I

(Acts whose publication is obligatory)

COMMISSION REGULATION (EC) No 454/95
of 28 February 1995
laying down detailed rules for intervention on the market in butter and cream

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Community,

Having regard to Council Regulation (EEC) No 804/68 of 27 June 1968 on the common organization of the market in milk and milk products (1), as last amended by the Act of Accession of Austria, Finland and Sweden, and in particular Articles 6 (6), 28 and 30 thereof,

Whereas Council Regulation (EEC) No 985/68 of 15 July 1968 laying down general rules for intervention on the market in butter and cream (2), as last amended by Regulation (EEC) No 2045/91 (3), will be repealed with effect from 1 March 1995 by Regulation (EC) No 2807/94 (4); whereas the rules which have not been incorporated into Regulation (EEC) No 804/68 should be included in the detailed rules of application for intervention on the market in butter and cream provided for in Commission Regulation (EEC) No 685/69 (5), as last amended by Regulation (EC) No 393/94 (6); whereas, in view of the number of adjustments to be made to Regulation (EEC) No 685/69 and the many amendments which have already been made to that Regulation, it should be recast in the interests of clarity and transparency; whereas Regulation (EEC) No 685/65 should therefore be repealed;

Whereas the intervention agencies may buy in only butter which complies with the definition and fulfils the conditions laid down concerning quality and presentation; whereas, in view of the technical specifications regarding the determination of butter quality, the quality requirements to be met by butter and the methods of analysis and quality control procedures should be laid down; whereas, however, it should be possible for Member States to set up a system of self-checking on certain conditions;

Whereas the national quality classes for butter remain valid as a condition for eligibility for private storage aid and may also be indicated on the packaging of butter bought into intervention; whereas the relevant national classes must be determined;

Whereas the butter may be bought in by an intervention agency belonging to a Member State other than that on whose territory it was manufactured; whereas provision should be made in these circumstances for a means whereby the intervention agency which does the buying-in can verify that the conditions relating to quality and presentation are complied with, and it should be specified that the distance between the seller's warehouse and the frontier of the Member State where the intervention agency which does the buying-in is situated is not taken into account in calculating the maximum distance for the payment of additional transport costs;

Whereas, in order to ensure the proper functioning of the intervention arrangements, it is necessary to specify the conditions for the approval of manufacturing undertakings and verification of compliance therewith and to strengthen the obligations of the Member States with a view to the proper conservation of butter in storage by providing, in particular, for access to and identification of lots and insurance to cover the risks for the stored produce; whereas it is also necessary, in order to ensure a uniform frequency and level of controls, to specify the nature and number of inspections of storage premises to be performed by the national authorities;

Whereas, in order to ensure that no payment for butter bought in is made unduly, the trial period for the butter should be reduced and it should be specified that compliance with all the conditions laid down must be checked before payment; whereas the possibility afforded to sellers of replacing defective quantities of butter may be abolished without prejudicial consequences for those concerned;

Whereas, to facilitate checks on the presence of products stored under private storage contracts, there should be

(1) OJ No L 148, 28. 6. 1968, p. 13.
(2) OJ No L 169, 18. 7. 1968, p. 1.
(3) OJ No L 187, 13. 7. 1991, p. 1.
(4) OJ No L 298, 19. 11. 1994, p. 1.
(5) OJ No L 90, 15. 4. 1969, p. 12.
(6) OJ No L 53, 24. 2. 1994, p. 11.

provision for them to be removed from storage in lots unless the Member State authorizes a smaller quantity;

Whereas, in view of the current situation on the market in butter and cream, the date for the start of private storage operations should be postponed and the maximum storage period reduced; whereas, in order to make administration of the private storage scheme more transparent, the amount of the aid should be fixed annually;

Whereas, for the rest, the provisions concerning intervention measures, including disposal of butter held by the intervention agencies, have proven satisfactory and, subject to certain technical adjustments, may therefore remain unchanged;

Whereas the operative events for the agricultural conversion rate applicable to the public and private storage arrangements are laid down in Regulation (EEC) No 1765/93 [1], as last amended by Commission Regulation (EC) No 267/95 [2];

Whereas the Management Committee for Milk and Milk Products has not delivered an opinion within the time limit set by its chairman,

HAS ADOPTED THIS REGULATION:

Article 1

This Regulation lays down the detailed rules for the application of intervention measures in the milk and milk products sector as provided for in Article 6 of Regulation (EEC) No 804/68.

TITLE I

Buying-in and disposal of butter in public intervention

Article 2

Intervention agencies shall buy in only such butter as meets the definition laid down in Article 6 (1) of Regulation (EEC) No 804/68 and the quality requirements in Annex I, and for which the following conditions have been fulfilled:

(a) the quality has been checked in accordance with the analysis methods set out in Annexes I, III and IV hereto and from a sample taken in accordance with the procedure set out in Annex V hereto. However, where quality control is concerned, the Member States may, if the Commission agrees, set up a system of self-checking under their supervision in respect of certain quality requirements and in the case of certain approved undertakings;

(b) the radioactivity levels in the butter do not exceed the maximum levels permitted under Community regulations.

The levels applicable to products of Community origin shall be those fixed in Article 3 of Council Regulation (EEC) No 737/90 [3]. The level of radioactive contamination of the product shall be monitored if the situation so requires and during the period necessary only. Where necessary, the duration and scope of the controls shall be determined in accordance with the procedure laid down in Article 30 of Regulation (EEC) No 804/68;

(c) the butter was made during the 14 days preceding the day on which the intervention agency received the offer to sell it;

(d) the minimum quantity, presentation and packaging referred to in Article 4 of this Regulation are complied with.

Article 3

1. Undertakings as referred to in Article 6 (1) of Regulation (EC) No 804/68 shall be approved only if they:

(a) are approved pursuant to Article 10 of Council Directive 92/46/EEC [4] and have the appropriate technical equipment;

(b) undertake to keep permanent records in the form determined by the competent agency of each Member State, listing the origin of the raw materials, the quantities of butter obtained and the market preparation, identification and exit date of each production lot intended for public intervention;

(c) agree to submit their production of butter liable to be offered for intervention to a specific official inspection and, where applicable, to meet the requirements for the national classification referred to in Annex II;

(d) undertake to inform the competent agency responsible for inspection, at least two working days in advance, of their intention to manufacture butter for public intervention. The Member State, however, may set a shorter time limit.

2. To ensure compliance with this Regulation, the competent agencies shall carry out unannounced on-the-spot inspections, on the basis of the intervention butter production schedule of the plants concerned.

These inspections must include at least:

— one inspection per period of 28 days of manufacture for intervention with at least one inspection every six months, to examine the records referred to in paragraph 1 (b),

[1] OJ No L 161, 2. 7. 1993, p. 48.
[2] OJ No L 31, 10. 2. 1995, p. 6.
[3] OJ No L 82, 29. 3. 1990, p. 1.
[4] OJ No L 268, 14. 9. 1992, p. 1.

— one inspection every six months, to verify compliance with the conditions for approval referred to in paragraph 1.

3. Approval shall be withdrawn if the prior conditions laid down in paragraph 1 are no longer satisfied. Approval may be reinstated at the request of the enterprise concerned, after a minimum period of six months, following a thorough inspection.

Where an enterprise is found not to have complied with one of its undertakings as referred to in paragraph 1 (b), (c) and (d), except in cases of *force majeure*, approval shall be suspended for from one to 12 months depending on the seriousness of the irregularity.

The Member State may decide not to impose the said suspension where it is found that the irregularity was not committed deliberately or as a result of serious negligence and it is of minor importance with regard to the effectiveness of the checks provided for in paragraph 2.

4. A report must be drawn up on the inspections carried out pursuant to paragraphs 2 and 3 specifying:

— the date of inspection,

— its length,

— the operations carried out.

The report must be signed by the inspector responsible.

5. Member States shall inform the Commission of the measures taken with regard to the inspections provided for in paragraphs 2 and 3 within one month of their adoption.

6. Where the butter is offered to intervention in a Member State other than that in which it was produced buying-in shall be subject to the presentation of a certificate supplied by the competent agency of the Member State of production containing the information referred to in Article 4 (4) (a), (b) and (d).

Where the Member State of production has performed the checks referred to in Article 2 (a) the certificate shall also contain the results of those checks. In that case the packaging referred to in Article 4 (3) must be sealed by means of a numbered label issued by the competent agency of the Member State of production. The number must be entered on the certificate.

Article 4

1. The minimum quantity offered shall be 10 tonnes. Member States may provide for butter to be offered by the complete tonne only.

2. The butter shall be made up in blocks of at least 25 kilograms net.

3. The packaging shall be new, of strong material, and so designed as to ensure protection of the butter throughout transportation, storage and marketing.

4. The packaging shall be marked with at least the following, in code where appropriate:

(a) the approval number identifying the factory and the Member State of production;

(b) the date of manufacture;

(c) the date of entry into storage;

(d) the delivery number and package number;

(e) the words 'sweet cream' if the aqueous phase of the butter has the corresponding pH;

(f) the national quality class referred to in Annex II, where this is required by the Member State of production.

Member States may specify that if the person responsible for the storage depot undertakes to keep records in which the information specified in the previous subparagraph is inserted on the day of entry into storage, the obligation to mark the date of entry into store on the packaging shall not apply.

5. The intervention agency shall record the day on which the offer of sale was received, the corresponding quantities and dates of manufacture and the place where the butter offered is stored. After checking the details of the offer the intervention agency shall without delay issue a dated and numbered delivery order indicating:

(a) the quantity to be delivered;

(b) the timetable for delivery;

(c) the cold stores to which delivery must be made.

Article 5

1. For the purposes of this Title, the day of taking over shall be the day of entry of the butter into the cold store designated by the intervention agency but no earlier than the day following that on which the delivery order was issued. The butter shall be paid for between the 45th and the 65th day after the day of taking over, provided compliance with the requirements set out in Article 2 is verified.

2. The butter shall be put through a storage test period. This period shall be fixed at 30 days starting from the day of taking over.

3. By his offer the seller shall undertake that, where the inspection on entry into the store designated by the intervention agency shows that the butter does not meet the requirements laid down in Article 2, or where, at the end of the storage test period, the minimum organoleptic quality of the butter proves to be below that set in Annex I:

— he will take back the butter in question,

— he will pay the storage costs of the butter concerned from the day on which it was taken over until the date of its removal from storage.

Such storage costs shall be payable at a flat rate per tonne as follows:

(a) ECU 28,00 for fixed costs;

(b) ECU 0,45 per day of storage for cold storage costs.

The amounts shall be credited to the account of the European Agricultural Guidance and Guarantee Fund (EAGGF), Guarantee Section.

Article 6

1. The Member States shall lay down technical standards, providing for in particular a storage temperature of not more than − 15 °C, for the cold stores referred to in the third subparagraph of Article 6 (1) of Regulation (EEC) No 804/68 and shall take any other measures needed to ensure the proper conservation of the butter. The relevant risks shall be covered by insurance in the form either of a contractual obligation on storers or of a comprehensive coverage of the liability borne by the intervention agency; the Member States may also act as their own insurers.

2. The intervention agencies shall require that butter is placed and kept in storage on pallets and in easily identifiable and readily accessible lots.

3. The competent agency responsible for checks shall also perform the unannounced check on the presence of the products in store as provided for in Article 4 of Commission Regulation (EEC) No 618/90 (1).

Article 7

1. The intervention agency shall choose the available cold storage depot nearest to the place where the butter is stored.

However, provided that the choice of another cold storage depot does not result in additional storage costs it may:

(a) choose another depot situated within the distance referred to in paragraph 2;

(b) choose a storage depot situated beyond that distance if the resulting expenditure, inclusive of storage and transport costs, is smaller. In this case the intervention agency shall notify the Commission of its choice forthwith.

2. The maximum distance referred to in the fourth subparagraph of Article 6 (1) of Regulation (EEC) No 804/68 shall be 350 km. Beyond that distance, the additional transport costs borne by the intervention agency shall be ECU 0,065 per tonne and per kilometre.

However, where the intervention agency buying in the butter is in a Member State other than the one in whose territory the offered butter is stored, no account shall be taken, for calculating the maximum distance referred to in paragraph 2 of this Article, of the distance between the store of the vendor and the border of the Member State of the purchasing intervention agency.

3. The additional charges referred to in paragraph 2 shall be borne by the intervention agency only if the temperature of the butter does not exceed 6 °C on arrival at the depot.

Article 8

1. At the time of removal from store the intervention agency shall, in the case of delivery ex-cold store, make the butter available on pallets at the store's loading bay loaded, where appropriate, on to the means of transport where this is a lorry or a railway wagon. The costs involved shall be borne be the intervention agency and any stowage and depalletizing costs shall be borne by the purchaser of the butter.

2. Where butter is supplied as food aid paragraph 1 shall apply.

3. Other provisions on the disposal of butter held by intervention agencies shall be determined case-by-case.

TITLE II

Aid for private storage of butter or cream

Article 9

Contracts relating to the private storage of butter or cream as referred to in Article 6 (2) of Regulation (EEC) No 804/68 shall be concluded between the intervention agencies of the Member States and natural or legal persons, hereinafter called 'contractors'.

Article 10

1. A private storage contract may be concluded only if the butter or cream referred to in Article 6 (2) of Regulation (EEC) No 804/68 was manufactured in an undertaking approved in accordance with Article 3 (1) during the 28 days preceding the day of commencement of contractual storage. The butter must correspond to the national quality class of the Member State of production referred to in Annex II and must not exceed the maximum permitted levels of radioactivity referred to in Article 2 (b).

(1) OJ No L 67, 15. 3. 1990, p. 21.

A storage contract may not be concluded for butter or cream :

(a) in respect of which an application for a direct consumption aid has been made under other Community provisions ;

(b) which has been placed under the procedure referred to in Article 5 (1) of Council Regulation (EEC) No 565/80 (1) ; subsequent placing under this procedure shall be regarded as removal from store within the meaning of paragraph 7.

2. For the purposes of this Regulation a lot shall be a quantity of butter or cream weighing at least one tonne and of homogeneous composition and quality from a single approved undertaking, taken into storage in a single warehouse on a single day.

3. Where the butter is stored in a Member State other than the Member State of production conclusion of the storage contract shall be conditional on the presentation of a certificate supplied by the competent agency of the Member State of production containing the information specified in paragraph 6 (a), (b) and (d) and confirming that the butter corresponds to the national quality class of the Member State of production, as referred to in Annex II.

4. Storage contracts shall be concluded in writing for one or more lots and shall include, in particular, provisions concerning :

(a) the quantity of butter or cream to which the contract applies ;

(b) the amount of the aid, without prejudice to Article 16 ;

(c) the dates relating to the execution of the contract, taking account of the second sentence of the fourth subparagraph of Article 6 (2) of Regulation (EEC) No 804/68 ;

(d) the identity of the cold stores.

5. The control measures, particularly those referred to in Article 11, and the information referred to in paragraph 6 shall be the subject of specifications drawn up by the intervention agency of the Member State of storage. The storage contract shall refer to these specifications.

6. The specifications shall provide that the packaging of each lot is to bear at least the following information, which may be coded :

(a) the approval number identifying the factory and the Member State of manufacture ;

(b) the date of manufacture ;

(c) the date of entry into store ;

(d) the number of the lot ;

(e) the word 'salted' in the case of butter as referred to in the third indent of the first subparagraph of Article 6 (2) of Regulation (EEC) No 804/68 ;

(f) the national quality class referred to in Annex II ;

(g) the net weight.

Member States may provide that the obligation to indicate the date of entry into store on the packaging does not apply if the store manager undertakes to keep a register in which the particulars referred to in the preceding subparagraph will be entered on the date of entry into store.

7. The day of commencement of contractual storage shall be the day following that of entry into store. The last day of contractual storage shall be the day before that of removal from storage.

Applications for aid by storers may relate only to lots of butter or cream the entry into store of which has been completed.

Applications must reach intervention agencies within at most 30 days of the date of entry into store. Intervention agencies shall register their dates of arrival. However, where the application reaches the intervention agency within five working days following the abovementioned deadline the storage contract may be concluded but the aid will be reduced by 30 %.

Storage contracts shall be concluded within at most 30 days of the date of registration of the application.

In the case referred to in paragraph 3, storage contracts shall be concluded within at most 60 days of the date on which the application is registered.

Article 11

1. The Member State shall ensure that all the conditions granting entitlement to payment of the aid are fulfilled.

2. The contractor shall make available to the competent agency reponsible for verifying execution of the measure any documentation permitting the following particulars of products placed in private storage to be verified :

(a) the approval number identifying the factory and the Member State of manufacture ;

(b) the date of manufacture ;

(c) the date of entry into storage ;

(d) the lot number ;

(e) presence in the store ;

(f) the date of removal from storage.

3. The contractor or, where applicable, in his place the operator of the store shall for each contract keep stock records available at the store, covering :

(1) OJ No L 62, 7. 3. 1980, p. 5.

(a) identification, by lot number, of the products placed in private storage;

(b) the dates of entry into and removal from storage;

(c) the quantity of butter or cream, indicated per lot;

(d) the location of the products in the storeroom.

4. Products stored must be easily identifiable, easily accessible and identified individually by contract.

5. On entry into storage or during the 21-day period following the date on which the aid application was registered, the competent agency shall conduct checks to ensure that products stored are eligible for aid. These checks shall involve checking the weight and identity of the products, as well as checking their nature by sampling.

6. The national authorities responsible for checks shall undertake:

(a) — either the sealing of the products by contract, lot or smaller quantity at the time of the checks provided for in paragraph 5,

— or an unannounced check, by sampling, to see that the products are present in the storeroom. The sample concerned must be representative and must correspond to at least 10 % of the overall quantity under contract for a private storage aid measure;

(b) a check, by sampling, to verify the weight and identity of the products when they leave the store. For this purpose the storer shall inform the competent agency at least five working days before the start of removal operations, indicating the lots to be removed. The Member State, however, may accept a shorter time limit.

7. Checks conducted pursuant to paragraphs 5 and 6 must be the subject of a report stating:

— the date of the check,

— its duration,

— the operations conducted.

The report must be signed by the inspector responsible.

8. In the case of irregularities affecting at least 5 % of the quantities of products subjected to the checks, the latter shall be extended to a larger sample to be determined by the competent agency.

Article 12

1. Private storage aid as provided for in Article 6 (2) of Regulation (EEC) No 804/68 may be granted only where the storage period is at least 90 days. The amount of the aid may not exceed an amount corresponding to a storage period of 210 days.

2. Entry into store may take place only between 15 April and 15 August of the same year. Removal from store may take place only from 16 August of the year of storage.

3. Private storage aid may be granted only where the storage period is at least 90 days. Removal from store shall be in whole lots or, if the competent agency so authorizes, in smaller quantities. However, in cases as referred to in the first indent of Article 11 (6) (a) only a sealed quantity may be removed from store.

4. The amount of the aid referred to in Article 6 (2) of Regulation (EEC) No 804/68 shall be determined each year in accordance with the procedure laid down in Article 30 of that Regulation for private storage contracts which commence during that year.

If the market situation so requires, the amount of the aid, the periods of entry to and removal from storage and the maximum length of storage may be altered during the year in respect of contracts still to be concluded.

5. The aid shall be paid on application by the interested party at the latest within 120 days of the last day of contractual storage. However, if it has been necessary to commence an administrative inquiry into entitlement to the aid, payment shall not be made until entitlement has been recognized.

After 60 days of contractual storage a single advance payment of the aid may be made at the storer's request, on condition that he lodges a security equal to the advance payment plus 10 %. The advance payment shall be calculated on the basis of a storage period of 120 days.

Article 13

1. Where, at the end of the first 60 days of storage, the deterioration in the quality of the butter or cream is greater than is normal in store, contractors may be authorized, once per lot, to replace the defective quantity, at their own expense, by an equal quantity of butter or cream as specified in Article 6 (2) of Regulation (EEC) No 804/68.

2. In the case referred to in paragraph 1, for the purpose of calculating the aid, the first day of contractual storage shall be the day of commencement of storage indicated in the contract in accordance with the first subparagraph of Article 10 (7).

Article 14

1. As regards butter and by way of derogation from Article 12 (3), on the expiry of a period of contractual storage of 60 days, the contractor may release from storage

one or more lots under contract, or a smaller quantity if the competent agency authorizes this, provided that within 60 days following its release from storage, the butter, as such or after processing into butteroil :

— has left the customs territory of the Community,

— has reached its destination in the cases referred to in Article 5 (1) of Commission Regulation (EEC) No 365/87 (1), or

— has been placed in a victualling warehouse approved pursuant to Article 38 of Regulation (EEC) No 3665/87.

The contractor shall inform the intervention agency thereof at least five working days before the commencement of stock release operations, indicating the quantities intended for export. The Member State, however, may accept a shorter time limit.

2. In the case referred to in paragraph 1 :

(a) evidence of export shall be provided as in the case of refunds, pursuant to Regulation (EEC) No 3665/87 ;

(b) the aid determined in accordance with Article 12 (4) shall be calculated by reference to the reduction in the duration of storage ;

(c) if the time limit of 60 days referred to in paragraph 1 is not complied with, the amount of the aid for the quantity in question shall be reduced by 15 %, and by an additional 5 % in respect of the part remaining per day by which the 60-day period is exceeded ;

(d) the security referred to in the second subparagraph of Article 12 (5) shall be released once evidence of export is provided pursuant to (a), minus an amount corresponding to the reduction in the aid as referred to in (b).

Article 15

1. Aid for the storage of cream may be granted only for pasteurized cream the fat content of which is not less than 35 % and not more than 80 %.

However, Member States may permit contractors to undertake voluntarily to observe, for all lots of all contracts concluded during the milk year, a single minimum fat content fixed in advance within the limits specified in the preceding subparagraph.

2. For the purpose of calculating the aid the quantities of cream shall be converted into butter equivalent by reference to butter with a fat content of 82 % by multiplying the fat content of the cream by 1,20.

3. The fat content specified in the first subparagraph of paragraph 1 shall be checked prior to freezing of the cream by a laboratory approved by the competent agency.

4. Where the second subparagraph of paragraph 1 is applied, the aid shall be granted on the basis of the minimum fat content fixed in advance.

In such cases Member States shall check the fat content in accordance with paragraph 3 by random sampling in the course of frequent unannounced visits.

If such a check reveals a minimum fat content lower than that fixed in advance the following provisions shall apply to all lots taken into store since the last satisfactory check :

(a) no aid shall be paid for these lots. However, if that fat content is found to be less than 2 % lower than the minimum content fixed in advance, the aid shall be paid on the basis of the fat content found but 10 % of the amount shall be deducted ;

(b) the second subparagraph of paragraph 1 shall not be applicable to the storer concerned for the remainder of the storage period.

Article 16

Where the maximum buying-in price fixed by invitation to tender in accordance with Commission Regulation (EEC) No 1589/87 (2) expressed in national currency and applying on the day of commencement of contractual storage :

(a) is higher than that applying on the last day of contractual storage, the aid determined in accordance with Article 12 (4) shall be increased by an amount equal to any reduction in the maximum buying-in price in excess of 2 % of the price applying on the day of commencement of contractual storage ;

(b) is lower than that applying on the last day of contractual storage, the aid determined in accordance with Article 12 (4) shall be reduced by an amount equal to any increase in the maximum buying-in price in excess of 2 % of the price applying on the day of commencement of contractual storage. However, the reduction in the aid may not exceed the total aid.

(1) OJ No L 351, 14. 12. 1987, p. 1.

(2) OJ No L 146, 6. 6. 1987, p. 27.

Article 17

Regulation (EEC) No 685/69 is hereby repealed. However, it shall continue to apply to private storage contracts concluded before the entry into force of this Regulation.

References to Regulation (EEC) No 685/69 shall be understood as references to this Regulation.

Article 18

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

It shall apply from 1 March 1995.

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

Done at Brussels, 28 February 1995.

For the Commission

Franz FISCHLER

Member of the Commission

ANNEX I

COMPOSITIONAL REQUIREMENTS, QUALITY CHARACTERISTICS AND ANALYTICAL METHODS

Butter is a solid emulsion mainly of the water-in-oil-type with the following composition and quality characteristics:

Parameters	Content, quality characteristics	Reference method
Fat	Minimum 82 %	IDF-Standard 80/1977
Water	Maximum 16 %	IDF-Standard 80/1977
Solids non-fat	Maximum 2 %	IDF-Standard 80/1977
Free fatty acids (1)	Maximum 1,2 mmol/100 g fat	IDF-Standard 6B/1989
Peroxide value (1)	Maximum 0,3 mequiv. oxygen/1 000 g fat	IDF-Standard 74A/1991 Milkfat isolation: as described in IDF-Standard 6B/1989 (protection from light)
Coliforms	Not detectable in 1 g	IDF-Standard 73A/1985
Non-milk fat (1)	Not detectable by triglyceride analysis	Annex III
Sterol tracers (2)	Not detectable	Commission Regulation (EC) No 86/94 (OJ No L 17, p. 7, 20. 1. 1994)
Other tracers (2)	Not detectable	Methods approved by competent authority
Sensory characteristics	At least 4 out of 5 points for appearance, flavour and consistency	Annex IV
Water dispersion	At least 4 points	IDF-Standard 112A/1989

(1) The standards for these three parameters shall be applicable six months after publication in the Official Journal. Based on the data obtained in Member States and submitted to the Commission, the standards will if necessary be revised.

(2) Tracers accepted under Regulations (EEC) No 3143/85, (EEC) No 570/88 and (EEC) No 429/90.

ANNEX II

NATIONAL QUALITY CLASS

— 'beurre marque de contrôle' as regards Belgian butter,

— 'smør of første kvalitet' as regards Danish butter,

— 'Markenbutter' as regards German butter,

— 'pasteurisé A' as regards French butter,

— 'Irish creamery butter' as regards Irish butter,

— 'produced exclusively from cream which has been subjected to centrifugal and pasteurizing treatment' as regards Italian butter,

— 'Marque Rose' or 'Beurre de première qualité' as regards Luxembourg butter,

— 'Extra kwaliteit' as regards Dutch butter,

— 'Extra selected' as regards Great Britain butter and 'premium' as regards Northern Ireland butter,

— 'produced exclusively from cream which has been subjected to centrifugal and pasteurizing treatment' as regards Greek butter,

— 'produced exclusively from pasteurized cow's milk or cream' as regards Spanish butter,

— 'produced exclusively from pasteurized cow's milk or cream' as regards Portuguese butter,

— 'Teebutter' as regards Austrian butter,

— 'meijerivoi/mejerismör' as regards Finnish butter,

— 'svenskt smör' as regards Swedish butter.

ANNEX III

REFERENCE METHOD FOR THE DETECTION OF FOREIGN FATS IN MILK FAT BY GAS CHROMATOGRAPHIC ANALYSIS OF TRIGLYCERIDES — REVISION 1

1. Scope and field of application

This standard lays down a method for the detection of foreign fats, of both vegetable fats and animal fats such as beef tallow and lard in milk fat of milk products using gas chromatographic analysis of triglycerides.

Using defined triglyceride formulae vegetable and animal fats are qualitatively and quantitatively detected in pure milk fat irrespective of feeding or lactation conditions.

Note 1: Although butyric acid (C4) occurring exclusively in milk fats enables quantitative estimations of low to mean amounts of milk fat in vegetable fats to be made, qualitative and quantitative information can hardly be provided in the range of an addition of up to at least 20 % (weight %) foreign fat to pure milk fat because of the large variation of C4 ranging approximately between 3,5 to 4,5 % (weight %).

Note 2: Quantitative results can practically only be obtained by triglyceride analyses, because the sterol content of the vegetable fats is different as a function of production and treatment conditions.

2. Definition

Foreign fats in milk fat: foreign fats as defined in this standard are all vegetable and animal fats except milk fat.

3. Principle of the method

After extraction of the milk fat a stock solution is prepared.

From this solution the triglycerides (total carbon numbers) are determined gas chromatographically on packed columns. By inserting the weight % of the fat molecules of different size (C24 — C54 — only even numbers) in the triglyceride formula the foreign fats are either qualitatively detected or quantitatively determined.

Note: Observing the evaluation described here capillary gas chromatography can be used, if it is guaranteed that comparable results are achieved (1).

4. Reagents

Analysis-grade chemicals must be used.

4.1. Carrier gas: nitrogen, purity degree ≥ 99,996 %.

4.2. Standard triglycerides (2), saturated as well as cholesterol for standardizing a standard milk fat according to section 6.5.4.

4.3. Methanol, water-free.

4.4. n-Hexane

4.5. n-Heptane

4.6. Toluene

4.7. Dimethylchlorosilane solution: 50 ml dimethylchlorosilane are dissolved in 283 ml toluene.

4.8. Combustible gas: hydrogen and synthetic air

4.9. Stationary phase, 3 -% OV-1 on 125/150 µm (100/120 mesh) Gas ChromQ (3).

4.10. 10 % cocoa butter solution

(1) Suited methods have already been described, see D. Precht and J. Molkentin: Quantitative triglyceride analysis using short capillary columns, Chrompack News 4, 16-17 (1993).
(2) Suitable products are commercially available.
(3) Trade names such as, e.g., Extrelut, Gas ChromQ, Chrompack are examples for suited products available in the specialized trade. This information shall serve the purpose of easy handling of the standard by the user and does not represent a request of the product. The indication of grain was transferred to the SI-unit µm according to BS 410 :1988 'British Standard Specification for test sieves'.

5. **Instruments**

Normal laboratory apparatus and particularly the following:

5.1. High temperature gas chromatograph suited for temperatures of at least 400 — 450 °C, equipped with a flame ionization detector (FID) and constant mass flow controller for the carrier gas. Combustion gas: 30 ml/min H_2, 270 ml/min synthetic air.

Note: Because of the high temperatures occurring during triglyceride analyses the glass inserts in the FID or in the injector system must be frequently cleaned.

The gas chromatograph must be equipped with septa, withstanding high temperatures, which can be frequently used and exhibit generally a very low degree of 'bleeding'.

Note: Suited are Chromblue (tm) septa (Chrompack).

The septa must be exchanged at regular intervals, e.g. after roughly 100 injections or as soon as the resolution deteriorates (see figure 4).

5.2. Chromatography column

U-shaped glass column (inside diameter 2 mm, 500 mm in length), which is first silanized according to section 6.1 with dimethylchlorosilane in order to deactivate the glass surface.

Note: Suited are also somewhat longer (80 — 200 mm in length) packed columns. With them a slightly better reproducibility of the results can be achieved. On the other hand, the stationary phase exhibits occasionally fractures after operation, which may lead, in turn, to worse quantitative results. Further, the FID flame is easily extinguished as a result of the required extremely high carrier gas flow of 75 to 85 ml/min.

5.3. Arrangement for filling the column (see figure 1)

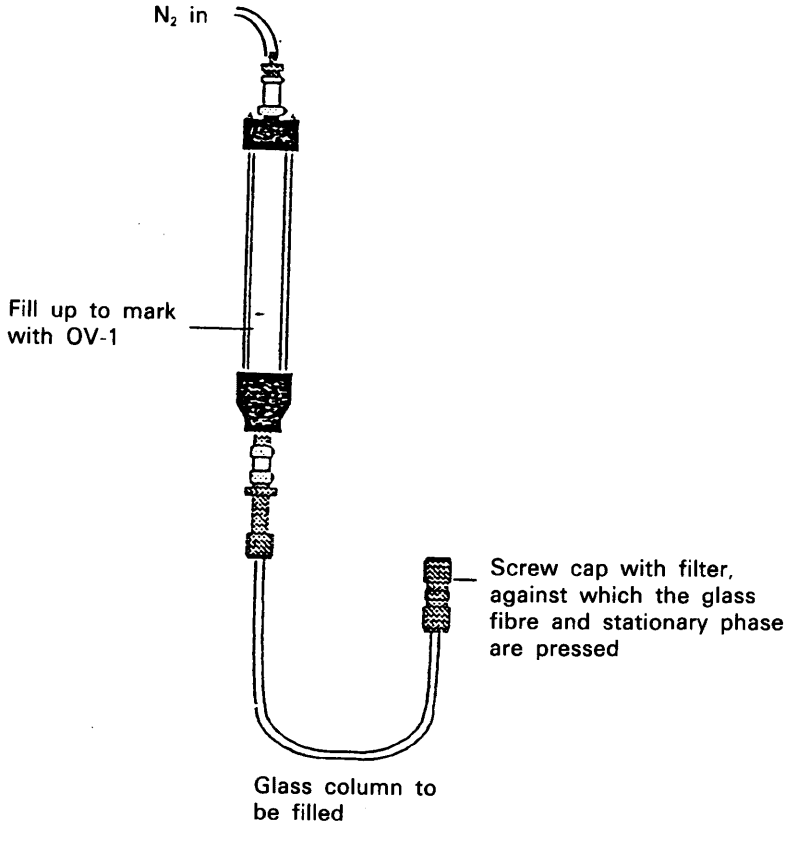


Figure 1: Filling of the column

5.3.1. Plastic column with screwed-on end caps, provided with a mark up to which the required quantity of stationary phase can be filled

5.3.2. Fine sieve (mesh size approximately 100 µm) with screw cap, suited for sealing the glass column according to figure 1.

5.3.3. Deactivated, silanized glass wool

5.3.4. Vibrator for uniform distribution of the stationary phase during filling

5.4. 1 to 3 ml Extrelut column (1) with silica gel. This column can alternatively be used for the extraction for obtaining milk fat

5.5. Graphite seal 6,4 mm (1/4") with 6 mm bore

5.6. Devices for silanizing the glass surface of the column according to section 6.1.

5.6.1. Woulff bottle

5.6.2. Water suction pump

5.7. Water bath, adjustable to (50 ± 2) °C

5.8. Drying cabinet, adjustable to (50 ± 2) °C and (100 ± 2) °C

5.9. Microlitre pipette

5.10. 5 ml graduated pipette for dosing 1,5 ml methanol

5.11. 50 ml round-bottomed flask

5.12. Erlenmeyer flask, nominal volume 50 ml

5.13 Funnel

5.14. Fine-pored filter

5.15. Rotary evaporator

5.16. Ampoules, nominal volume 1 ml, sealable with an aluminium cap, with a septum in the interior

5.17. Injection syringe, the plunger of the syringe used must not reach into the tip of the needle.

Note: Such syringes allow a better reproducibility of the results to be obtained.

In order to avoid deterioration of the septum, the tip of the needle should be checked at regular intervals (e.g. with a stereomicroscope).

6. Procedure

6.1. Preparation of the column (silanization)

After connecting the Woulff bottle, as shown in figure 2, with the water suction pump tube 2 is dipped into the solution according to section 4.7. By closing the stopcock the column is filled; subsequently the two tubes are removed.

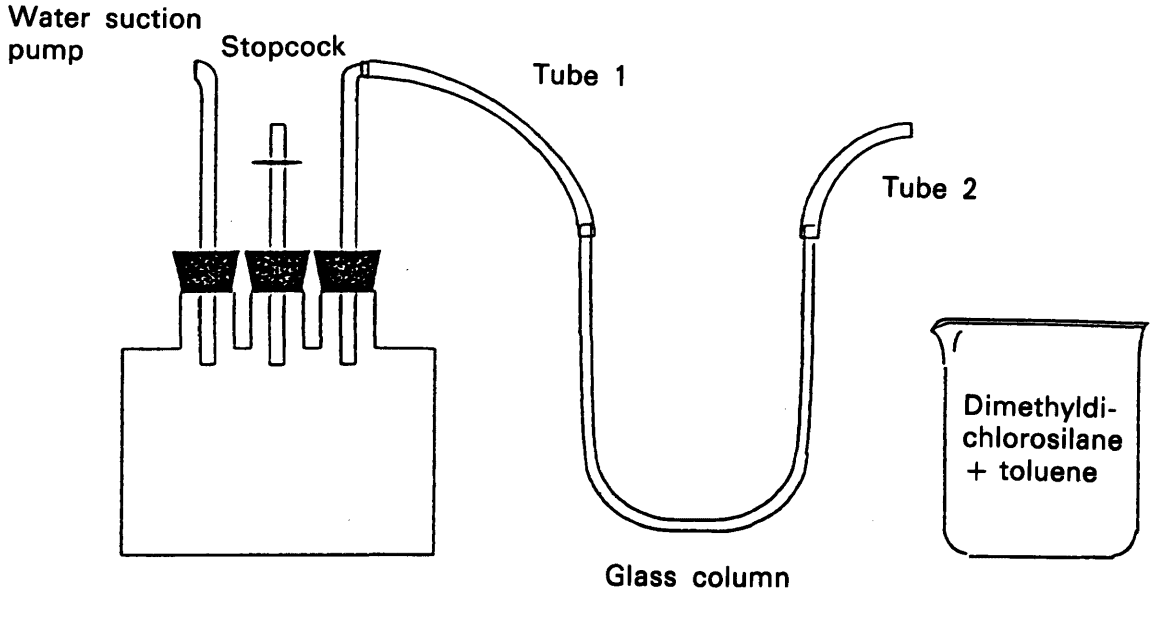


Figure 2: Arrangement for silanization

(1) See footnote 3 on page 11.

The column is fixed on a stand and completely filled with the dimethyldichlorosilane solution by means of a pipette.

After 20 — 30 min the Woulff bottle is replaced by a filter flask and the column emptied by connecting it with the water suction pump (see figure 3).

6.2. Filling of the column

This is followed by successive rinsing using 75 ml toluene and 50 ml methanol ; then the emptied column is dried in the drying cabinet at 100 °C for approximately 30 minutes.

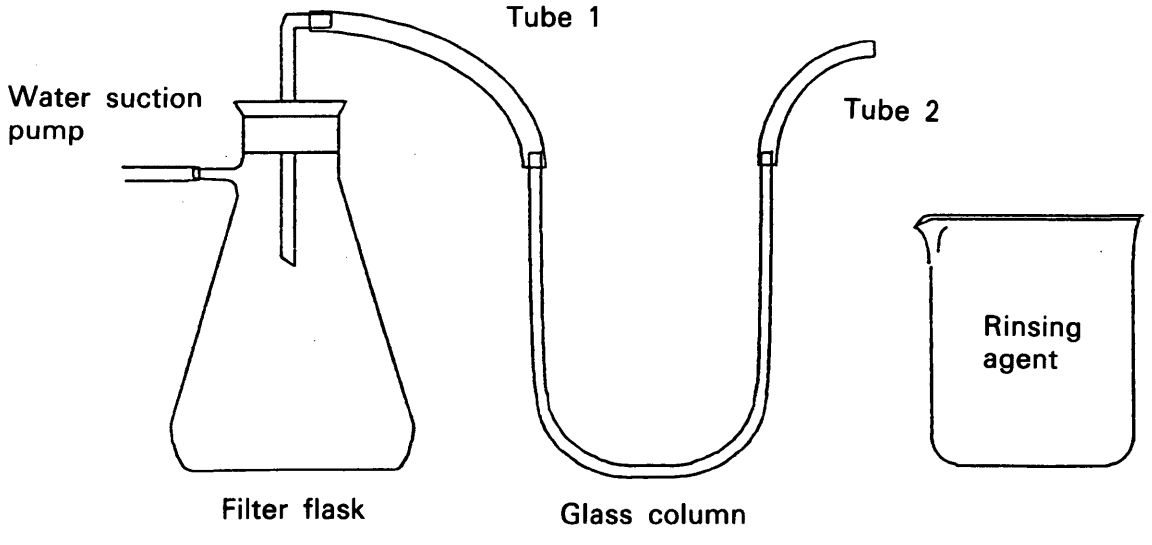


Figure 3 : Arrangement for rinsing

For filling of the column the arrangement as represented in figure 1 is used. The stationary phase according to 4.9 is filled into the plastic column up to the mark. The lower end of the glass column to be filled is sealed with an approximately 1 cm long plug of glass wool, which had been silanized before, and which is pressed in using a steel rod. Then the end of the column is closed with the sieve according to section 5.3.2.

The column is filled under pressure (3 bar, with N_2) with the stationary phase. To obtain a uniform, continuous and firm packing, a vibrator is moved up and down the glass column during filling.

After filling a solid plug of silanized glass wool is pressed into the other end of the packed column, the protruding ends are cut off and the plug pressed into the column a few millimetres with a spatula.

6.3. Preparation of the samples

For sample preparation one of the three following methods is used :

6.3.1. Isolation of the milk fat from butter

5 to 10 g of butter is melted in a suitable vessel in a water bath according to section 5.7 at 50 °C.

A 50 ml Erlenmeyer flask and a funnel with inserted filter according to section 5.14 are heated in the drying cabinet to 50 °C. The fat layer of the molten butter sample is filtered using the preheated device.

Such a milk fat is almost phospholipid-free.

6.3.2. Extraction of the fat fraction according to the Röse-Gottlieb method

Extraction is done either according to IDF Standard 1C : 1987, 16C : 1987, 116A : 1987 or 22B : 1987.

With such a milk fat phospholipids allow a cholesterol peak to be obtained which is increased by approximately 0,1 %.

The triglyceride spectrum standardized to 100 with the cholesterol is thereby influenced only to a negligible extent.

6.3.3. Extraction from milk using silica gel columns

0,7 ml of a milk sample tempered to 20 °C are applied to a 1 to 3 ml Extrelut column with a micro-litre pipette according to section 5.4 and allowed to distribute uniformly on the silica gel for approximately five minutes.

For denaturing the protein-lipid complexes 1,5 ml of methanol is added by pipette. Subsequently the sample is extracted with 20 ml n-hexane. The n-hexane is slowly added in small amounts and the solvent draining off collected in a 50 ml round-bottomed flask that had been dried to a constant, known weight.

After extraction led the column drain until empty.

From the eluate the solvents are distilled off on a rotatory evaporator at a water bath temperature of 40 to 50 °C.

The flask is dried and the fat yield determined by weighing.

Note : Fat extractions according to Gerber, Weibull-Berntrop, Schmid-Bondzynski-Ratzlaff or isolation of milk fat using detergents (BDI method) are not suited for triglyceride analysis, because with these methods more or less large quantities of partial glycerides or phospholipids can pass into the fat phase.

6.4. Preparation of the sample solution

For gas chromatography a 5 % solution of the fat in n-heptane obtained according to section 6.3 is used. For preparing this sample solution corresponding amounts of the sample material obtained according to the sections 6.3.1 and 6.3.2 are weighed and dissolved in corresponding amounts of n-heptane.

With sample preparation according to section 6.3.3 the amount of n-heptane to be added to the sample material in the flask is calculated on the basis of weighing and the remainer dissolved in it.

Approximately 1 ml of the sample solution is filled into an ampoule according to section 5.16.

6.5. Chromatographic triglyceride determination

With the high temperatures of up to 350 °C for eluting the long-chain triglycerides C52-C56 an increase in baseline occurs easily, particularly if the columns have not been adequately conditioned in the beginning. This rise in baseline at high temperatures can be avoided completely by either combining two columns or baseline subtraction.

With the compensating mode or operation with single columns, as well as for the glass inserts in the injector and in the detector the graphite seals according to section 5.5 have to be used.

6.5.1. Baseline correction

To avoid baseline rising one of the four methods is used :

6.5.1.1. Combination of columns

Two packed columns are used in compensating mode.

6.5.1.2. Baseline correction by the gas chromatograph

By application of a run by the gas chromatograph without injection of a fat solution and subsequent subtraction of the stored baseline rising of the baseline can be avoided.

6.5.1.3. Baseline correction by integration software

By application of a run by the integration system without injection of a fat solution and subsequent subtraction of the stored baseline rising of the baseline can be avoided.

6.5.1.4. Baseline correction by adequate conditioning

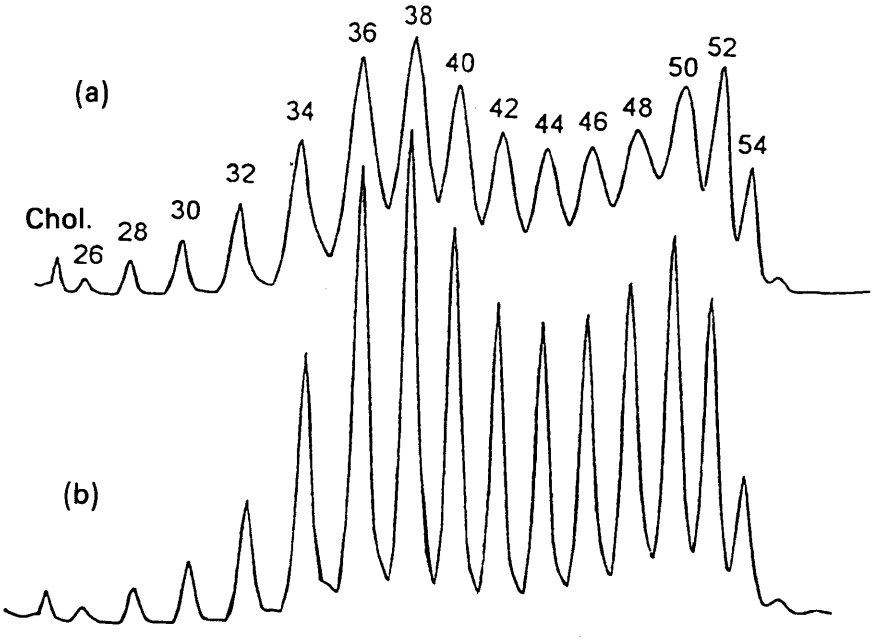
With adequate initial conditioning of the column and approximately 20 injections with milk fat solutions baseline rising at high temperatures is frequently so low that baseline corrections are not necessary.

6.5.2. Injection technique

To avoid discrimination effects the 'hot injection' technique is applied to achieve better quantitative results with the high-boiling triglyceride components. Here, the fat solution is drawn up in the syringe and the cold needle of the syringe warmed up prior to injection for approximately three seconds in the injector head. Then, the syringe content is rapidly injected.

Note: With this injection technique the risk of fractionation phenomena inside the syringe or the injection block is reduced. 'On-column' direct injection in the upper, extended heated part of the column is not applied, because the fragments of the septum, which accumulate here, as well as contaminations can be easily eliminated with the used technique by regularly changing an injector insert without dismounting the column.

Bending of the tip of the needle caused by touching the bottom of the sample beaker (even if it is hardly visible to the eye) must be absolutely avoided in order not to damage the septum.



(a) bad resolution as a result of a damaged septum

b) good resolution

Figure 4: Triglyceride chromatogram of a milk fat sample

6.5.3. Conditioning of a packed column

During steps (a) to (c) the top of the column is not connected to the detector to avoid contamination.

The columns filled according to section 6.2 are conditioned as follows:

(a) 15 min 40 ml/min N_2-flow at 50 °C;

(b) Heating with 1 K/min up to 355 °C at 10 ml N_2/min;

(c) Holding for 12 to 15 h at 355 °C;,

(d) two injections of 1 µl of the cocoa butter solution according to section 4.10 and respective temperature program;

e) 20 injections of 0,5 µl of a milk fat solution for two to three days according to section 6.4.

Note : Cocoa butter consists almost exclusively of high-boiling C50 — to C56 triglycerides. Injection with cocoa butter serves the purpose of special conditioning in this long-chain range. With the high-boiling triglycerides C50 to C54 partly response factors of up to approximately 1,20 may occur. Normally, with repeated injection of a milk fat solution a reduction of the initially high response factors for C50 to C54 has to be expected. With triglycerides with low acyl-c number the factors approximate 1.

Three pairs, respectively, of the columns filled according to section 6.2 are prepared. The conditioned pairs are checked, respectively, with a milk fat analysis for routine testing.

The pair with the best quantitative results (response factors almost 1) is used in the following. With response factors > 1,20 the column is not used.

6.5.4. Calibration

For calibration the response factors of the corresponding triglycerides, as well as of cholesterol of a milk fat (standardized fat) should be determined using the standardized triglycerides (at least the saturated triglycerides C24, C30, C36, C42, C48 and C54, as well as cholesterol ; better still additionally C50 and C52). Intermediate response factors can be found by mathematical interpolation.

Using the standardized fat two to three calibrations have to be performed every day. If almost identical results are obtained, well reproducible quantitative results are achieved with triglyceride analysis of the samples.

The standardized milk fat has a stock life of several months at a storage temperature of maximally −18 °C and can, thus, be used as a standard.

6.5.5. Temperature programme, carrier gas and other conditions for triglyceride analysis

Temperature programme : initial column temperature 210 °C, hold for one minute, then program at 6 °C/min to 350 °C and hold at final temperature for five minutes.

Detector- and injector temperature : 370 °C, respectively.

Note : Detector, injector, and oven temperatures (initial temperature) should be maintained at a constant level (also overnight, during weekends and holidays).

Carrier gas : nitrogen, flow rate 40 ml/min.

Note : If 80 cm columns are used, the flow must be at least 75 ml/min N_2. The carrier gas flow must be constantly maintained (also overnight, as well as during weekends and holidays). The exact carrier gas flow should be adjusted in a manner that independent of column length C54 is eluted at 341 °C.

Duration of analysis : 29,3 minutes.

Injection volume : 0,5 µl.

Note : The syringe has to be rinsed several times with pure heptane after each injection.

FID conditions : according to section 5.1

Note : The flame ionization detector is ignited, respectively, at the beginning of each working day.

7. Integration, evaluation and control of the measuring conditions

Triglycerides with odd acyl-c number (2n + 1) are combined with the preceding even-numbered triglyceride (2n). The less reproducible low C56 contents are not taken into account. The remaining triglycerides (peak area) in the chromatogram, including cholesterol (peak near to C24) are multiplied by the respective response factors of the standard fat (last calibration) and altogether normalized to 100. Besides free cholesterol the triglycerides C24, C26, C28, C30, C32, C34, C36, C38, C40, C42, C44, C46, C48, C50, C52 and C54 are, thus, evaluated. Results are given in weight % (g/100 g).

Evaluation of the chromatogram peaks should be done with an integrator, with which the baseline can be plotted. Reintegration with optimized integration parameters should be possible.

Figures 5 and 6 demonstrate two examples of triglyceride chromatograms. Figure 5 shows a chromatogram which can be well evaluated, whilst figure 6 represents a sporadic error in the C50 to C54 range, the baseline running incorrectly compared with figure 5. Such typical errors can be detected with a high degree of certainty and avoided only by use of an integrator with which the baseline is plotted.

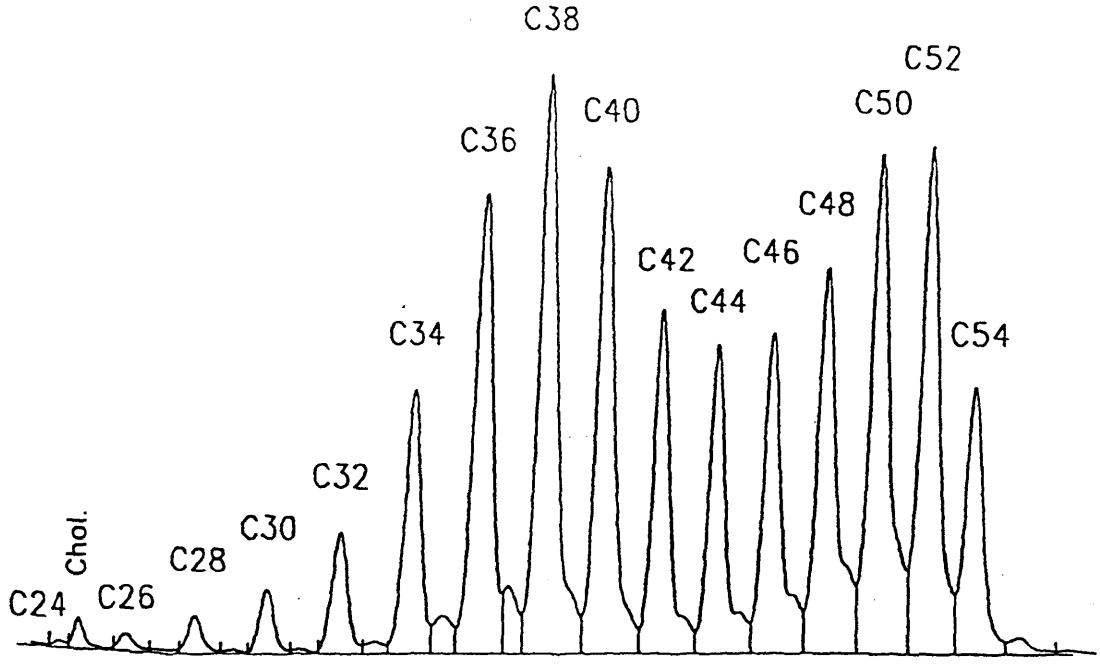


Figure 5 : Easy-to-evaluate triglyceride chromatogram of a milk fat with base line drawn in

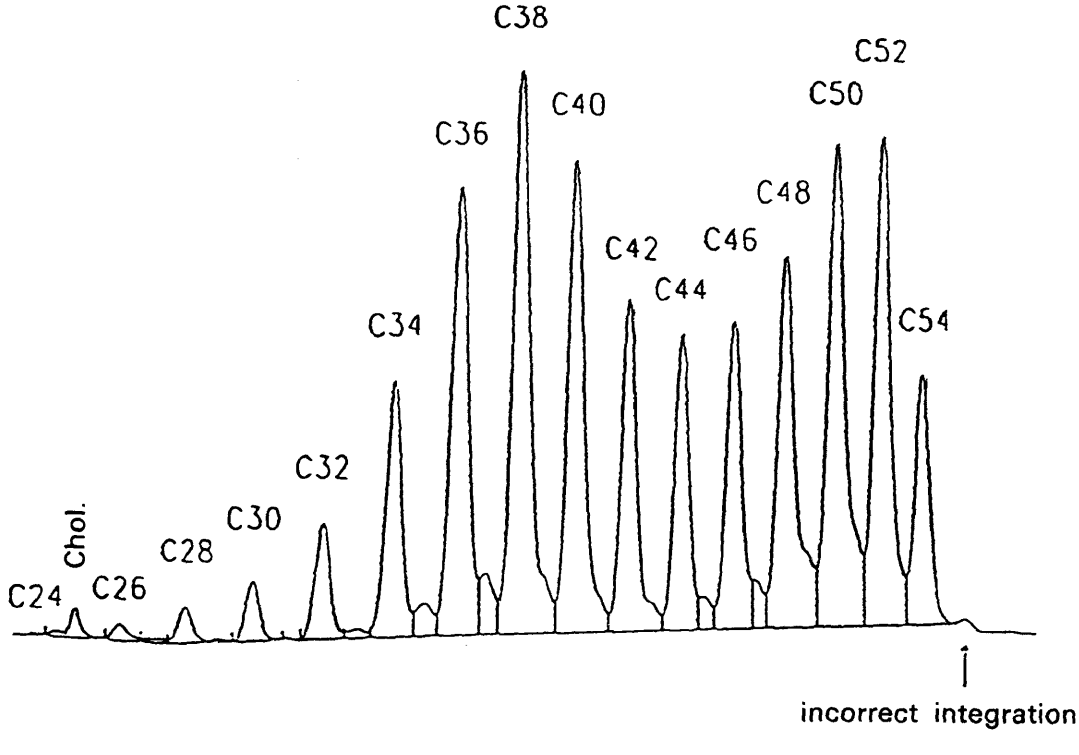


Figure 6 : Wrongly integrated chromatogram of milk fat

For controlling the measuring conditions Table 1 shows the mean values and standard deviations (SD) of a typical winter milk fat for the different triglycerides from 19 analyses of the same fat :

Table 1 : Triglyceride composition of a milk fat
Mean values and SD from 19 analyses

(g/100 g)

Triglyceride	Mean	SD
C24	0,04	0,004
C26	0,26	0,007
C28	0,66	0,020
C30	1,31	0,023
C32	2,92	0,030
C34	6,73	0,053
C36	12,12	0,030
C38	12,92	0,054
C40	9,70	0,019
C42	7,62	0,020
C44	7,35	0,025
C46	7,91	0,029
C48	9,09	0,048
C50	9,97	0,038
C52	7,76	0,042
C54	3,32	0,020

With SD deviating to a greater extent compared with the values in Table 1, the chromatograms are not tolerable any more and the septa or the gas flow should be checked. Further, small components of the septum may have formed deposits on the glass wool at the entrance of the column or the column has become unsuited for use as a result of ageing, temperature influences etc. (see figure 3).

8. Qualitative foreign fat detection

For the detection of foreign fats triglyceride formulae (Table 2) with limits S (Table 3) have been developed, in which the S-values of pure milk fats can fluctuate. If these limits are transgressed, the presence of a foreign fat can be assumed.

The most sensitive formula for the detection of lard addition is, e.g.

$$6{,}5125 \cdot C26 + 1{,}2052 \cdot C32 + 1{,}7336 \cdot C34 + 1{,}7557 \cdot C36 + 2{,}2325 \cdot C42 + 2{,}8006 \cdot C46 + 2{,}5432 \cdot C52 + 0{,}9892 \cdot C54 = S \quad (1)$$

Note : Using 755 different milk fat samples a 99 % confidence range of S = 98,05 – 101,95 was established for pure milk fat samples with a standard deviation for all S-values SD = 0,39897.

Starting from the triglyceride composition of an unknown fat sample such a formula allows, without using a computer, to verify in a simple manner whether the sum of the triglyceride contents stated here with the corresponding factors falls outside the range of 98,05 – 101,95 and one has most probably to do with foreign fat addition.

For detecting different foreign fats Table 2 shows different triglyceride formulae. For the detection of the foreign fats soybean oil, sunflower oil, olive oil, rape-seed oil, linseed oil, wheat germ oil, maize germ oil, cotton seed oil, and hydrogenized fish oil, for the vegetable fats coconut- and palm kernel fat, as well as for palm oil and beef tallow a common formula can be used, respectively.

Since the triglyceride composition of the foreign fats is also subjected to fluctuations, up to four different, experimentally measured foreign fat triglyceride data of the same type were used. (With the same foreign fat types the least favourable limit has been considered, respectively (see Table 4)).

With the following 'Total formula' similarly good results can be obtained for all foreign fats:

$$-2{,}7575 \cdot C26 + 6{,}4077 \cdot C28 + 5{,}5437 \cdot C30 - 15{,}3247 \cdot C32 + 6{,}2600 \cdot C34 \quad (2) + 8{,}0108 \cdot C40 - 5{,}0336 \cdot C42 + 0{,}6356 \cdot C44 + 6{,}0171 \cdot C46 = S$$

Calculations for the detection of any foreign fat combination in milk fat have shown that, e.g., although with the formula for lard given in Table 2 the limit for this foreign fat in low, namely 2,7 %, other fats such as coconut fat, palm oil or palm kernel fat with detection limits of 26,8, 12,5 and 19,3 %, respectively, can, with this formula, only be detected if extremely high amounts have been added to milk fat. This applies also to other formulae in Table 2.

Table 2: Triglyceride formulae for detecting various foreign fats in milk fat, indicating the standard deviations SD for S

Formula for soybean, sunflower, olive, rape-seed, linseed, wheat germ, maize germ, cotton seed and fish oil
$2{,}0983 \cdot C30 + 0{,}7288 \cdot C34 + 0{,}6927 \cdot C36 + 0{,}6353 \cdot C38 + 3{,}7452 \cdot C40 - 1{,}2929 \cdot C42 + 1{,}3544 \cdot C44 + 1{,}7013 \cdot C46 + 2{,}5283 \cdot C50 = S;\ SD = 0{,}38157$
Formula for coconut and palm kernel fat
$3{,}7533 \cdot C32 + 1{,}1134 \cdot C36 + 1{,}3648 \cdot C38 + 2{,}1544 \cdot C42 + 0{,}4273 \cdot C44 + 0{,}5809 \cdot C46 + 1{,}1226 \cdot C48 + 1{,}0306 \cdot C50 + 0{,}9953 \cdot C52 + 1{,}2396 \cdot C54 = S;\ SD = 0{,}11323$
Formula for palm oil and beef tallow
$3{,}6644 \cdot C28 + 5{,}2297 \cdot C30 + 12{,}5073 \cdot C32 + 4{,}4285 \cdot C34 - 2{,}2010 \cdot C36 + 1{,}2791 \cdot C38 + 6{,}7433 \cdot C40 - 4{,}2714 \cdot C42 + 6{,}3739 \cdot C46 = S;\ SD = 0{,}81094$
Formula for lard
$6{,}5125 \cdot C26 + 1{,}2052 \cdot C32 + 1{,}7336 \cdot C34 + 1{,}7557 \cdot C36 + 2{,}2325 \cdot C42 + 2{,}8006 \cdot C46 + 2{,}5432 \cdot C52 + 0{,}9892 \cdot C54 = S;\ SD = 0{,}39897$

Therefore for checking an unknwon fat sample all formulae given in Table 2 and the Total formula (2) must be used, if the sample is likely to be a mixture of milk fat and one of the 14 different foreign fats or a combination of these foreign fats. If, by inserting the triglyceride of a fat sample to be analysed an S-value is obtained, which falls outside the ranges of Table 3 of only one of the five formulae, then the sample is most likely a modified milk fat. Detection of a foreign fat in milk fat by means of one of the four formulae in Table 2 does not allow conclusions to be drawn on the type of the foreign fat admixture.

Table 3: S-Limits for milk fats

Formula for detection of	S-range
Soybean, sunflower, olive, rape-seed, linseed, wheat germ, maize germ, cotton, fish oil	98,05 — 101,95
Coconut and palm kernel fat	99,42 — 100,58
Palm oil and beef tallow	95,90 — 104,10
Lard	97,96 — 102,04
Total formula	95,68 — 104,32

In Table 4 the detection limits for the different foreign fats with a 99 % confidence are given. The first column shows the minimal detection limits for the best milk fat formulae in Table 2. In the second column the detection limits for the total formula are given. although the limits are some what higher, only this formula is necessary to detect a little bit higher amounts of foreign fats. With all formulae also combinations of the different foreign fats can be detected. The ranges of variation of the triglycerides of different foreign fats of one type have no considerable influence on the detection limits.

Table 4 : 99 % limits of detection by addition of foreign fat to milk fat in %

	Individual formula	Total formula
Soybean oil	2,1	4,4
Sunflower oil	2,3	4,8
Olive oil	2,4	4,7
Coconut fat	3,5	4,3
Palm oil	4,4	4,7
Palm kernel fat	4,6	5,9
Rape-seed oil	2,0	4,4
Linseed oil	2,0	4,0
What germ oil	2,7	6,4
Maize germ oil	2,2	4,5
Cotton seed oil	3,3	4,4
Lard	2,7	4,7
Beef tallow	5,2	5,4
Hydrogenized fish oil	5,4	6,1

Note : The S-ranges are calculated in that way, that a foreign fat is only assumed, if the limits of the individual formulae are exceeded (see Table 4).

9. Quantitative foreign fat determination

In order to obtain quantitative information on the foreign fat concentration in a milk fat sample, the following formula is used

$$X\ (\%) = 100 \cdot | (100 - S) / (100 - S_F) |, \qquad (3)$$

X being the quantity of an unknown foreign fat or foreign fat mixture in an unknown milk fat. S results from addition of an unknown foreign fat by inserting the triglycerides of the foreign fat/milk fat mixture in the above total triglyceride formula. If an unknown foreign fat is added to milk fat, the mean S-value of the different foreign fats for the Total formula is chosen for S_F ; this mean S-value is obtained by inserting the triglyceride data of the pure foreign fats in this formula and by calculating a mean value (S_F = 7,46). Good quantitative results concerning any foreign fat additions are also obtained using the palm oil/beef tallow formula (Table 2) and a mean S_F-value of 10,57.

With known foreign fat types the following S_F-values must be inserted in the above formula and the respective foreign fat formula from table 2 has to be chosen :

Table 5 : S_F-values of various foreign fats

Foreign fat	S_F
Soybean oil	8,18
Sunflower oil	9,43
Olive oil	12,75
Coconut fat	118,13
Palm oil	7,55
Palm kernel oil	112,32
Rape-seed oil	3,30
Linseed oil	4,44
Wheat germ oil	27,45
Maize germ oil	9,29
Cotton seed oil	41,18
Lard	177,55
Beef tallow	17,56
Fish oil	64,12

10. **Range of application of the detection method**

The described method applies to bulk milks and is based on the representativeness of the milk fat samples.

Highly specific detection would be possible, if, for a representative number of milk fats, formulae as described above were derived for different countries.

There could be particularly suited possibilities of detection obtained, if in the different countries formulae, as have been described here, were set up of a representative number of milk fats. In this case, the use of complex computer programmes is not required, if the triglyceride combinations used in table 2 are applied and the factors redetermined by using the method of least squares.

By applying the S-ranges as shown in table 3 the formulae are, under particular feeding conditions as, for instance, underfeeding or feeding of cows with feed yeast or Ca-soaps, generally applicable. Only in the case of extreme feeding conditions (e.g. high uptake of pure feed oils, high administration of Ca-soaps comibned with feed fat etc.) the formulae partly indicate a modified milk fat.

Note: Fractionated milk fats are generally recognized as unmodified milk fat, if a modification is assumed only, when the limits are exceeded. Only with fractionated milk fats with unusual milk fat composition, as it is, e.g., the case with a hard fraction, obtained with fractionation by physical methods at high temperatures of approximately 30 °C with low yields of a few percent or with fractionation with overcritical CO_2, the formulae indicate a modification.

Milk fat fractionation may, however, be detected using other procedures e.g. Differential-Scanning-Calorimetry.

11. **Accuracy of the method**

Determined using milk fat on the basis of the formulae from table 2 and the S-ranges in table 3.

11.1. Repeatability

As difference of the S-values of two determinations carried out within the shortest feasible time interval by one operator using the same procedure and identical sample material under the same conditions (same person, same instruments/same device, same laboratory):

Table 6: Repeatability limits (r) for the different formulae

Formula for detection of	r
Soybean, sunflower, olive, rape-seed, linseed, wheat germ, maize germ, cotton, fish oil	0,67
Coconut and palm kernel fat	0,12
Palm oil and beef tallow	1,20
Lard	0,58
Total formula	1,49

11.2. Reproducibility

As difference of the S-values of two determinations carried out by operators in different laboratories, according to the same procedure using identical sample material under different conditions (different person, different instruments) at different times.

Table 7: Reproducibility limits (R) for the different formulae

Formula for detection of	R
Soybean, sunflower, olive, rape-seed, linseed, wheat germ, maize germ, cotton, fish oil	1,08
Coconut and palm kernel fat	0,40
Palm oil and beef tallow	1,81
Lard	0,60
Total formula	2,07

11.3. Critical difference

With the repeatability (r) and the reproducibility limits (R) the critical differences for all S-ranges of table 3 can be calculated (duplicate analyses). The respective values are given in table 8.

Table 8 : Critical differences for all triglyceride formulae

Formula for detection of	range
Soybean, sunflower, olive, rape-sed, linseed, wheat germ, maize germ, cotton, fish oil	97,43 — 102,57
Coconut and palm kernel fat	99,14 — 100,86
Palm oil and beef tallow	94,91 — 105,09
Lard	97,65 — 102,35
Total formula	94,58 — 105,42

11.4. Acceptability of results

All calibrated with two rounded decimals calculated triglyceride contents of C24, C26, C28 to C54 as well as cholesterol must be exactly normalized to 100.

The results of the duplicate analysis is used as a check on the repeatability. If the absolute difference between the two S-results for all five triglyceride formulae do not transgress the repeatability limits r in table 6, then the repeatability requirement is met.

For control of optimal gas chromatographic conditions and especially of the quality of the column it should be guaranteed that with 10 repetition runs the difference of the maximum and minimum S-values of all five triglyceride formulae do not transgress the range $x \cdot r$, with $x = 1{,}58$ (for 10 runs, see literature (16)), and the repeatability limits r for the different formulae in table 6.

12. Quoted standards

DIN 10 336 : 1994	Nachweis und Bestimmung von Fremdfetten in Milchfett anhand einer gaschromatographischen Triglyceridanalyse
IDF Standard 1C : 1987	Milk. Determination of Fat Content — Röse Gottlieb Gravimetric Method
IDF Standard 16C : 1987	Cream. Determination of Fat Content — Röse Gottlieb Gravimetric Method
IDF Standard 116A : 1987	Milk-Based Edible Ices and Ice Mixes. Determination of Fat Content — Röse Gottlieb Gravimetric Method
IDF Standard 22B : 1987	Skimmed Milk, Whey & Buttermilk. Determination of Fat Content — Röse Gottlieb Gravimetric Method.

13. References

1. Commission of the European Communities : *Detection of foreign fats in milk fat by means of gas chromatographic triglyceride analysis,* Doc. No VI/5202/90-EN, VI/2645/91

2. Commission of the European Communities : *Control of butter fat purity of 100 different samples of different feeding periods from 11 EEC countries;* Doc. No VI/4577/93

3. Commission of the European Communities : *Consideration of results from the first, second, third, fourth, fifth and sixth EEC collaborative trial : Determination of triglycerides in milk fat;* Doc. No VI/2644/91, VI/8.11.91, VI/1919/92, VI/3842/92, VI/5317/92, VI/4604/93

4. Timms, R. E. : *Detection and quantification of non-milk fat in mixtures of milk and non-milk fats.* Dairy Research 47 295-303 (1980)

5. Precht, D., Heine, K. : *Nachweis von modifiziertem Milchfett mit der Triglyceridanalyse. 2. Fremdfettnachweis im Milchfett mit Hilfe von Triglyceridkombinationen,* 41 406-410 (1986)

6. Luf, W., Stock, A., Brandl, E. : *Zum Nachweis von Fremdfett in Milchfett über die Trigylceridanalyse.* Österr. Milchwirtsch. Wissensçh. Beilage 5, 42 29-35 (1987)

7. Precht, D.: *Bestimmung von pflanzlichen Fetten oder tierischen Depotfetten in Milchfett.* Kieler Milchwirtsch. Forschungsber. 42 143-157 (1989)

8. Precht, D.: *Schnelle Extraktion von Milchfett,* Kieler Milchwirtsch. Forschungsber. 42 119-128 (1990)

9. Precht, D.: *Schnelle gaschromatographische Triglyceridanalyse von Milchfett.* Kieler Milchwirtsch. Forschungsber. 42 139-154 (1900)

10. Precht, D.: *Control of milk fat purity by gas chromatographic triglyceride analysis.* Kieler Milchwirtsch. Forschungsber. 43 (3) 219-242 (1991)

11. Precht, D.: *Detection of adulterated milk fat by fatty acid and triglyceride analysis.* Fat Sci. Technol. 93 538-544 (1991)

12. Precht, D.: *Detection for foreign fat in milk fat. I. Qualitative detection by triacylglycerol formulae. II. Quantitative evaluation of foreign fat mixtures.* Z. Lebensm. Unters. Forsch. 194 1-8, 107-114 (1992)

13. Precht, D.: *Gas chromatography of triacylglycerols and other lipids on packed columns* in CRC Handbook of Chromatography: Analysis of lipids, p. 123-138, Ed. K.D. Mukherjee, N. Weber, J. Sherma, CRC Press, Boca Raton (1993)

14. Precht, D., Molkentin, J.: *Quantitative triglyceride analysis using short capillary columns,* Chrompack News 4 16-17 (1993)

15. Molkentin, J., Precht, D.: *Comparison of packed and capillary columns for quantitative gas chromatography of triglycerides in milk fat.* Chromatographia 39 (5/6) 265-270 (1994)

16. Stange, K.: *Angewandte Statistik, Erster Teil, Eindimensionale Probleme,* Springer-Verlag, Berlin, P. 378 (1970)

ANNEX IV

SENSORY EVALUATION OF BUTTER

1. Scope

The purpose of this procedure for sensory evaluation of butter is to provide a uniform method applicable in all Member States.

2. Definitions

Sensory evaluation (assessment) means the examination of the attributes of a product by the sense organs.

Panel means a group of selected assessors working, during the assessment, without intercommunication, and without influencing one another.

Scoring means sensory evaluation by a panel, using a numerical scale. A nomenclature of defects must be used.

Grading means a quality classification which is performed on the basis of scoring.

Control documents: documents used to record the individual scores for each attribute and the final grade of the product. (This document may also be used to record chemical composition.)

3. Test room

3.1. Precautions must be taken in order that the assessors in the test room are not influenced by external factors.

3.2. The test room must be free from foreign odours and easy to clean. The walls must be of a light colour.

3.3. The test room and its lighting must be such that the properties of the products to be scored are not affected. The room must be equipped with appropriate temperature control.

4. Selection of assessors

The assessor must be familiar with butter products and be competent to carry out sensory grading. His competence should be assessed on a regular basis (at least once a year) by the competent authority.

5. Requirements for the panel

The number of assessors in the panel should be uneven, the minimum number being three. The majority must be employees of the competent authority or authorized persons not employed by the dairy industry.

6. Assessment of the value of each attribute

6.1. The sensory evaluation is to be carried out in relation to the following three attributes; appearance, consistency and flavour.

Appearance involves the following features: colour, visible purity, mould growth and water dispersion. Water dispersion is tested according to IDF-Standard 112A/1989.

Consistency involves the following features: firmness and spreadability.

Physcial methods may be applied for the evaluation of butter consistency. The Commission envisages the future harmonization of these methods.

Flavour involves the following features: taste and odour.

A significant deviation from the recommended temperature prevents a reliable evaluation of consistency and flavour. The temperature is of paramount importance.

6.2. Each attribute has to be sensory evaluated separately. The scoring has to be done according to table 1.

6.3. It may be desirable for the assessors to score together, before starting the assessment, one or more reference samples for appearance, consistency and flavour, in order to achieve uniformity.

6.4. Scoring for acceptance is as folllows :

	Maximum	Required
Appearance	5	4
Consistency	5	4
Flavour	5	4

Where the required score is not obtained, a description of the defect has to be given. The score given by each assessor for each attribute must be recorded in the control document. The product is accepted or rejected on the basis of a majority decision. Cases where differences between the individual scoring for each attribute are wider than adjacent points should not occur frequently (not more than once per 20 samples). Otherwise the competence of the panel should be checked by the panel leader.

7. Supervision

A panel leader who must be an official employee of the competent authority and may be a member of the panel must be generally responsible for the entire procedure. He must record the individual scores for each attribute in the control document and certify whether the product is accepted or rejected.

8. Sampling and preparation of the sample

8.1. — It is desirable that the identity of the samples are concealed during the assessment so that any possible bias is avoided.

— This should be organized by the panel leader prior to the evaluation without the presence of the other panel members.

8.2. When the sensory evaluation is carried out at the cold store, the sample is taken using a butter trier. If the sensory evaluation is carried out at another location other than the cold store, then at least a 500 g sample should be taken.

8.3. During the evaluation, the butter should have the temperature 10 to 12 °C. Large deviations should be avoided at all cost.

9. Nomenclature

Refer to the appended table 2.

Table 1 : Butter scoring

Appearance			Consistency			Flavour + aroma		
Points	No (1)	Remarks	Points (quality class)	No (1)	Remarks	Points (quality class)	No (1)	Remarks
5		*Very good* ideal type highest quality (equal dry)	5		*Very good* ideal type highest quality (well spreadable)	5		*Very good* ideal type highest quality (absolutely pure finest aroma)
4		*Good* (2) No evident defects	4		*Good* (2)	4		*Good* (2) No evident defects
				17	hard			
				18	soft			
3		*Fair (slight defects)*	3		*Fair (slight defects)*	3		*Fair (slight defects)*
	1	loose (free), moisture		14	short, brittle, crumbly		21	unclear
	2	not uniform, two coloured		15	pasty, doughy, greasy		22	foreign flavour
				16	sticky		25	acid
	3	streaky		17	hard		27	cooked flavour, scorched flavour
	4	mottled, marbled		18	soft			
	5	speckled					33	feed flavour
	6	oil separation					34	coarse, bitter
	7	overcoloured					35	oversalted
	8	weak, open texture						
2		*Poor (evident defects)*	2		*Poor (evident defects)*	2		*Poor (evident defects)*
	1	loose (free) moisture		14	short, brittle, crumbly		21	unclean
	3	streaky		15	pasty, doughy, greasy		22	foreign flavour
	4	mottled, marbled		16	sticky		23	stale
	5	speckled		17	hard		25	acid
	6	oil separation		18	soft		32	oxidized flavour, metallic flavour
	10	foreign matter						
	11	mouldy					33	feed flavour
	12	undissolved salt					34	coarse, bitter
							35	oversalted
							36	musty-flat, putrid
							38	chemical flavour
1		*Very poor (strong defects)*	1		*Very poor (strong defects)*	1		*Very poor (strong defects)*
	1	loose (free) moisture		14	short, brittle, crumbly		22	foreign flavour
	3	streaky		15	pasty, doughy, greasy		24	cheesy, lactic cheese flavour
	4	mottled, marbled,		16	sticky			
	5	speckled		17	hard		25	acid
	6	oil separation		18	soft		26	yeasty
	7	overcoloured					28	mouldy flavour
	9	granular					29	rancid
	10	foreign matter					30	oily, fishy
	11	mouldy					31	tallowy
	12	undissolved salt					32	oxidized flavour, metallic flavour
							34	coarse, bitter
							36	musty-flat, putrid
							37	malty
							38	chemical flavour

(1) Table 2.

(2) The defects mentioned under 'good' are only very small deviations from the ideal type.

Table 2 : Table of butter defects

I. *Appearance*

1. loose (free), moisture
2. not uniform, two coloured
3. streaky
4. mottled, marbled
5. speckled
6. oil separation
7. overcoloured
8. weak (open texture)
9. granular
10. foreign matter
11. mouldy
12. undissolved salt

II. *Consistency*

14. short, brittle, crumbly
15. pasty, doughy, greasy
16. sticky
17. hard
18. soft

III. *Flavour & Aroma*

20. without flavour
21. unclean (1)
22. foreign flavour
23. stale
24. cheesy, lactic cheese flavour
25. acid
26. yeasty
27. a) cooked flavour
    b) scorched flavour
28. mouldy flavour
29. rancid
30. oily, fishy
31. tallowy
32. a) oxidized flavour
    b) metallic flavour
33. feed flavour
34. coarse, bitter
35. oversalted
36. musty-flat, putrid
37. malty
38. chemical flavour

(1) This designation should be used as seldom as possible and only when the defect cannot be described more accurately.

ANNEX V

SAMPLING FOR CHEMICAL AND MICROBIOLOGICAL ANALYSIS AND SENSORY EVALUATION

1. Chemical and microbiological analysis

Quantity of butter (kgs)	Minimum number of samples (> 100 g)
≤ 1 000	2
> 1 000 ≤ 5 000	3
> 5 000 ≤ 10 000	5
> 10 000 ≤ 15 000	5
> 15 000 ≤ 20 000	6
> 20 000 ≤ 25 000	7
> 25 000	7 + 1 per 25 000 kg or part thereof

Sampling for microbiological analysis must be carried out aseptically.

Up to five samples at 100 g may be combined to one sample, which is analysed after thorough mixing.

Samples should be taken randomyl from different parts of the offered quantity and tested before or at the time of entry into the cold store designated by the intervention agency.

Preparation of composite butter sample (Chemical analysis)

(a) Using clean dry butter trier or similar suitable instrument, extract a core of butter of at least 30 g and place in a sample container. The composite sample may then be sealed and forwarded to the laboratory for analysis.

(b) At the laboratory the composite sample is warmed in the original unopened container at 30 °C until by frequent shaking a homogeneous fluid emulsion, free of unsoftened pieces is obtained. The container should be one-half to two-thirds full.

For each manufacturer offering butter for intervention, two samples per year have to be analysed for non-milk fat and one sample for tracers.

2. Sensory evaluation

Quantity of butter (kgs)	Minimum number of samples to be taken
1 000 ≤ 5 000	2
> 5 000 ≤ 25 000	3
> 25 000	3 + 1 per 25 000 kgs or part thereof

Samples should be taken randomly from different parts of the offered quantity between the 30th and 35th day following the take-over of the butter and graded.

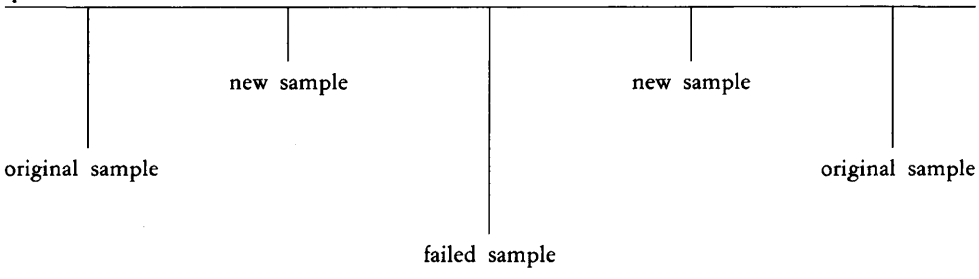
Each sample should be assessed individually in accordance with Annex IV. No resampling and re-evaluation is allowed.

3. **Guidelines to be followed in the event of sample failure**

(a) Chemical and microbiological analysis :

— where individual samples are analysed one sample with one single defect per five to 10 samples or two samples with one single defect each per 11 to 15 samples, respectively, may be permitted. In the event of a sample failure, two new samples must be taken in either side of the failed sample and checked for the failed parameter. If both samples do not meet the specification the quantity of butter between the two original samples on either side of the failed sample must be rejected from the quantity offered,

quantity to be rejected in the event of a new sample failure



— where composite samples are analysed, in the event of a composite sample failing for one parameter, the quantity represented by that composite sample is rejected from the quantity offered.

(b) Sensory evaluation :

— where a sample fails the sensory evaluation the quantity of butter between the two adjacent samples on either side of the failed sample is rejected from the quantity offered.

(c) In the event of a chemical or microbiological failure and sensory failure the total quantity is rejected.