtained by method 1, Annex II) — fat content (obtained by method 3, Annex II).

METHOD 2: DETERMINATION OF MOISTURE
(oven 102 °C).**1. SCOPE AND FIELD OF APPLICATION**

This method determines the loss of mass on drying of:

- dried high fat milk or high fat milk powder,
- dried whole milk or whole milk powder,
- dried partly skimmed milk or partly skimmed-milk powder,
- dried skimmed milk or skimmed-milk powder.

2. DEFINITION

Moisture content: the loss of mass on drying as determined by the method specified.

3. PRINCIPLE

The residual mass of a test portion is determined after drying at atmospheric pressure in an oven at 102 °C ± 1 °C to constant mass. The loss of mass is calculated as a percentage by mass of the sample.

4. APPARATUS

- 4.1. Analytical balance.
- 4.2. Dishes, preferably of nickel, aluminium, stainless steel or glass. The dishes must have lids which fit very well but which can readily be removed. Suitable dimensions are: diameter 60 to 80 mm and depth about 25 mm.
- 4.3. Atmospheric-pressure drying oven, well ventilated, thermostatically controlled with temperature regulation (at $102^{\circ}\text{C} \pm 1^{\circ}\text{C}$). The temperature should be uniform throughout the oven.
- 4.4. Desiccator, containing freshly activated silica gel with a water content indicator or an equivalent desiccant.

5. PROCEDURE

- 5.1. Uncover the dish (4.2) and place it and its lid in the oven (4.3) and heat for about one hour.
- 5.2. Place the lid on the dish and transfer the covered dish to the desiccator (4.4). Allow it to cool to room temperature and accurately weigh to the nearest 0.1 mg (M_0).
- 5.3. Introduce approximately 2 g of dried milk sample into the dish, cover the dish with the lid and accurately weigh to the nearest 0.1 mg the covered dish as quickly as possible (M_1).
- 5.4. Uncover the dish and put it with its lid in the oven for two hours.
- 5.5. Replace the lid, transfer the covered dish to the desiccator, allow it to cool to room temperature and accurately weigh to the nearest 0.1 mg as quickly as possible.
- 5.6. Uncover the dish and heat it and its lid for one hour in the oven.
- 5.7. Repeat process 5.5.
- 5.8. Repeat processes 5.6 and 5.5 until the decrease in mass between the successive weighings does not exceed 0.5 mg or until the mass increases. If an increase in mass occurs use the lowest mass obtained in the calculation (6.1). Let the final weight recorded be M_2 g.

6. EXPRESSION OF RESULTS**6.1. Method of calculation**

Calculate the loss of mass on drying of the sample, expressed as a percentage by mass, by the formula:

$$\frac{M_1 - M_2}{M_1 - M_0} \times 100$$

where:

M_0 = mass, in g of the dish and its lid after process 5.2;

M_1 = mass, in g of the dish, its lid and sample after process 5.3;

M_2 = mass, in g of the dish, its lid and final sample after process 5.5.

6.2. Repeatability

The difference in results between two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.1 g of moisture per 100 g of product.

METHOD 3: DETERMINATION OF FAT CONTENT IN CONDENSED MILKS (RÖSE-GOTTLIEB METHOD)**1. SCOPE AND FIELD OF APPLICATION**

This method determines the fat content of:

- unsweetened condensed high fat milk,
- unsweetened condensed milk,
- unsweetened condensed partly skimmed milk,
- unsweetened condensed skimmed milk,
- sweetened condensed milk,
- sweetened condensed partly skimmed milk,
- sweetened condensed skimmed milk.

2. DEFINITION

The fat content of condensed milks: fat content as determined by the method specified.

3. PRINCIPLE

The fat content is determined by extraction of the fat from an ammoniacal alcoholic solution of the sample with diethyl ether and light petroleum followed by evaporation of the solvents and weighing of the residue and calculation as a percentage by mass of the sample, according to the principle of Röse-Gottlieb.

4. REAGENTS

All reagents should conform to the requirements specified in the blank test (6.1). If necessary, reagents may be redistilled in the presence of about 1 g of butterfat for 100 ml of solvent.

4.1. Ammonia solution, approximately 25 % (m/m) NH_3 (density at 20 °C approximately 0.91 g/ml), or a stronger solution of known concentration.

4.2. Ethanol, 96 ± 2 % (v/v) or, if not available, ethanol denatured with methanol, ethyl methyl ketone or light petroleum.

4.3. Diethyl ether, peroxide-free.

Note 1:

To test for peroxides, add to 10 ml of the ether in a small glass stoppered cylinder, previously rinsed with the ether, 1 ml freshly prepared 10 % potassium iodide solution. Shake and let stand for one minute. No yellow colour should be observed in either layer.

Note 2:

Diethyl ether may be maintained free from peroxides by adding wet zinc foil that has been completely immersed in dilute acidified copper sulphate solution for one minute and subsequently washed with water. Use per litre approximately 8 000 mm² zinc foil; cut in strips long enough to reach at least halfway up the container.

4.4. Light petroleum (petroleum ether), with any boiling range between 30 and 60 °C.

4.5. Mixed solvent, prepared shortly before use by mixing equal volume of diethyl ether (4.3) and light petroleum (4.4) (where the use of mixed solvent is indicated, it may be replaced by either diethyl ether or light petroleum alone).

5. APPARATUS

5.1. Analytical balance.

5.2. Suitable extraction tubes or flasks, provided with ground glass stoppers or other closures unaffected by the solvents used.

5.3. Flasks, thin-walled and flat-bottomed, 150 to 250 ml capacity.

- 5.4. Atmospheric pressure drying oven, well ventilated and thermostatically controlled (adjusted to operate at $102^{\circ}\text{C} \pm 1^{\circ}\text{C}$).
- 5.5. Anti-bumping granules, fat-free, non porous, non friable in use, e.g. glass beads or pieces of silicon carbide (the use of this material is optional; see clause 6.2.1).
- 5.6. Siphon, to fit extraction tubes.
- 5.7. Centrifuge (optional).

6. PROCEDURE

6.1. Blank test

At the same time as the determination of the fat content of the sample, carry out a blank determination on 10 ml of water using the same type of extraction apparatus, the same reagents in the same amounts and the same procedure as described hereafter, excluding clause 6.2.2. If the blank exceeds 0.5 mg, the reagents should be checked and the impure reagent or reagents should be purified or replaced.

6.2. Determination

- 6.2.1. Dry a flask (5.3) (together with, if required, some anti-bumping granules (5.5) to promote gentle boiling during the subsequent removal of the solvents) in the oven (5.4) for half to one hour. Allow the flask to cool to the temperature of the balance room and accurately weigh the cooled flask to the nearest 0.1 mg.
- 6.2.2. Stir the prepared sample and immediately weigh, to the nearest 1 mg, 2 to 2.5 g of the sample if sweetened or 4 to 5 g of the sample if unsweetened directly in, or by difference into, the extraction apparatus (5.2). Add water to 10.5 ml and shake gently with slight warming (40 to 50°C) until the product is completely dispersed. The sample must be dispersed completely otherwise the determination should be repeated.
- 6.2.3. Add 1.5 ml ammonia (25 %) (4.1) or a corresponding volume of a stronger solution, and mix well.
- 6.2.4. Add 10 ml ethanol (4.2) and mix the liquids gently but thoroughly in the unclosed apparatus.
- 6.2.5. Add 25 ml diethyl ether (4.3). Cool under running water. Close the apparatus and shake vigorously and invert repeatedly for one minute.
- 6.2.6. Remove the stopper carefully and add 25 ml light petroleum (4.4) using the first few millilitres to rinse the stopper and inside of the neck of the apparatus, allowing the rinsings to run into the apparatus. Close by replacing the stopper and shake and invert repeatedly for 30 seconds. Do not shake too vigorously if centrifuging is not to be used in 6.2.7.
- 6.2.7. Allow the apparatus to stand until the upper liquid layer has become clear and has distinctly separated from the lower aqueous layer. Alternatively carry out the separation using a suitable centrifuge (5.7).

Note:

When a centrifuge which is not driven by a three-phase motor, is used, sparks may occur and care must therefore be taken to avoid an explosion or fire from any ether vapours, coming, for example, from a broken tube.

- 6.2.8. Remove the stopper, rinse it and the inside of the neck of the apparatus with a few millilitres of mixed solvent (4.5) and allow the rinsings to run into the apparatus. Carefully transfer as much as possible of the supernatant layer by decantation or by means of a siphon (5.6) into the prepared flask (6.2.1).

Note:

If the transfer is not made using a siphon, it may be necessary to add a little water in order to raise the interface between the two layers thus aiding decantation.

- 6.2.9. Rinse the outside and the inside of the neck of the apparatus or the tip and the lower part of the siphon with a few millilitres of mixed solvent (4.5). Allow the rinsings from the outside of the ap-

paratus to run into the flask and the rinsings from the inside of the neck and from the siphon to run into the extraction apparatus.

6.2.10. Make a second extraction by repeating the procedure of 6.2.5 to 6.2.9 inclusive but using only 15 ml diethyl ether and 15 ml light petroleum.

6.2.11. Make a third extraction by repeating the procedure of 6.2.10 but omit the final rinsing (6.2.9).

Note:

It is not mandatory to carry out this third extraction when analysing skimmed unsweetened condensed milk and skimmed sweetened condensed milk samples.

6.2.12. Carefully evaporate or distil off as much solvent (including the ethanol) as possible. If the flask is of small capacity, it will be necessary to remove some of the solvent as above after each extraction.

6.2.13. When there is no appreciable odour of solvent place the flask on its side in the oven and heat for one hour.

6.2.14. Remove the flask from the oven, allow to cool to the temperature of the balance room and accurately weigh to the nearest 0.1 mg.

6.2.15. Repeat 6.2.13 and 6.2.14 for heating periods of 30 to 60 minutes until the difference in mass of two successive weighings is less than 0.5 mg or until the mass increases. If an increase in mass occurs use the lowest mass obtained in the calculation (7.1). Let the final weight recorded be M_1 g.

6.2.16. Add 15 to 25 ml light petroleum in order to confirm that the extracted matter is wholly soluble. Warm gently and swirl the solvent until all the fat is dissolved.

6.2.16.1. If the extracted matter is wholly soluble in the light petroleum, the mass of fat is the difference between the weights determined at stages 6.2.1 and 6.2.15.

6.2.16.2. If any insoluble matter is present, or in case of doubt, completely extract the fat from the flasks by repeated washing with warm light petroleum, allowing the undissolved material to settle before each decantation. Rinse the outside of the neck of the flask three times. Heat the flask, placed on its side, for one hour in the oven, allow to cool to the temperature of the balance room as before (6.2.1) and weigh to the nearest 0.1 mg. The mass of fat is the difference between the mass obtained at 6.2.15 and this final mass.

7. EXPRESSION OF RESULTS

7.1. Calculation

The mass, in g of fat extracted is:

$$(M_1 - M_2) - (B_1 - B_2)$$

and the fat content of the sample, expressed as a percentage is:

$$\frac{(M_1 - M_2) - (B_1 - B_2)}{S} \times 100$$

where:

M_1 = mass, in g of flask M with fat after stage 6.2.15;

M_2 = mass, in g of flask M after stage 6.2.1 or, in the case of undissolved material or doubt, stage 6.2.16.2;

B_1 = mass, in g of flask B of the blank after stage 6.2.15;

B_2 = mass, in g of flask B after stage 6.2.1 or, in the case of undissolved material or doubt, stage 6.2.16.2;

S = mass, in g of sample used.

7.2. Repeatability

The difference between results of two determinations carried out obtained simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.05 g fat per 100 g of the product.

**METHOD 4: DETERMINATION OF FAT CONTENT IN DRIED MILKS
(RÖSE-GOTTLIEB METHOD)**

1. SCOPE AND FIELD AND APPLICATION

This method determines the fat content of:

- dried high fat milk or high fat milk powder,
- dried whole milk or whole milk powder,
- dried partly skimmed milk or partly skimmed-milk powder,
- dried skimmed milk or skimmed-milk powder.

2. DEFINITION

The fat content of dried milks: fat content as determined by the method specified.

3. PRINCIPLE

The fat content is determined by extraction of the fat from an ammoniacal alcoholic solution of sample with diethyl ether and light petroleum, followed by evaporation of the solvents and weighing of the residue and calculation as a percentage by mass of the sample, according to the principle of Röse-Gottlieb.

4. REAGENTS

All reagents should conform to the requirements specified in the blank test (6.1). If necessary, reagents may be redistilled in the presence of about 1 g of butterfat per 100 ml of solvent.

4.1. Ammonia solution, approximately 25 % (m/m) NH_3 (density at 20 °C approximately 0.91 g/ml), or stronger solution of known concentration.

4.2. Ethanol, 96 ± 2 % (v/v) or, if not available, ethanol denatured with methanol, ethyl methyl ketone or light petroleum.

4.3. Diethyl ether, peroxide-free

Note 1:

To test for peroxide, add to 10 ml of the ether in a small glass stoppered cylinder, previously rinsed with the ether, 1 ml freshly prepared 10 % potassium iodide solution. Shake and let stand for one minute. No yellow colour should be observed in either layer.

Note 2:

Diethyl ether may be maintained free from peroxides by adding wet zinc foil that has been completely immersed in dilute acidified copper sulphate solution for one minute and subsequently washed with water. Use per litre approximately 8 000 mm² zinc foil cut in strips long enough to reach at least halfway up the container.

4.4. Light petroleum (petroleum ether), with any boiling range between 30 and 60 °C.

4.5. Mixed solvent, prepared shortly before use by mixing equal volumes of diethyl ether (4.3) and light petroleum (4.4) (when the use of mixed solvent is indicated, it may be replaced by either diethyl ether or light petroleum alone).

5. APPARATUS

5.1. Analytical balance.

5.2. Suitable extraction tubes or flasks, provided with ground glass stoppers or other closures unaffected by the solvents used.

5.3. Flasks, thin-walled, flat-bottomed, of 150 to 250 ml capacity.

5.4. Atmospheric pressure drying oven, well ventilated and thermostatically controlled (adjusted to operate at $102 \text{ °C} \pm 1 \text{ °C}$).

- 5.5. Anti-bumping granules, fat-free, non porous, non friable in use, e.g. glass beads or pieces of silicon carbide (the use of this material is optional: see clause 6.2.1).
- 5.6. Waterbath, at 60 to 70 °C.
- 5.7. Siphon to fit extraction tubes.
- 5.8. Centrifuge (optional).

6. PROCEDURE

6.1. Blank test

At the same time as the determination of the fat content of the sample, carry out a blank determination on 10 ml of water using the same type of extraction apparatus, the same reagents in the same amounts and the same procedure as described hereafter, excluding clause 6.2.2. If blank exceeds 0.5 mg, the reagents should be checked and the impure reagent or reagents should be purified or replaced.

6.2. Determination

- 6.2.1. Dry the flask (5.3) together with, if required, some anti-bumping granules (5.5) to promote gentle boiling during the subsequent removal of the solvents) in the oven (5.4) for half to one hour. Allow the flask to cool to the temperature of the balance room and accurately weigh the cooled flask to the nearest 0.1 mg.
- 6.2.2. Accurately weigh, to the nearest 1 mg, directly in, or by difference into, the extraction apparatus (5.2) about 1 g of whole milk powder or about 1.5 g of partly skimmed or skimmed-milk powder. Add 10 ml water and shake gently until the milk powder is completely dispersed (heat may be necessary for some samples).
- 6.2.3. Add 1.5 ml ammonia (25 %) (4.1) or a corresponding volume of a stronger solution and heat in a waterbath (5.6) for 15 minutes at 60 to 70 °C, shaking occasionally. Cool, for example, in running water.
- 6.2.4. Add 10 ml ethanol (4.2) and mix the liquids gently but thoroughly in the unclosed apparatus.
- 6.2.5. Add 25 ml diethyl ether (4.3). Cool in running water. Close the apparatus and shake vigorously and invert repeatedly for one minute.
- 6.2.6. Remove the stopper carefully and add 25 ml light petroleum (4.4) using the first few millilitres to rinse the stopper and inside of the neck of the apparatus, allowing the rinsings to run into the apparatus. Close by replacing the stopper and shake and invert repeatedly for 30 seconds. Do not shake too vigorously if centrifuging is not to be used in 6.2.7.
- 6.2.7. Allow the apparatus to stand until the upper liquid layer has become clear and has distinctly separated from the lower aqueous layer. Alternatively carry out the separation using a suitable centrifuge (5.8).

Note:

When a centrifuge which is not driven by a three-phase motor is used, sparks may occur and care must therefore be taken to avoid an explosion or fire from any ether vapours coming, for example, from a broken tube.

- 6.2.8. Remove the stopper, rinse it and the inside of the neck of the apparatus with a few millilitres of mixed solvent (4.5) and allow the rinsings to run into the apparatus. Carefully transfer as much as possible of the supernatant layer by decantation or by means of a siphon (5.7) into the prepared flask (6.2.1).

Note:

If the transfer is not made using a siphon, it may be necessary to add a little water in order to raise the interface between the two layers thus aiding decantation.

- 6.2.9. Rinse the outside and the inside of the neck of the apparatus or the tip and the lower part of the siphon with a few millilitres of mixed solvent. Allow the rinsings from the outside of the apparatus to run into the flask and the rinsings from the inside of the neck and from the siphon to run into the extraction apparatus.

- 6.2.10. Make a second extraction by repeating the procedure of 6.2.5 to 6.2.9 inclusive but using only 15 ml diethyl ether and 15 ml light petroleum.
- 6.2.11. Make a third extraction by repeating the procedure of 6.2.10 but omit the final rinsing (6.2.9).
Note:
It is not mandatory to carry out this third extraction when analysing dried skimmed milk samples.
- 6.2.12. Carefully evaporate or distil off as much solvent (including the ethanol) as possible. If the flask is of small capacity it will be necessary to remove some of the solvent as above after each extraction.
- 6.2.13. When there is no appreciable odour of solvent, place the flask on its side in the oven and heat for one hour.
- 6.2.14. Remove the flask from the oven, allow to cool to the temperature of the balance room as previously (6.2.1) and accurately weigh to the nearest 0.1 mg.
- 6.2.15. Repeat 6.2.13 and 6.2.14 for heating periods of 30 to 60 minutes until the difference in mass of two successive weights is less than 0.5 mg or until the mass increases. If an increase in mass occurs use the lowest mass obtained in the calculation (7.1). Let the final weight recorded be M_1 g.
- 6.2.16. Add 15 to 25 ml light petroleum in order to confirm that the extracted matter is wholly soluble. Warm gently and swirl the solvent until all the fat is dissolved.
- 6.2.16.1. If the extracted matter is wholly soluble in the light petroleum, the mass of fat is the difference between the weights determined at stages 6.2.1 and 6.2.15.
- 6.2.16.2. If any insoluble matter is present, or in case of doubt completely extract the fat from the flask by repeated washing with warm light petroleum, allowing the undissolved material to settle before each decantation. Rinse the outside of the neck of the flask three times.
Heat the flask, placed on its side, for one hour in the oven, allow to cool to the temperature of the balance room, as before (6.2.1) and weigh to the nearest 0.1 mg. The mass of fat is the difference between the mass under 6.2.15 and this final mass.

7. EXPRESSION OF RESULTS

7.1. Calculation

The mass, in g of fat extracted is:

$$(M_1 - M_2) - (B_1 - B_2)$$

and the fat content of the sample, expressed as a percentage, is:

$$\frac{(M_1 - M_2) - (B_1 - B_2)}{S} \times 100$$

where:

M_1 = mass, in g of flask M with fat after stage 6.2.15;

M_2 = mass, in g of flask M after stage 6.2.1 or, in the case of undissolved material or doubt, stage 6.2.16.2;

B_1 = mass, in g of flask B of the blank after stage 6.2.15;

B_2 = mass, in g of flask B after stage 6.2.1 or, in the case of undissolved material or doubt, stage 6.2.16.2;

S = mass, in g of sample used.

7.2. Repeatability

The difference between results of two determinations carried out simultaneously or in rapid succession on the same sample, the same analyst, under the same conditions, shall not exceed 0.2 g fat per 100 g of product with the exception of skimmed-milk powder for which the difference must not exceed 0.1 g fat per 100 g of product.

**METHOD 5: DETERMINATION OF SUCROSE CONTENT
(POLARIMETRIC METHOD)****1. SCOPE AND FIELD OF APPLICATION**

This method determines the sucrose content of:

- sweetened condensed milk,
- sweetened condensed partly skimmed milk,
- sweetened condensed skimmed milk.

Samples must not contain invert sugar.

2. DEFINITION

The sucrose content of sweetened condensed milks: the sucrose content as determined by the method specified.

3. PRINCIPLE

The method is based on the principle of the Clerget inversion, a mild treatment of the sample with acid which produces complete hydrolysis of sucrose but almost none of lactose or other sugars. The sucrose content is obtained from the change in rotating power of the solution.

A clear filtrate of the sample, without mutarotation by lactose, is prepared by treatment of the solution with ammonia followed by neutralization and clearing by the successive addition of zinc acetate and potassium hexacyanoferrate II solutions.

In a portion of the filtrate the sucrose is hydrolyzed in a specified manner.

From the rotation of the filtrate before and after inversion, the sucrose content is calculated using the appropriate formulae.

4. REAGENTS

- 4.1. Zinc acetate solution, 1 M: dissolve 21.9 g crystallized zinc acetate dihydrate $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ and 3 ml glacial acetic acid in water and make up to 100 ml with water.
- 4.2. Potassium hexacyanoferrate (II) solution, 0.25 M: dissolve 10.6 g crystallized potassium hexacyanoferrate (II) trihydrate $\text{K}_4[\text{Fe}(\text{CN})_6] \cdot 3\text{H}_2\text{O}$ in water and make up to 100 ml with water.
- 4.3. Hydrochloric acid solution, 6.35 ± 0.20 M (20 to 22 %) or 5.0 ± 0.2 M (16 to 18 %).
- 4.4. Ammonia solution, 2.0 ± 0.2 M (3.5 %).
- 4.5. Acetic acid solution, 2.0 ± 0.2 M (12 %).
- 4.6. Bromothymol blue indicator, 1 % (m/v) solution in ethanol.

5. APPARATUS

- 5.1. Balance, sensitivity 10 mg.
- 5.2. Polarimeter tube, 2dm, of exactly calibrated length.

5.3. Polarimeter or saccharimeter:

- (a) Polarimeter with sodium light or mercury green light (mercury vapour lamp with prism or the special Wratten Screen No 77 A), to be read with an accuracy of at least 0.05 angular degrees,
- (b) Saccharimeter with international sugar scale, using white light passing through a filter of 15 mm of a 6 % solution of potassium bichromate, or sodium light, to be read with an accuracy of at least 0.1 ° on the international sugar scale.

5.4. Water bath, regulated at $60^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

6. PROCEDURE

6.1. Control determination

In order to standardize the procedure, reagents and apparatus, carry out a control determination in duplicate as described below using a mixture of 100 g of milk and 18 g pure sucrose or a mixture of 110 g of skimmed milk and 18 g pure sucrose, each corresponding to 40 g of condensed milk containing 45 % sucrose. Calculate the sugar content using the formulae under 7, substituting for M, F and P respectively in formula 1 the quantity of milk taken and the fat and protein content of this milk, and in formula 2 for M, the value of 40.00. The mean of the values found shall not differ by more than 0.2 % from 45.0 %.

6.2. Determination

- 6.2.1. Weigh to within 10 mg, approximately 40 g of the well mixed sample into a 100 ml glass beaker. Add 50 ml of hot water (80 to 90°C) and mix well.
- 6.2.2. Transfer the mixture quantitatively to a 200 ml measuring flask, rinsing the beaker with successive quantities of water at 60°C , until the total volume is between 120 and 150 ml. Mix and cool to room temperature.
- 6.2.3. Add 5 ml of the dilute ammonia solution (4.4). Mix again and then allow to stand for 15 minutes.
- 6.2.4. Neutralize the ammonia by adding an equivalent quantity of the diluted solution of acetic acid (4.5). Determine the exact number of ml beforehand by titration of the ammonia solution using bromothymol blue as indicator (4.6). Mix.
- 6.2.5. Add, with gently mixing by rotating the tilted flask, 12.5 ml of zinc acetate solution (4.1).
- 6.2.6. Add 12.5 ml of potassium hexacyanoferrate (II) solution (4.2) in the same way as for the acetate solution.
- 6.2.7. Bring the contents of the flask to 20°C and make up to the 200 ml mark with water at 20°C .

Note:

During any of the stages so far described all additions of water or reagents should have been made in such manner as to avoid the formation of air bubbles, and with the same object in view, all mixing should have been carried out by rotation of the flask rather than by shaking. If air bubbles are found to be present before making up to 200 ml volume, their removal can be assisted by temporarily connecting the flask to a vacuum pump, and rotating the flask.

- 6.2.8. Close the flask with a dry stopper and mix thoroughly by vigorous shaking.
- 6.2.9. Allow to stand for a few minutes and then filter through a dry filter paper, rejecting the first 25 ml of filtrate.
- 6.2.10. Direct polarization: determine the optical rotation of the filtrate at $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$.
- 6.2.11. Inversion: pipette 40 ml of the filtrate obtained above into a 50 ml volumetric flask. Add 6.0 ml of 6.35 M hydrochloric acid or 7.5 ml of 5.0 M hydrochloric acid (4.3).

Place the flask in a waterbath of 60°C for 15 minutes, ensuring that the entire bulb of the flask has been immersed. Mix by a rotatory movement during the first five minutes, in which time the

contents of the flask should have attained the temperature of the bath. Cool to 20 °C, and make up to volume with water at 20 °C. Mix and allow to stand for one hour at this temperature.

6.2.12. *Invert polarization*

Determine the rotation of the inverted solution at 20 °C ± 0.2 °C. (However, if temperature T of the liquid in the polarization tube differs by more than 0.2 °C during the measurement, the temperature correction referred to under 7.2 must be applied.)

7. EXPRESSION OF RESULTS

7.1. Method of calculation

Calculate the sucrose content by means of the following formulae:

$$(1) \quad v = \frac{M}{100} (1.08 F + 1.55 P)$$

$$(2) \quad S = \frac{D - 1.25 I}{Q} \times \frac{V - v}{V} \times \frac{V}{L \times M} \%$$

where:

S = sucrose content;

M = mass of the weighed sample in grams;

F = percentage of fat in the sample;

P = percentage of protein (N × 6.38) in the sample;

V = volume in ml to which the sample is diluted before filtration;

v = correction in ml for the volume of the precipitate formed during clarification;

D = direct polarimeter reading (polarization before inversion);

I = polarimeter reading after inversion;

L = length in dm of the polarimeter tube;

Q = inversion factor, the values of which are given below.

Remarks:

(a) When exactly 40.00 g of condensed milk are weighed and a polarimeter with sodium light, angular degrees and a 2dm polarimeter tube at 20.0 °C ± 0.1 °C is used the sucrose content of normal condensed milk (C = 9) can be calculated from the following formula:

$$S = (D - 1.25 I) \times (2.833 - 0.00612 F - 0.00878 P)$$

(b) If the invert polarization is measured at a temperature other than 20 °C, the figures should be multiplied by:

$$(1 + 0.0037 (T - 20)).$$

7.2. Values of the inversion factor Q

The following formulae give accurate values for Q, for various sources of light with corrections for concentration and temperature:

Sodium light and polarimeter with angular degrees:

$$Q = 0.8825 + 0.0006 (C - 9) - 0.0033 (T - 20).$$

Mercury green light and polarimeter with angular degrees:

$$Q = 1.0392 + 0.0007 (C - 9) - 0.0039 (T - 20).$$

White light with dichromate filter and saccharimeter with international sugar scale degrees:

$$Q = 2.549 + 0.0017 (C - 9) - 0.0095 (T - 20).$$

In the above formulae:

C = Percentage of total sugars in the inverted solution as polarized,

T = Temperature of the inverted solution in the polarimetric reading.

Note 1:

The percentage of total sugars C in the inverted solution may be calculated from the direct reading and the change on inversion in the usual manner, using the usual values for the specific rotations of sucrose, lactose and invert sugar.

The correction 0.0006 (C - 9) etc., is only accurate when C is approximately 9; for normal condensed milk, this correction can be neglected, C being close to 9.

Note 2:

Variation in temperature from 20 °C of 1 °C makes little difference in the direct reading, but variation of over 0.2 °C in the invert reading necessitates a correction. The correction $- 0.0033 (T - 20)$ etc., is only accurate between 18 °C and 22 °C.

7.3. Repeatability

The difference between results of two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.3 g of sucrose per 100 g of condensed milk.

METHOD 6: DETERMINATION OF LACTIC ACID AND LACTATES CONTENT**1. SCOPE AND FIELD OF APPLICATION**

This method determines the lactic acid and lactates, expressed as lactic acid, contents of:

- dried high fat milk or high fat milk powder,
- dried whole milk or whole milk powder,
- dried partly skimmed milk or partly skimmed-milk powder,
- dried skimmed milk or skimmed-milk powder.

2. DEFINITION

Lactic acid and lactates content of dried milks: the lactic acid and lactates, expressed as lactic acid, contents as determined by the method specified.

3. PRINCIPLE

Fat, protein and lactose are simultaneously removed from a solution of the sample by addition of copper sulphate and calcium hydroxide followed by filtration.

The lactic acid and lactates in the filtrate are converted into acetaldehyde by concentrated sulphuric acid in the presence of copper II sulphate.

The lactic acid content is determined colorimetrically using p-hydroxydiphenyl.

The lactic acid and lactates content is expressed as mg of lactic acid per 100 g of solids-non-fat.

4. REAGENTS

- 4.1. Copper (II) sulphate solution: dissolve 250 g of copper (II) sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and dilute to 1 000 ml with water.
- 4.2. Calcium hydroxide suspension.
 - 4.2.1. Grind 300 g of calcium hydroxide ($\text{Ca}(\text{OH})_2$) in a mortar with water, using totally 900 ml. The suspension should be freshly prepared before use.
 - 4.2.2. Calcium hydroxide suspension: grind 300 g of calcium hydroxide ($\text{Ca}(\text{OH})_2$) in a mortar with water, using totally 1 400 ml. The suspension should be freshly prepared before use.
- 4.3. Sulphuric acid — copper (II) sulphate solution: Add to 300 ml of sulphuric acid, 95.9 to 97.0 % (m/m) of H_2SO_4 , 0.5 ml of the copper (II) sulphate solution (4.1).
- 4.4. p-hydroxydiphenyl ($\text{C}_6\text{H}_5\text{C}_6\text{H}_4\text{OH}$) solution: dissolve, by shaking and by heating slightly 0.75 g of p-hydroxydiphenyl in 5 ml of an aqueous solution of sodium hydroxide, containing 5 g of NaOH per 100 ml. Dilute to 50 ml with water in a volumetric flask. Keep the solution in a brown coloured glass bottle in a dark and cool place. Do not use if the colour changes or turbidity occurs. The maximum shelf life is 72 hours.

- 4.5. Lactic acid standard solution: dissolve, shortly before use, 0.1067 g of lithium lactate ($\text{CH}_3\text{CHOHCOOLi}$) in water and dilute to 1 000 ml in a volumetric flask. 1 ml of this solution corresponds to 0.1 mg of lactic acid.
- 4.6. Standard reconstituted milk: analyse in advance several samples of high quality dried milk. For the preparation of the calibration curve select the sample having the lowest lactic acid content, containing not more than 30 mg of lactic acid per 100 g of solids-non-fat. Follow the operating procedure described under 6.2.1 and 6.2.2 below.

5. APPARATUS

- 5.1. Analytical balance.
- 5.2. Spectrophotometer suitable for readings at a wavelength of 570 nm.
- 5.3. Waterbath at $30^\circ\text{C} \pm 2^\circ\text{C}$.
- 5.4. Mortar and pestle.
- 5.5. Filter paper (Schleicher and Schull 595, Whatman 1 or equivalent).
- 5.6. Test tubes, pyrex or equivalent (dimensions 25×150 mm).

Note:

All glassware must be perfectly clean and designated for use solely in this determination. Rinse glassware containing precipitate residues with concentrated hydrochloric acid before washing.

6. PROCEDURE

6.1. Blank test

Carry out a blank test by placing 30 ml of water into a 50 ml graduated tube and treating this tube as described under 6.2.4 to 6.2.11 inclusive. If the blank measured against water exceeds an equivalent of 20 mg of lactic acid per 100 g solids-non-fat, the reagents should be checked and the impure reagents or reagent should be replaced. Carry out the blank test at the same time as the analysis of the sample.

6.2. Determination

Note: Avoid contamination with impurities especially with saliva and sweat.

- 6.2.1. Determine the solids-non-fat content (a) g of the sample by subtracting the fat content (obtained by method 4) and the moisture content (obtained by method 2) from 100.
- 6.2.2. Weigh $\frac{1000}{(a-10)}$ g of the sample to the nearest 0.1 g. Add this quantity of sample to 100 ml of water and mix thoroughly.
- 6.2.3. Pipette 5 ml of the solution obtained into a 50 ml graduated tube and dilute with water to about 30 ml.
- 6.2.4. Add slowly while shaking, 5 ml of the copper (II) sulphate solution (4.1) and allow to stand for 10 minutes.
- 6.2.5. Add slowly while shaking, 5 ml of the calcium hydroxide suspension (4.2.1) or 10 ml of the calcium hydroxide suspension (4.2.2).
- 6.2.6. Dilute to 50 ml with water, shake vigorously, allow to stand for 10 minutes then filter. Discard the first runnings.
- 6.2.7. Pipette 1 ml of the filtrate into a test tube (5.6).
- 6.2.8. Add to the tube by means of a burette or graduated pipette 6.0 ml of the sulphuric acid-copper (II) sulphate solution (4.3). Mix.
- 6.2.9. Heat in the boiling water bath for five minutes. Cool to ambient temperature under running water.

- 6.2.10. Add two drops of p-hydroxydiphenyl reagent (4.4) and shake vigorously to spread the reagent evenly throughout the liquid. Place the tube in the waterbath at $30^{\circ}\text{C} \pm 2^{\circ}\text{C}$; leave for 15 minutes shaking from time to time.
- 6.2.11. Place the tube in the boiling waterbath for 90 seconds. Cool to ambient temperature under running water.
- 6.2.12. Measure the optical density against the blank test (6.1) within three hours at the wavelength specified under 5.2.
- 6.2.13. If the optical density exceeds that of the highest point of the standard curve, repeat the test using an adequate dilution of the filtrate obtained under 6.2.6.

6.3. Preparation of the standard

- 6.3.1. Pipette 5 ml of the reconstituted milk (4.6) into five 50 ml graduated tubes. Pipette into these tubes 0, 1, 2, 3 and 4 ml respectively of the standard solution (4.5), so as to obtain a range of standards corresponding to 0, 20, 40, 60 and 80 mg of added lactic acid per 100 g of solids-non-fat, of the dried milk.
- 6.3.2. Dilute with water to about 30 ml and treat as described under 6.2.4 to 6.2.11.
- 6.3.3. Measure the optical densities of the standards (6.3.1) against the blank test (6.1) at the wavelength specified under 5.2. Plot in a diagram the optical densities against the quantities of lactic acid given under 6.3.1, i.e. 0 mg, 20 mg, 40 mg, 60 mg and 80 mg per 100 g of solids-non-fat. Draw the best fitting straight line through the points and prepare the standard curve by moving this line parallel to itself in such a way that it passes through the origin.

7. EXPRESSION OF RESULTS

7.1. Method of calculation

Convert the optical density measured under 6.2.12 or 6.2.13 into mg of lactic acid per 100 g of solids-non-fat in the sample by reference to the standard curve. Multiply this result by the dilution factor where the filtrate has been diluted according to 6.2.13.

7.2. Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 8 mg of lactic acid per 100 g of solids-non-fat for contents up to 80 mg. For higher values, this difference may not exceed 10 % of the lowest value.

**METHOD 7: DETERMINATION OF PHOSPHATASE ACTIVITY
(MODIFIED SANDERS AND SAGER PROCEDURE)**

1. SCOPE AND FIELD OF APPLICATION

This method describes the determination of phosphatase activity in:

- dried high fat milk or high fat milk powder,
- dried whole milk or whole milk powder,
- dried partly skimmed milk or partly skimmed-milk powder,
- dried skimmed milk or skimmed-milk powder.

2. DEFINITION

The phosphatase activity of dried milks is a measure of the quantity of active alkaline phosphatase present. It is expressed as the quantity of phenol in μg liberated by 1 ml of reconstituted milk, as determined by the procedure described below.

3. PRINCIPLE

The phosphatase activity of dried milks is determined by the ability of the phosphatase to liberate the phenol from disodiumphenylphosphate. The quantity of phenol liberated under prescribed conditions is determined by a spectrophotometric measurement of the colour developed with Gibb's reagent.

4. REAGENTS

4.1. Solution A

Barium borate hydroxide buffer: pH 10.6 ± 0.1 at 20°C .

Dissolve: 25.0 g of barium hydroxide ($\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$) in water and dilute to 500 ml.

Dissolve: 11.0 g of boric acid (H_3BO_3) in water and dilute to 500 ml.

Warm the two solutions to 50°C and mix.

Shake and cool the mixture to room temperature.

Adjust the pH to 10.6 ± 0.1 with the barium hydroxide solution and filter.

Store the solution in a tightly stoppered container.

Before use, dilute the buffer with an equal quantity of water.

4.2. Solution B:

Colour development buffer.

Dissolve: 6.0 g of sodium metaborate (NaBO_2) (or 12.6 g of $\text{NaBO}_2 \cdot 4\text{H}_2\text{O}$) and 20.0 g of sodium chloride (NaCl) in water and dilute to 1 000 ml with water.

4.3. Solution C

Buffer substrate solution.

4.3.1. Dissolve 0.5 g of disodiumphenylphosphate ($\text{Na}_2\text{C}_6\text{H}_5\text{PO}_4 \cdot 2\text{H}_2\text{O}$) in 4.5 ml of Solution B (4.2). Add 2 drops of Solution E (4.5) and allow to stand 30 minutes. Extract the colour with 2.5 ml butanol (4.10). If necessary, repeat the colour extraction. After separation, discard the butanol. This solution can be kept for several days in a refrigerator. Develop and extract the colour once more before use.

4.3.2. Pipette 1 ml of this solution into a 100 ml volumetric flask and make up to volume with Solution A. Prepare the buffer solution immediately before use.

4.4. Solution D

Precipitant.

Dissolve 3.0 g of zinc sulphate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) and 0.6 g of copper (II) sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and make up to 100 ml with water.

4.5. Solution E

Gibb's reagent.

Dissolve 0.040 g of 2,6-dibromoquinone 1,4 — chloroimide ($\text{O} \cdot \text{C}_6\text{H}_2\text{Br}_2 \cdot \text{NCI}$) in 10 ml of 96 % ethanol. Store the solution in a dark glass bottle kept in a refrigerator. Discard this reagent when it has become discoloured.

4.6. Colour dilution buffer

Dilute 10 ml of Solution B (4.2), colour development buffer, to 100 ml with water.

4.7. Copper sulphate solution

Dissolve 0.05 g of copper (II) sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and make to 100 ml with water.

4.8. Phenol standard solution

Dissolve 0.200 ± 0.001 g of pure phenol in water and make up to 100 ml in a volumetric flask with water. This solution can be stored for several months in a refrigerator. Dilute 10 ml of this solution to 100 ml with water. This diluted solution contains 200 μg of phenol in 1 ml and can be used for preparing more dilute solutions.

4.9. Boiled distilled water.

- 4.10. n-Butanol.

5. APPARATUS

- 5.1. Analytical balance.
- 5.2. Waterbath, thermostatically controlled at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$.
- 5.3. Spectrophotometer suitable for readings at a wavelength of 610 nm.
- 5.4. Filter paper (Schleicher and Schull 597, Whatman 42 or equivalent filter paper).
- 5.5. Waterbath, boiling.
- 5.6. Aluminium foil.

6. PROCEDURE

Precautions:

1. Avoid direct exposure to sunlight.
2. All the glassware, stoppers and removal material should be perfectly clean. It is recommended that they be rinsed and boiled with water or that they be treated with steam.
3. Avoid using plastic materials (stoppers for example) as they may contain phenols.
4. Saliva contains phosphatase; contamination by traces of saliva must therefore be carefully avoided.

6.1. Preparation of the sample

- 6.1.1. Weigh, to within 0.1 g, 10 g of the sample and dissolve in 90 ml of water. The temperature for dissolving the powder shall, under no circumstances, exceed 35°C .

6.2. Determination

- 6.2.1. Introduce in each of two test tubes 1 ml of reconstituted milk prepared as described in 6.1.1.
- 6.2.2. Heat one of the tubes in boiling water for two minutes. Cover the tube and the waterbath (5.5) or, for example, a beaker with aluminium foil (5.6) to ensure that the entire tube will be heated. Cool in cold water to room temperature. Use this tube for the blank test. For all subsequent operations treat the two tubes identically.
- 6.2.3. Add 10 ml of Solution C (4.3.2). Mix and place the tube in the waterbath at 37°C (5.2).
- 6.2.4. Incubate for 60 minutes in the waterbath shaking periodically.
- 6.2.5. Transfer the tubes immediately to a boiling waterbath (5.5) and heat for two minutes; cool to room temperature in cold water.
- 6.2.6. Add 1 ml of Solution D (4.4), mix and filter through a dry filter paper; discard the first filtrates until a clear liquid is obtained.
- 6.2.7. Put 5 ml of each filtrate into test tubes, add 5 ml of Solution B (4.2) and 0.1 ml of Solution E (4.5). Mix.
- 6.2.8. Allow the colour to develop at room temperature for 30 minutes away from direct sunlight.
- 6.2.9. Measure the optical density of the sample solution, against the blank, at the wavelength indicated in 5.3.
- 6.2.10. Repeat the determination if the optical density of the solution is above that of the standard sample with $20\text{ }\mu\text{g}$ of phenol prepared according to 7.
- If this limit is exceeded, dilute a suitable volume of reconstituted milk according to 6.1.1 with a suitable volume of this milk carefully boiled as indicated in 6.2.2 to inactivate the phosphatase present.

7. PREPARATION OF THE STANDARD CURVE

- 7.1. Pipette into four 100 ml volumetric flasks, 1, 3, 5 and 10 ml of the standard solution diluted according to 4.8 and make up to volume with water; these dilutions contain respectively 2, 6, 10 and 20 µg of phenol per ml.
- 7.2. Pipette 1 ml of water or 1 ml of each standard solution (7.1) into the test tubes in order to obtain a series of samples containing 0 (blank value obtained using the 1 ml of water) — 2 — 6 — 10 and 20 µg of phenol.
- 7.3. Pipette successively into each test tube 1 ml of the solution of copper (II) sulphate (4.7), 5 ml of the colour dilution buffer solution (4.6), 3 ml of water and 0.1 ml of Solution E (4.5). Mix.
- 7.4. Leave the test tubes for 30 minutes at room temperature away from direct sunlight.
- 7.5. Measure the absorbance of the solutions in each of the tubes, compared to the blank value, at the wavelength indicated in 5.3.
- 7.6. Prepare the standard curve by plotting the absorbance values against the quantities of phenol in µg as indicated in 7.2.

8. EXPRESSION OF THE RESULTS**8.1. Calculation**

- 8.1.1. Convert the figures as determined under 6.2.9 to µg of phenol, by reference to the standard curve.
- 8.1.2. Calculate the phosphatase activity expressed as µg of phenol per ml of reconstituted milk according to the following formula:
$$\text{Phosphatase activity} = 2.4 \times P$$
where P = the quantity of phenol in µg according to 8.1.1.
- 8.1.3. If it was necessary to dilute as indicated under 6.2.10 multiply the result obtained in 8.1.2 by the dilution factor.

8.2. Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 2 µg of phenol liberated by 1 ml of reconstituted milk.

**METHOD 8: DETERMINATION OF PHOSPHATASE ACTIVITY
(ASCHAFFENBURG AND MÜLLEN PROCEDURE)****1. SCOPE AND FIELD OF APPLICATION**

This method describes the determination of phosphatase activity in:

- dried high fat milk or high fat milk powder,
- dried whole milk or whole milk powder,
- dried partly skimmed milk or partly skimmed-milk powder,
- dried skimmed milk or skimmed-milk powder.

2. DEFINITION

The phosphatase activity of dried milks is a measure of the quantity of active alkaline phosphatase present in the product. It is expressed as the quantity of p-nitrophenol in micrograms liberated by 1 ml of the reconstituted sample, under the conditions described.

3. PRINCIPLE

The reconstituted sample is diluted with a buffer substrate at pH 10.2 and incubated at a temperature of 37 °C for two hours. Any alkaline phosphatase present in the sample will, under

these circumstances, liberate p-nitrophenol from added disodium p-nitrophenyl phosphate. The p-nitrophenol liberated is determined by direct comparison with standard colour glasses in a simple comparator using reflected light.

4. REAGENTS

4.1. Sodium carbonate-bicarbonate buffer solution.

Dissolve 3.5 g of anhydrous sodium carbonate and 1.5 g of sodium bicarbonate in water and dilute to 1 000 ml in a volumetric flask with water.

4.2. Buffer substrate.

Dissolve 1.5 g of disodium p-nitrophenylphosphate in sodium carbonate-bicarbonate buffer (4.1) and dilute to 1 000 ml in a volumetric flask with buffer (4.1).

This solution is stable if stored in a refrigerator ($\leq 4^{\circ}\text{C}$) for one month but a colour control test should be carried out on such stored solutions — see 6, precaution number 3.

4.3. Clarification solutions.

4.3.1. Zinc sulphate solution.

Dissolve 30.0 g of zinc sulphate (ZnSO_4) in water and dilute to 100 ml in a volumetric flask with water.

4.3.2. Potassium hexacyanoferrate (II) solution.

Dissolve 17.2 g of potassium hexacyanoferrate (II) trihydrate ($\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$) and dilute to 100 ml in a volumetric flask with water.

5. APPARATUS

5.1. Analytical balance.

5.2. Waterbath, thermostatically controlled at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

5.3. Comparator, with special disc containing standard colour glasses calibrated in μg p-nitrophenol per ml milk, and 2×25 mm cells.

6. PROCEDURE

Precautions:

1. After use, test tubes must be emptied, rinsed in water, washed in hot water containing an alkaline detergent, followed by thorough rinsing in clean hot tap water. Finally, they must be rinsed in water and dried before use.

Pipettes must be thoroughly rinsed in clean cold tap water immediately after use, followed by rinsing in water and dried before use.

2. The test tube stoppers must be thoroughly rinsed in hot tap water immediately after use, followed by boiling for two minutes in water.
3. The buffer substrate solution (4.2) should remain stable for at least one month if stored in a refrigerator at 4°C or less. Any instability is denoted by the formation of a yellow colour. Whilst the test is always read against a boiled product control containing the same buffer substrate solution, it is recommended that the solution should not be used if it gives a colour reading in excess of $10 \mu\text{g}$ when read in a 25 mm cell in the comparator using distilled water in the other 25 mm cell.
4. Use a separate pipette for each sample and avoid contaminating the pipette with saliva.
5. The test must not be exposed to direct sunlight at any time.

6.1. Preparation of sample

Dissolve 10 g of the powder in 90 ml of water. The temperature for dissolving the powder must not exceed 35°C .

6.2. Determination

- 6.2.1. Pipette 15 ml of buffer substrate (4.2) into a clean, dry test tube, followed by 2 ml of the reconstituted sample (6.1) to be tested. Stopper the tube, mix by inversion and place in the 37 °C water bath (5.2).
- 6.2.2. At the same time, place in the water bath a control tube containing 15 ml of buffer substrate and 2 ml of boiled reconstituted sample similar to that under test.
- 6.2.3. After two hours remove both tubes from the water bath, add 0.5 ml of zinc sulphate precipitant (4.3.1), replace the stopper, shake vigorously and allow to stand for three minutes. Add 0.5 ml of potassium hexacyanoferrate (II) precipitant (4.3.2), mix thoroughly and filter through the fluted filter paper (5.4) and collect the clear filtrate in the clean test tube.
- 6.2.4. Transfer the filtrate to a 25 mm cell and compare against the filtrate of the boiled sample control in the comparator using the special disc (5.3).

7. EXPRESSION OF RESULTS**7.1. Calculation**

The direct reading obtained under 6.2.4 is recorded as µg p-nitrophenol per ml sample or per ml of reconstituted sample.

7.2. Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 2 µg of p-nitrophenol liberated by 1 ml of reconstituted milk.
