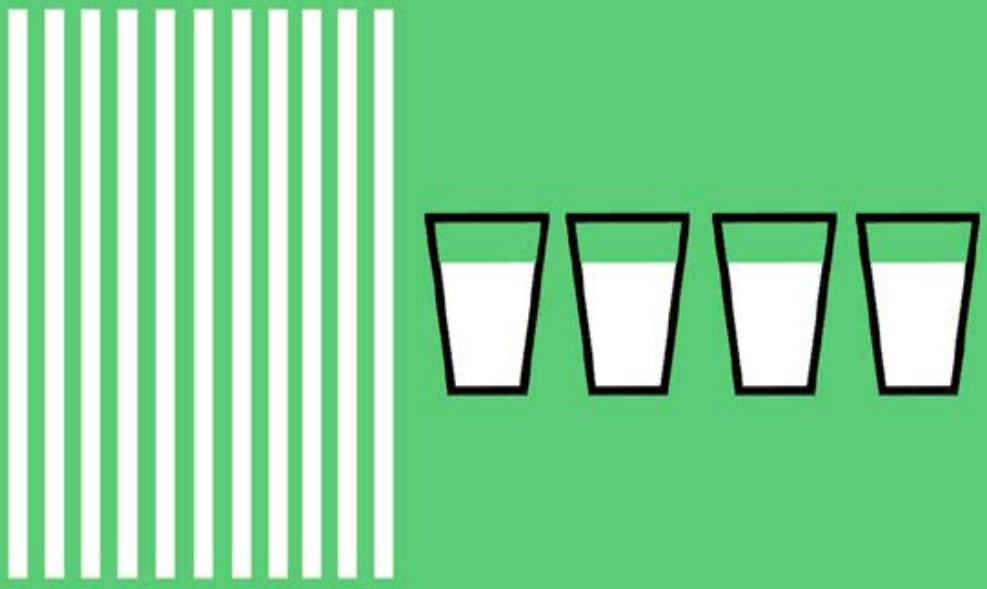
Joint FAO/WHO Food Standards Programme

JOINT FAO/WHO COMMITTEE OF GOVERNMENT EXPERTS ON THE CODE OF PRINCIPLES CONCERNING MILK AND MILK PRODUCTS

Report of the Nineteenth Session

Held in Rome, Italy, 12-17 June 1978



FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS
WORLD HEALTH ORGANIZATION
Rome

The following reports of earlier sessions in this series have been issued:

First session, Rome, Italy, 8-12 September 1958
(Meeting Report No. 1958/15)

Second session, Rome, Italy, 13-17 April 1959
(Meeting Report No. 1959/AN-2)

Third session, Rome, Italy, 22-26 February 1960
(Meeting Report No. AN 1960/2)

Fourth session, Rome, Italy, 6-10 March 1961
(Meeting Report No. AN 1961/3)

Fifth session, Rome, Italy, 2-6 April 1962
(Meeting Report No. AN 1962/3)

Sixth session, Rome, Italy, 17-21 June 1963
(Meeting Report No. AN 1963/5)

Seventh session, Rome, Italy, 4-8 May 1964
(Meeting Report No. AN 1964/4)

Eighth session, Rome, Italy, 24-29 May 1965
(Meeting Report No. AN 1965/3)

Ninth session, Rome, Italy, 20-25 June 1966
(SP-10/105 - 9th)

Tenth session, Rome, Italy, 25-31 August 1967
(SP-10/105 - 10th)

Eleventh session, Rome, Italy, 10-15 June 1968
(Cx 5/70 - 11th)

Twelfth session, Rome, Italy, 7-12 July 1969
(Cx 5/70 - 12th)

Thirteenth session, Rome, Italy, 15-20 June 1970
(Cx 5/70 - 13th)

Fourteenth session, Rome, Italy, 6-11 September 1971
(Cx 5/70 - 14th)

Fifteenth session, Rome, Italy, 25-30 September 1972
(Cx 5/70 - 15th)

Sixteenth session, Rome, Italy, 10-15 September 1973
(Cx 5/70 - 16th)

Seventeenth session, Rome, Italy, 14-19 April 1975
(Cx 5/70 - 17th)

Eighteenth session, Rome, Italy, 13-18 September 1976
(Cx 5/70 - 18th)

CODE OF PRINCIPLES CONCERNING MILK AND MILK PRODUCTS:

First Edition	1960
Second Edition	1961

Third Edition	1962
Fourth Edition	1963
Fifth Edition	1966
Sixth Edition	1968
Seventh Edition	1973

Issued by the Secretariat of the
Joint FAO/WHO Food Standards Programme, FAO, Rome

Ref. No. Cx 5/70, 19th Session, October 1978

CX 5/70 - 19th Session

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FOOD AMD AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

First printing 1978
Second printing 1979

M-83

ISBN 92-5-100661-X

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SUMMARY OF POINTS FOR ACTION BY GOVERNMENTS

1. Governments are requested to make their comments available by 31 October 1979 at the latest. All communications should be sent, if possible, in duplicate and addressed to the Technical Secretary, Committee on the Code of Principles concerning Milk and Milk Products, Animal Production and Health Division, FAO, Rome.

2. Governments may send observations regarding any matter they would wish to raise.

   Those specific points on which the Committee agreed that comments should be sought are the following:

Redraft of the:	- Submitted to governments for acceptance
- Recommended General Standard for Cheese, A-6	(see paras 13 to 41 of this Report and Appendix II)
- Recommended General Standard A-8(a) for Named Variety Process(ed) Cheese and Spreadable Process(ed) Cheese	(see paras 53 to 75 of this Report and Appendices III-A, III-B and III-C)
- Recommended General Standard A-8(b) for "Process(ed) Cheese" and "Spreadable Process(ed) Cheese"	" " "
- Recommended General Standard A-8(c) for Processed Cheese Preparations (Process(ed) Cheese Food and Process(ed) Cheese Spread)	" " "
at Step 7 of the Committee's Procedure for the Elaboration of Milk and Milk Product Standards	
When considering acceptance of compositional standards A-1 to A-7, A-9, A-10, A-11(a) and A-11(b). Governments should bear in mind Decision No. 5 (see 7th Edition of the Code of Principles and paras. 65 to 70 of the Report of the 17th Session).	
- Compositional Standards A-1 to A-5 and A-7, redrafts at Step 7 of the above Procedure	- Governments to continue to submit their acceptance or confirm their acceptances. (See 7th Edition of the Code of Principles).
- Compositional Standard A-10 for Cream Powder at Step 7 of the above Procedure	- Governments to continue to submit their acceptances. (See 7th Edition of the Code of Principles).
- Compositional Standard A-11(a) for Yoghurt and Sweetened Yoghurt at Step 7 of the above Procedure	- Governments to continue to submit their acceptances. (See Report of the 17th Session, Appendix VII).
- Compositional Standard A-11(b) for Flavoured Yoghurt at Step 7 of the above Procedure	- Governments to continue to submit their acceptances. (See Report of the 18th Session, Appendix III).
- Compositional Standard A-9 for Cream at Step 7 of the above Procedure	- Governments to continue to submit their acceptances. (See Report of the 18th

	Session, Appendix IV).
- Compositional Standard A-12 for Edible Acid Casein at Step 7 of the above Procedure	- Governments to continue to submit their acceptances. (See Report of the 18th Session, Appendix V).
- Compositional Standard A-13 for Edible Caseinate at Step 7 of the above Procedure	- Governments to continue to submit their acceptances, (See Report of the 18th Session, Appendix VI).
<u>International Individual Cheese Standards</u>	
- C-1 to C-25 and C-26 to C-34 at Step 7 of the Procedure for the Elaboration of International Individual Cheese Standards	- Governments to continue to submit their acceptances. (See CAC/C1-C25 (1972) Recommended International Standards for Cheeses and Government Acceptances, Appendices VII-A to VII-E to the Report of the 15th Session and Appendices V-A to V-D to the Report of the 16th Session. See also para 111 of the Report of the 17th Session and paras 25 to 35 of the Report of the 18th Session).
- C-35 Extra Hard Orating Cheese	- Submitted to Governments for acceptance (see paras 42 to 32 of this Report and Appendix IV).
<u>Standard Methods of Analysis</u>	
- B-1 to B-8 and B-10 to B-15	- Governments to continue to submit their acceptances. (See 7th Edition of the Code of Principles).
- Milk Fat, Detection of Vegetable Fat by the Phytosteryl Test, Standard Method B-16 - Milk Fat, Detection of Vegetable Fat by Gas-liquid Chromatography of Sterols, Standard Method B-17 - Cheese, Determination of Chloride Content, Standard Method B-18	- Governments to continue to submit their acceptances. (See Appendices X, XI and XII respectively).
- Cheese - Determination of Nitrate and Nitrite Contents - Anhydrous milk fat - Peroxide Value - Butter - Water, Solids-non-fat and Fat on the same test portion	- Submitted to governments for acceptance (See Appendix IX-J, IX-J, IX-K of this Report).
- Caseins and caseinates - Determination of Water Content - Rennet caseins and caseinates - Determination of Ash - Caseins - Determination of "fixed ash"	- Revised texts submitted to the Committee for approval at its next session. (See Appendix IX-B to IX-H of this Report).

- Caseins and caseinates - Determination of protein content - Caseins - Determination of free acidity - Milk and Milk Products -Determination of Lactose in the presence of other reducing substances - Dried milk - Determination of titratable acidity	
- Caseins and caseinates - Lactose	- Governments to comment (See Appendix IX-A).
- Imitation milk products	- Governments to comment on the proposed Decision No.6 (See para 116 of this Report and Appendix V)
- Pasteurization, Sterilization, UHT-Processes	- Governments to submit information on national legislation on definitions for pasteurization, sterilization and UHT-processes. (See para 122 of this Report).

CX 5/70 - 19th Session
June 1978

RETORT OF THE

NINETEENTH SESSION OF THE JOINT FAO/WHO COMMITTEE OF GOVERNMENT EXPERTS ON THE CODE OF PRINCIPLES CONCERNING MILK AND MILK PRODUCTS

Rome. 12 - 17 June 1978

INTRODUCTION

1. The Nineteenth Session of the Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products was held at FAO Headquarters in Rome, from 12-17 June 1978. The session was attended by 107 participants including representatives and observers from 39 countries, and observers from 6 organizations (see Appendix I for the List of Participants).

2. The Committee was presided over by its Chairman, Mr. T.L. Hall (New Zealand) and its two Vice-Chairmen, Mr. K.P. Andersen (Denmark) and Dr. A. Farkhondeh (Iran). The Joint Secretaries were Dr. F. Winkelmann (FAO), Mr. W.L. de Haas (Joint FAO/WHO Food Standards Programme), and Dr. L. Reinius (WHO).

3. The Nineteenth Session of the Committed was convened by the Directors-General of FAO and WHO. The meeting was opened by Mr. G.O. Kermode, Officer-in-charge, Food Policy and Nutrition Division, who reviewed the programme of work of the Committee, the progress being made by the Codex Alimentarius Commission on standards and their acceptance by governments, the International Scheme for the Coordination of Dairy Development (ISCDD) and the activities of the FAO dairy training programme. Mr. Kermode mentioned in particular that the Commission at its 12th Session, had reviewed the direction of its work, especially in regard to Codex Commodity Committees, including the Committee of Government Experts on the Code of Principles. The Commission's discussion concerning the work of that Committee was reflected in document MDS 78/3(c) which contained an excerpt of the Commission's Report. Mr. Kermode stressed that it had been gratifying to note that at the Commission many delegates had expressed their appreciation for the excellent work done by the Committee, which had been the first to demonstrate the feasibility of reaching international agreement on food standards and their acceptance by governments. However, most delegations had held the view that the Committee had completed the major part of its work. The Commission had considered that the Committee should not start work on new topics but in one or two sessions bring to an end its current work on important matters. The Committee would then adjourn sine die but could be reactivated when this was found to be necessary.

4. In his introductory statement the Chairman of the Committee expressed his pride in the Committee's achievements which had led the Commission to believe that the Committee's main tasks had been almost completed. Dr. Hall specifically thanked Dr. E. Green (United Kingdom) and his working group on the redraft of the General Standard for Cheese, for the excellent work they had done between the Committee's last and present session which was essential for the finalization of the Standard during this session.

Election of Chairman and Vice-Chairmen for the 20th Session

5. The Committee unanimously elected Mr. K.P. Andersen (Denmark) Chairman of the Committee, to serve from the end of the 19th Session until the end of the 20th Session. The Committee also unanimously elected Dr. A. Farkhondeh (Iran) and Dr. J.M. Ng'ang'a (Kenya) to be first and second Vice-Chairmen, respectively, both to serve from the end of the 19th Session until the end of the 20th Session. The Committee expressed its appreciation of the outgoing Chairman of the Committee and of the two Vice-Chairmen.

Adoption of Agenda

6. Following a suggestion by the Chairman the provisional agenda was adopted with some rearrangement in the order of items to be discussed so as to deal with the most important items first.

Acceptance of the Code of Principles and Associated Standards

7. The Committee was informed of the latest position regarding government acceptances of the Code of Principles, Associated Standards and Methods of Analysis and Sampling. This was as follows:

8. Code of Principles

	Number of Acceptances
Group I	33
Group II	4
Group III	35

9.

Redraft of Standard		Accepted by *
A-1 for Butter	-	12 countries: Belgium*, Bulgaria*, Canada*, Finland, France*, F.R.of Germany*, Iran, Kenya, Netherlands*, New Zealand*, Norway*, Poland*.
A-2 for Butteroil	-	9 countries: Bulgaria*, Canada, Denmark*, Prance, Hungary, Netherlands*, New Zealand, Norway*, Poland*.
A-3 for Evaporated Milk	--	10 countries: Canada*, Denmark, Finland, F.R. of Germany*, Hungary, Iran, Kenya, Netherlands*, Poland*, Switzerland*.
A-4 for Sweetened Condensed Milk	-	12 countries: Belgium*, Bulgaria*, Canada*, Finland*, F.R, of Germany*, Hungary, Iran, Kenya, Netherlands*, New Zealand*, Poland*, Switzerland*
A-5 for Milk Powder	-	9 countries: Bulgaria*, Denmark, F.R. of Germany*, Iran, Kenya, Netherlands, New Zealand*, Poland*, Switzerland.*
A-7 for Whey Cheese	-	10 countries: Bulgaria*, Canada*, Denmark, Finland, F.R, of Germany*, Hungary, Iran, Netherlands*, Norway, Poland*.
New Standards	-	

A-9 for Cream	- 2 countries: Poland*, Philippines
A-10 for Cream Powder	- 5 countries: Bulgaria*, France*, Hungary, Iran, New Zealand*.
A-11(a) for Yoghurt and Sweetened Yoghurt	- 4 countries: Argentina*, France*, Iran, Poland
A-11(b) for Flavoured Yoghurt	- 1 country: the Philippines
A-12 for Edible Acid Casein	- 1 country: New Zealand
A-13 for Edible Caseinate	- 1 country: New Zealand

* "country" means acceptance with reservations of various kinds» Details of acceptances and remarks by governments will be published in the 8th Edition of the Code of Principles concerning Milk and Milk Products. The Government of Malawi intends to accept the standards contained in the 7th edition of the Code of Principles after a period of five years (target acceptance).

10.

	Methods of Sampling and Analysis	Number of Acceptances
B-1	Sampling Methods for Milk and Milk Products	49
B-2	Determination of the Fat Content of Dried Milk	48
B-3	Determination of the Fat Content of Cheese and Processed Cheese Products	47
B-4	Determination of the Acid Value of Fat from Butter	46
B-5	Determination of the Refractive Index of Fat from Butter	47
B-6	Determination of the Fat Content of Milk	18
B-7	Determination of the Fat Content of Evaporated Milks and of Sweetened Condensed Milks	28
B-8	Determination of the Salt (Sodium Chloride) Content of Butter	19
B-10	Determination of the Fat Content of Whey Cheese	8
B-11	Determination of the Dry Matter Content in Whey Cheese	12
B-12	Determination of the Phosphorus Content of Cheese and Processed Cheese Products	12
B-13	Determination of the Citric Acid Content of Cheese and Processed Cheese Products	12
B-14	Polarimetric Determination of the Sucrose Content of Sweetened Condensed Milk	12
B-15	Determination of the Fat Content of Cream	8
B-16	Milk Fat, Detection of Vegetable Fat by the Phytosteryl Method	12
B-17	Milk Fat, Detection of Vegetable Fat by Gas-liquid Chromatography of Sterols	2
B-18	Determination of Chloride Content	2

11. DETAILS OF ACCEPTANCE OF
INTERNATIONAL INDIVIDUAL CHEESE STANDARDS

Cheese Variety		Belgium	Bulgaria	Brazil	Canada	Denmark	Finland	France	F. R. of Germany	Hungary	Iran	Ireland	Kenya	Malta	Netherlands	New zealand	Norway	Philippines	Poland	Spain	Sweden	Switzerland	Trinidad & Tobago	U.K.	U.S.A.	Number of Acceptances
C-1	Cheddar		X	X	X	X	X	X	X		0	0			X	0	X		0	0	0	X	(**)	0	X	19
C-2	Danablu				X	0		X	X	0	0	0			X	X	0		X	0		X	(**)	0		15
C-3	Danbo				X	0	X	X	X	0	0	0			X	X	0		X	X		X	(**)	X		16
C-4	Edam	0	X	X	X	0	X	X			0	0			0		0		X	0		X	(**)	0	X	17
C-5	Gouda	0	X	X	X	X	X	X			0	0			0				X	0		X	(**)	0	X	16
C-6	Havarti				X	0	X	X		0	0	0				X	0		X	X		X	X	X		14
C-7	Samsoe				X	0	X	X	X	0	0	0			X	X	0		X	X		X	X	X		16
C-8	Cheshire	0			X	X	X	X	X	0	0			0	X		0		X	0	0	X	(**)	0	X	18
C-9	Emmentaler		X	X	X	X	X	X		0	0			0	X		X		X	0		X	(**)	X	X	17
C-10	Gruyère			X	X	0	X	0		0	0			0	X		0		0	0		X	(**)	0	X	16
C-11	Tilsiter			X		0	X	X	X	0	0				X		X		X			X	X	X		13
C-12	Limburger	X		X		0	X	X	X	0	0				X		X		X			X	X	X	X	14
C-13	Saint-Paul in						X	X		0	0								X		0	X	X			9
C-14	Svecia	X				0	X	X	X	0	0				X		0		X		0	X	X	X		14
C-15	Provolone	X		X		X	X	X			0				X				X			X	(**)	X	X	12
C-16	Cottage Cheese incl. Creamed Cottage Cheese	X					X	X		0	0								X			X	(**)		X	9
C-17	Butterkäse	0					X	X	X	0	0				X		X		X	X			X)	X	X	13
C-18	Coulommiers						X	0		0	0				X		0		X	X		X	X)	X	0	11
C-19	Gudbrandsdalsost (whey cheese)					0	X	X		0	0				X		0		X	0	0	X	X)			11
C-20	Harzer Käse					X	X	X	0	0	0				X		X		X	0			X)	X		12
C-21	Herrgärdsost					0	X	X		0	0				X		0		X	0	0		X)	X		12
C-22	Huahällsost					0	X	X		0	0				X		0		X	0	0		X)	X		12
C-23	Norvegia					0	X	X		0	0				X		0		X	0	0		X)	X		13
C-24	Maribo	X				0		X		0	0		0				X			X						8
C-25	Fynbo	X				0		X		0	0		0				X			X						8
C-26	Esrom					0		X	X	0	0				0		X	0	X	X						10
C-27	Romadur					0	0	0	0	0	0				0		X	0	X	X						11
C-28	Amsterdam					0		X	X	0	0				0		X	0	X	X						10
C-29	Leidse					0		X	X	0	0				c											10

C-30	Friese					0		X	X	0	0				0		X	0	X	0						10
C-31	Cream Cheese					X	X				0															4
C-32	Blue-Veined						X	X		0	0															5
C-33	Camembert						0	X		0	0				X				X							6
C-34	Brie						0	0		0	0															6

o = acceptance x = acceptance with certain reservations

(**) = 'target acceptance' according to the Codex
x) = any cheese meeting the standard concerned could be freely distributed In Trinidad and Tobago

12. The Committee noted that the former versions of the compositional standards A1 to A7 had been accepted by 45 to 64 countries and supported the request made by the Secretariat that governments accept or confirm acceptance of the redrafted standards. Following a suggestion of the delegation of Denmark the countries which had accepted the former versions of the standards are listed in Appendix VIII to this Report.

REDRAFT OF GENERAL STANDARD FOR CHEESE - A-6

13. The Committee considered at Step 6 of the Procedure the General Standard for Cheese (A-6). The Committee was reminded that lack of time had prevented it from reaching a conclusion at its 18th Session on the revision of the Standard and that it had requested a Working Group to prepare a redraft of the Standard.

14. The Group was convened during the annual session of IDF in Quebec (Canada) in October 1976. Following a round of comments within the Group the Standard had been further revised and was subsequently issued supplemented with notes on those issues for which no common solution had been found (document MDS "78/4); government comments on the revised Standard were contained in MDS 78/4-Comments and Conference Room Document LIM.1.

15. The Chairman, speaking on behalf of the Committee, thanked the members of the Working Group and in particular expressed his appreciation to Dr. E. Green, who had acted as coordinator, for the comprehensive work done. It was noted that the revised standard provided for cheese made by traditional methods as well as cheese produced by new alternative technological processes.

Scope (1)

16. There was some discussion on whether or not it was desirable to specify in the Scope that whey cheeses were not covered by the Standard. Some delegations held the view that as whey cheeses were dealt with in a separate standard (A-7) reference to these products in the present standard was not necessary or even justified. Other delegations were of the opinion that specific exclusion of whey cheese from the products covered by the standard was warranted. The Committee decided to retain the sentence "The standard does not apply to whey cheeses" in the Scope and deleted the square brackets.

Definition (2)

17. The question was raised whether in part (a) of the definition of cheese covering the product manufactured according to traditional methods, provision should also be made for suitable coagulating agents other than rennet, such as lactic acid. The Committee agreed that it was desirable to establish clearly that all appropriate coagulating agents could be used; the square brackets were deleted. It was not

considered necessary to state, as was suggested by one delegation, that the coagulating agents should be "harmless" as it was understood that all ingredients used in the manufacture of cheese must be safe.

Additions (3)

18. The Committee decided to use as an introductory phrase to the section, the provision contained in 3.2.1.

Flavouring Substances (3.1)

19. It was agreed to retain the first sentence and to remove the square brackets, and to delete the final clause of the provision, which was considered to be a labelling requirement

Other Additions (3.2)

20. The Committee agreed to revise the text so that 3.2.2(a) and 3.2.2(b) should be re-placed with the single paragraph on page 3 of MDS 78/4 as proposed by the Working Group. This specified that for cheeses not covered by international individual or group standards there should be a technological necessity for the additions used; and that they should be those permitted for a similar type of cheese or one near in character for which there was an international individual or group standard. The Committee held the view that the statement provided by implication for maximum levels of additions not exceeding those established for standardized cheeses.

Labelling (4)

21. The Committee was informed that the labelling provisions of the international individual cheese standards or group standards elaborated so far did not deviate from the General Standard for the Labelling of Prepackaged Foods. In view of this, it was agreed to delete in the introduction to the section the clause reading: "except where an international individual cheese standard or group standards provides otherwise".

22. It was pointed out that with regard to labelling the requirements for cheese in bulk differed from those for cheese offered for retail sale. The Committee agreed to deal with this question in a separate provision following the discussion of the other labelling provisions (see para 40 of this Report).

Name of the Food (4.1)

23. The Committee discussed at length the provision covering the designation of cheese (4.1.2). It was thought that in a number of instances it would not be necessary, as far as informing the consumer was concerned, to state on the label, in addition to the variety or fanciful name, that the product in question was "cheese".

24. The Committee finally agreed that for cheeses covered by an international individual or group standards or cheeses defined in national legislation, only the specified designation might be required. However, in countries where cheese as such or certain varieties of cheese were not generally known the label could also carry the designation "cheese".

25. The Committee agreed that the form of declaration of the fat content in cheese (4.1.3 (b)) could be either the minimum fat in dry matter or the fat content expressed as a percentage by mass, or could be both} the word "or" was replaced by "and/or". As a consequence the text in square brackets was deleted.

26. When discussing the designation of cheeses made of milk other than cow's milk (4.1.4) it was pointed out that some of these were so well known internationally that specification of the origin of the milk was not, always necessary in importing countries. The Committee decided to broaden, beyond the home market, the provision that the declaration of the animal species would not be required provided the omission would not mislead the consumer.

27. It was pointed out that where milk from more than one species of animal was used, it was desirable to declare the origin of the milk in descending order of proportion. The Committee agreed to amend the provision (4.1.4) accordingly. The Committee also considered a proposal to require on the label an indication of the percentage of the various milks used as it was thought that the mandatory declaration of a certain milk present in small quantities could only be misleading. A further proposal to specify that a minimum quantity of milk of a particular species should be present in the blend in order to qualify for declaration at all, was also discussed. The Committee did not amend the provision further.

Country of Manufacture (old 4.3 - new 4.5)

28. The Committee briefly considered a proposal of one delegation that cheese designated with the name of a variety, but not manufactured in the country of origin of the variety, could be sold in the country of manufacture provided there was an indication of the name of a well recognized geographical area in the country of manufacture. The Committee did not change the provision requiring declaration of the country of manufacture.

Ingredients (old 4.4 - new 4.2)

29. The Committee discussed the alternative texts for the declaration of ingredients contained in the present document. It was noted that the first text was in compliance with the General Standard for the Labelling of Prepackaged Foods which required that a complete list be declared on the label.

30. It was further noted that the Labelling Standards allowed exemptions only in certain specific cases. The second text excluded milk, starter cultures and coagulating agents from being declared on the label as ingredients. The Secretariat informed the Committee that a departure from the Labelling Standard would require a justification for consideration by the Codex Committee on Food Labelling.

31. In the view of many delegations the justification for using the second text was that cheese was a product in respect of which the provisions of paragraph 3.2(a)(iii) of the General Standard for the Labelling of Prepackaged Foods would apply, i.e. "the food is of a well-known composition and the absence of a full list of ingredients is not prejudicial to the consumer". These delegations were further of the opinion that the other information provided on the label would enable the consumer to understand the nature of the food.

32. The delegations also considered that, in particular, it was not necessary to list those Substances which were not "present in the final product" (i.e. which were not ingredients as defined in the General Standard for the Labelling of Prepackaged Foods). Cheese was a product derived from the action of starter cultures and other substances on milk ingredients. These raw materials or part of them would be present in a modified form in the end product whereas other constituents were removed during processing. The delegations thought that it would not be misleading to the consumer for the resulting food to be covered by a general term "cheese".

33. Following an extended discussion the Committee decided to adopt a revised version of the second text which exempted from ingredient listing: (i) ingredients obtained from milk; (ii) starter cultures, and (iii) rennet and other coagulating enzymes. The delegations of Canada, United Kingdom and USA did not fully accept that there was sufficient justification presented for limiting the ingredient listing (paragraphs 30, 31., 32). The delegation of the USA reserved its position concerning the provision (new 4.2) as amended.

Lot Identification (4.6)

34. Some delegations expressed the opinion that it was not feasible to mark cheese which had been cut in pieces or slices and was offered for retail sale in prepackaged form with a lot or batch-number which would give within factory identification. The Committee did not amend the provision.

Bate Marking (new provision 4.8)

35. The Committee considered the feasibility of a general provision for date marking in the Standard. A distinction was made between prepacked and non-packaged cheese, whole cheese and the cut or sliced product, fresh cheese and ripening or ripened cheese; the different forms of date marking were also considered.

36. Following an extensive survey of the chief features of date marking, commercial handling practices of cheese and consumer behaviour, the Committee ultimately concurred with a compromise proposal made by the delegation of France to distinguish between: (i) fresh cheese packaged by the manufacturer, (ii) cut, sliced or grated prepackaged ripened cheese, and (iii) whole cheese which was still ripening.

37. For the fresh cheese (i), and the prepacked out or sliced product (ii), declaration of the date of minimum durability including storage instructions for fresh cheese was proposed. For cheese covered by the third category date marking should not be required at the retail level. It was noted that for certain cheeses, depending on e.g. consumer habits and health risks associated with handling, alternate forms of date marking might be required. When accepting the present General Standard countries could specify these exemptions.

38. The delegation of the Netherlands also considered that whole prepackaged ripened cheese should be date marked at the point of retail sale. The delegation of the USA. expressed preference for date marking for all cheeses offered for sale to the consumer. The delegation of Denmark expressed regret that the question of date marking could become a critical issue in the acceptance of the standard and too strict provisions could make it difficult for many countries to accept this basic standard. In the opinion of the delegation of Denmark only date marking for prepacked unripened cheese for direct consumption should be mandatory. The text agreed to is contained in sub-section 4.8 of the Standard as contained in Appendix II.

Cheese made from Recombined or Reconstituted Milk (4.7)

39. The question was raised whether the text presently in square brackets provided for the exemption of a label declaration when reconstituted skimmed milk was used for the standardization of milk used to produce a low-fat cheese. It was accepted that in such a case a label declaration would be required as the reconstituted skimmed milk was used as an ingredient rather than for restricted standardization purposes. The Committee agreed to remove the square brackets thereby retaining the clause.

Cheese in Bulk (new provision 4.9)

40. It was pointed out that a considerable proportion of the cheese in international trade was shipped in bulk. It was further pointed out that for such cheese, due to loss of moisture, declaration of the net weight was often not feasible. The Committee held the view that for cheese sold in bulk an exemption from the various labelling provisions could be made, including the net weight declaration. It was agreed to insert a new provision stating that for cheese in bulk the information required in sub-sections 4.1-4.7 (inclusive) should be given on the cheese or on the accompanying documents.

Status of the Standard

41. The Committee adopted the General Standard for Cheese at Step 6 of the Procedure for the elaboration of milk product standards and requested the Secretariat to submit it to governments for acceptance at Step 7. The revised standard is contained in Appendix II to this Report. The Committee once more expressed its appreciation for the work done by Dr. Green.

STANDARD FOR EXTRA HARD GRATING CHEESE AT STEP 6 OF THE COMMISSION'S PROCEDURE (C-35)

42. The Committee had before it the Draft Standard for Extra Hard Grating Cheese at Step 6 of the Procedure as contained in Appendix III of the Report of the 17th session and document MDS 76/8 (March 1976) listing written comments received from governments on the draft standard. At its 18th session the Committee, owing to lack of time, had not been able to discuss the standard.

Designation of Cheese (1)

43. Following a proposal by the delegation of Australia the sentence in brackets reading "i.e. cheese suitable for grating" was deleted.

Optional Additions (3.2.2)

44. Several delegations questioned the need of the following additions and requested their deletion: chlorophylls, incl. copper chlorophyll, benzoyl peroxide or mixture of benzoyl peroxide incl. the salts listed, and of sorbic acid and the salts listed. The delegation of Italy proposed to reduce the maximum level for sorbic acid and its salts to 100 mg/kg.

45. The Committee agreed to delete the provisions relating to benzoyl peroxide and the salts listed. It noted that chlorophyll was used for obtaining a whitish colour desired by the consumer and that sorbic acid and its salts were endorsed for a number of other cheeses covered by international individual cheese standards. The Committee agreed to retain the provisions relating to chlorophyll, incl. copper chlorophyll and sorbic acid and 'its salts in the standard, and to amend the last mentioned provision by adding the phrase "in the final product".

Rind-Appearance (4.4.2)

46. The Committee agreed to delete reference to the use of artificial colours.

Holes - Size (4.6.3)

47. The Committee agreed to change this provision to read "size approximately 1-2 mm".

Method of Manufacture (5)

48. In accordance with its decision to delete benzoyl peroxide from the list of additions the Committee agreed to delete the provision allowing its use for bleaching (5.3).

Maturation Procedure (5.5)

49. The Committee accepted a proposal of the delegation of Italy to insert the words "cool and well aerated or" after "held in a". In reply to a query raised by the delegation of Canada concerning hygienic requirements for cheeses manufactured from raw milk, the delegation of the United States explained that a storage of 6-month duration was sufficient to destroy pathogens which might be present in raw milk cheese.

Marking and labelling (7)

50. The Committee agreed to the following text for 7.1 which was the result of an agreement between the delegations of Italy and the USA:

> "7.1 - Cheese conforming to this standard may be designated Extra Hard Grating Cheese or any recognized variety name in the consuming country. A "coined" or "fanciful" name, however, may be used provided it is not misleading and is accompanied by the phrase "Extra Hard Grating Cheese"."

51. The Committee agreed to delete provision 7.3 as its contents were already covered by the preceding provision (7.2) which referred to the labelling provisions provided by Standard A-6.

Status of the Standard

52. The Committee agreed to submit the standard as amended to governments for acceptance at Step 7. The revised standard is contained in Appendix IV to this Report.

REDRAFTS OF GENERAL STANDARDS FOR PROCESSED CHEESES - A-8(a), (b) and (c)

53. The Committee considered at Step 4 the three redrafted general standards for processed cheese as contained in Appendices II (A), (b) and (C) to the Report of the 18th session in the light of government comments (MDS 78/8, MDS 78/Canada and LIM. 1).

Consideration of General Standard for Named Variety Process(ed) Cheese and Spreadable Process(ed) Cheese - A-8(a)

Definition (1)

54. The delegation of Switzerland supported by the delegation of Italy expressed the view that for products covered by the Standard, namely processed cheese with a variety name, the addition of optional ingredients as provided for in the definition should not be allowed and proposed the deletion of the clause. The addition of foodstuffs such as ham changed quite considerably the organoleptic properties of cheese of a named variety. Subsequently the designation would have no longer any meaning and could mislead the consumer. It was pointed out that in the labelling section provision was made for the declarations of the optional ingredients. The definition was not amended; the two delegations retained their position.

Food Additives (3)

55. The Committee agreed that the maximum levels given for the various additives applied to the final product. A proposal was considered to express the phosphorus contained in various emulsifying salts and acids as P_2O_5 or phosphorus so as to provide for an analytical control of the maximum level. It was noted that the P_2O_5 level of the various salts varied considerably (40-70%) and furthermore that there were also variations in the phosphorus content of the different cheeses used for processing. It was also noted, however, that a method existed to calculate the phosphate content originating from the processing salts derived from the total phosphorus determination (see para 124 of this Report). The Committee agreed to a maximum level of total added phosphorus compounds at a level of 9 g P/kg process(ed) cheese.

56. The Committee decided to allow for the use of chlorophyllin copper compounds as a colour (new 3.3) according to GMP. Vinegar was moved to the optional ingredients section. In the provision for preservatives (new 3.4) a correction was made so that the maximum level in the final product for sorbic acid and propionic acid and their salts, used singly or in combination, would be 3000 mg/kg expressed as acid. Calcium chloride was deleted; sodium hydrogen carbonate was regrouped under acidifying and pH controlling agents.

Heat Treatment

57. To ensure that the product was uniformly heat treated to the required minimum temperature the Committee agreed to insert the word "throughout" before "heated".

Composition of a Named Variety Process(ed) Cheese (5.2)

58. The Committee concurred with a proposal of the delegation of Switzerland to delete the reference to "Appenzeller" cheese from the list of cheeses exempted from the provision on general minimum dry matter content (5.2.2). The delegation of Australia supported by the delegation of New Zealand proposed that in addition to Process(ed) Gruyere and Emmental, also Process(ed) Cheddar be exempted from the general minimum dry matter requirement. It requested that a moisture differential of 6% be allowed, which would correspond to a minimum dry matter content of 55%. One delegation stated that adoption of the Australian proposal might possibly affect acceptance by governments of the Cheddar Standard. The Committee did not agree to expanding the list of exempted cheeses.

59. It was pointed out that the present numbering of the sub-sections implied that the provision "if national legislation differing from the above exists, the national legislation of the consuming country prevails" (5.2.4) applied to the whole section. Several delegations expressed the view that in this provision, specifying minimum compositional requirements for the various named variety process(ed) cheeses, it was not acceptable to include a clause (5.2.4) allowing national legislation of the consuming country to prevail if such legislation differed from the provision.

60. It was thought that the inclusion of a reference to national legislation was against the philosophy of international standardization. Following a detailed discussion the Committee agreed that the conditional clause allowing national legislation to prevail would apply only to varieties for which no international standards existed. It was stated that trade in these products was of limited significance. The delegations of Australia, Canada and France wished to place on record their objection to the retention of the conditional clause (old 5.2.4).

Composition of a Named Variety Spreadable Process(ed) Cheese (5.3)

61. The Committee agreed to bring the text in this provision in line with the proceeding one (5.2) covering non-spreadable process(ed) cheese.

The Name of the Food (6.1.4)

62. The provision was amended in line with the decision taken with regard to the form of declaration of the fat content in the General Standard for Cheese (see para 25).

63. The delegation of France supported by the delegation of Belgium stated that in their countries the word cheese need not be included in the name of the product when a variety name was used to describe Process(ed) Cheese or Spreadable Process(ed) Cheese. The delegation of Sweden expressed the view that the word cheese should at all times be part of the name.

Date Marking (new 6.6)

64. The Committee agreed to the inclusion of the following provision (as in the standard for Cream, A-9): "There shall be a clear indication of the minimum durability date".

Lot Identification (new 6.7)

65. It was decided also to provide for lot identification (as in the Standard for Cream, A-9): "Each container shall be permanently marked in code or in clear to identify the producing factory and the lot".

Methods of Sampling and Analysis

66. When discussing the phosphorus determination under the additives section (see para. 55) reference was made to a method for the calculation of phosphorus originating from the melting salts being developed by IDF. The Committee requested the Joint IDF/ISO/AOAC Working Group to consider this matter (see para 124 (ii) of this Report).

67. The Committee further agreed to request the Joint Working Group to select a method to determine the dry matter content (see para 124 (i) of this Report).

68. The delegation of Egypt requested that a method be included in the standard for the determination of lard in processed cheese. The Committee requested the Joint Working Group also to investigate this matter (see also para 126 of this Report).

Consideration of General Standard for "Processed Cheese" and "Spreadable Process(ed) Cheese" - A-8'(b)

69. The Committee agreed to make, where appropriate, the same amendments as were made in the Standard A-8(a).

Optional Ingredients (2)

70. The delegation of Norway, supported by the delegation of Denmark, questioned the appropriateness of allowing for the unrestricted addition of cream, butter and butteroil (2.1) and limited addition of milk products (2.2) in the manufacture of a product designated process(ed) cheese. It was pointed out that the use of milk fat was self restricting and that the manufacture of a largely composite product complying with the upper limit of 5% lactose in the final product would be rather difficult.

71. The delegation of Japan stated that most processed cheeses manufactured in Japan were covered by Standard A~8(b) and consequently it could not agree to the optional addition of other milk products (2.2). If, however, these milk products were

added, the resulting food should be classed under Standard A-8(c) as a process(ed) cheese preparation. The Committee amended only the provision 2.2 editorially to read: "Other milk products may be added to a maximum of 5% lactose content in the final product". The delegation of Denmark reserved its position with regard to the unlimited use of dairy products with low lactose content.

Optional Food Additives for Products Exclusively in Transparent Packs (3.2.4)

72. A number of delegations were of the opinion that it was unjustified to provide for antioxidants specifically to allow for the use of transparent packs. The Committee agreed to delete the provision (3.2.4).

73. The delegation of Egypt drew attention to the transfer of contaminants from the packaging material into the product. It was recognized that this was a general problem and the Committee agreed to consider the matter at its next session in the light of data provided by governments.

Consideration of General Standard for Process(ed) Cheese Preparation - A-8(c)

74. The Committee agreed to make, where appropriate, the same amendments as were made in the Standard A-8(a).

Status of the Standards

75. The Committee agreed to adopt the Standards A-8(a), (b) and (o) at Step 4 of the Procedure for the Elaboration of Milk and Milk Product Standards and further agreed to the omission of Steps 5 and 6 and to submit the Standards to governments for acceptance at Step 7. The revised Standards are contained in Appendix III (a), (b) and (0) to this Report. The delegation of Australia recognized the general wish of the Committee but reserved its position pending further consultation with its government.

CODE OF HYGIENIC PRACTICE FOR DRIED MILK

76. The Committee considered at Step 4 of the Procedure the Proposed Draft Code of Practice for Dried Milk (MDS 78/8) prepared by the delegation of Australia in the light of a list of recommendations made by an informal group consisting of members of some delegations attenting the session (LIU. 3). The Chairman gave the background for the preparation of the Code of Hygienic Practice for Dried Milk and expressed appreciation, on behalf of the Committee to the delegation of Australia for the preparation of an excellent working document.

77. The representative of WHO pointed out that in the preparation of the Code the revised version of the General Principles of Food Hygiene (ALINORM 78/13A, Appendix V -Step 6) had been adopted to a great extent. It was also pointed out that the recommendations of the 2nd Joint FAO/WHO Expert Consultation on Microbiological Specifications for Foods as to the microbiological criteria to be employed in guidelines had been taken into consideration. The Committee noted that the hygienic provisions of the Code were to be considered by the Codex Committee on Food Hygiene.

78. In introducing the Code the delegation of Australia informed the Committee that the Code had been redrafted taking into account comments made at the 18th Session of the Committee, the Revised Code of General Principles and written comments from Canada, Finland, Poland, United Kingdom and USA. The delegation also pointed out that it was not the intention to cover casein and casein related products by the Code.

79. The Committee agreed to discuss only those sections of the Code which referred specifically to dried milk products.

Scope (1)

80. The delegation of the Netherlands thought that the Scope should be amended to emphasize that the Code was of an advisory nature and was not intended to form part of mandatory legislation. Other delegations were of the opinion that the word "recommend" in the Scope and the conditional language throughout the Code was sufficient indication that the Code was a guideline.

Definitions (2)

Establishment (2.6)

81. It was pointed out that in the French version it was not clear whether "the official agency having jurisdiction" applied to authorities within the country or to those representing the importing countries. The rapporteur expressed the view that the authority indicated was the official agency of the producing country. After some further discussion the Committee decided to revise the text and adopted the following:

> "Establishment: any building(s) or area(s) in which dried milk products are prepared, processed, handled, packed or stored and the surroundings under the control of the same management".

Pasteurization (2.10)

82. The delegation of the USA pointed out that in its opinion the present text did not adequately cover products other than milk, skimmed milk and whey. It proposed to amplify the definition to include time/temperature relationship for milk products having a higher milk fat content than milk and/or containing sweeteners, and concentrated milk and concentrated milk products.

83. Other delegations thought that the text should be held in general terms or at the most refer to the phosphatase test as an indication of adequate pasteurization. It was pointed out that as a separate item on the agenda the Committee would also consider a definition for pasteurization and it was thought desirable to harmonize the provisions. After some discussion the Committee agreed to accept the proposal of the delegation of 'the USA.

Hygienic Requirements in Production Area (3)

84. The delegation of the Federal Republic of Germany proposed that the Code should be extended to cover raw milk. The rapporteur informed the Committee that after considerable discussion it had been decided to include general provisions for raw milk in the present Code (sub-section 7.1).

Establishment: Design and Facilities (4)

85. An enquiry was made as to whether salient points in the text could be emphasized by appropriate changes in typography. The Secretariat informed the Committee that the available equipment did not provide for this, but that the matter could be further investigated.

Steam (4.4.2.2)

86. The delegation of Cyprus expressed the opinion that the text should include a provision which excluded hazardous volatile chemicals in water used for steam production. The Committee agreed to take account of this observation and amended the text accordingly.

Sanitary design, construction and installation (old 4.5.3.1)

87. In order to ensure that there was a direct relationship between flow rate and temperature the Committee agreed to include provisions for a positive pump or timing device in the pasteurization line.

Processing (7.4)

88. The Committee agreed to restrict the necessity for extended storage, and included in the text (7.4.6) provisions for two feed-balance tanks to be used alternatively and cleaned and sterilized at intervals not exceeding two hours.

89. It was agreed that recordings of heat treatment should be kept for a period of at least one year (7.4.9 and 7.7.8). The Committee did not accept a proposal to retain records during the declared period of minimum durability of the product.

Sampling and Laboratory Control Procedures (7.7)

90. It was agreed to specify that the person in charge of the quality control programme should be aware of the significance of contamination and the hazards involved, and the Committee amended the text accordingly.

End-Product Specifications - General Requirements (8.1)

91. The Committee agreed to employ with appropriate modifications in this Code the general text endorsed for a number of codes of practice.

Draft Proposal for Microbiological Specifications for Dried Milk Products

92. The Committee considered the above document (MDS 78/8- Add. 1) which was introduced by the delegation of Australia.

93. The Committee noted that the specifications had been prepared in the light of the General Principles for the Establishment of Microbiological Criteria for Foods recommended by the Second Joint Expert Consultation on Microbiological Specifications for Foods, which had divided microbiological criteria into three categories:

(i) A microbiological standard is attached to a Codex Alimentarius Standard, which is mandatory. It is intended for use in case of disputes. It shall not be introduced de novo but shall be derived from specifications which have accompanied codes of practice through the Codex procedure and which have been extensively applied to the food.

(ii) A microbiological specification is attached to a code of practice, which is of an advisory nature, and is intended to increase assurance that the provisions of hygienic significance in the code have been adhered to.

(iii) A microbiological guideline is to be used where no standard or code of practice for the particular food exists. A guideline should be established only when a microbiological criterion is urgently required for a food moving in international trade.

94. The appropriate category to codes of practice, that is number (ii), was applied in drawing up the present specifications.

95. In the general discussion that followed, some delegations were of the opinion that Section 5, which referred to Routine Sampling Plans and Microbiological Limits, was not appropriate to the specifications since these concerned Good Manufacturing Practice which was already covered by the Code of Practice itself. Other delegations thought that the methods to which reference was made should be routine methods. The Committee agreed that the End-Product Specifications were to be; retained.

Sampling Plans and Microbiological Limits

96. There was considerable discussion as to which microorganisms should be included in the specifications. Some delegations were of the opinion that only organisms of public health significance should be included, in which case specifications for Salmonella were necessary. Others, however, thought that it was important to retain both the mesophilic aerobic count and the coliform count because these were an indication that the end-product had undergone satisfactory processing.

97. Several delegations proposed that Staphylococcus aureus should also be considered in view of the danger that the heat-stable toxin would persist even after destruction of the causative organisms.

98. It was pointed out that although the First and Second Expert Consultations had not considered Dried Milk Products as such when drawing up their list of priority items, as work was already in progress in this Committee, they had nevertheless information from some 19 countries on the organisms at present determined in milk products and that the tests most frequently carried out concerned Salmonella, standard plate count and coliforms. However, the First Consultation had considered the general question of whether Staphylococcus aureus was of significance in international trade and had expressed the view that this microorganism, either as a potential producer of enterotoxin or as an indicator of contamination by man, could be expected in international specifications for many foods. Estimation of enterotoxins was not likely to become routine until the reagents necessary for their detection became more readily available and more rapid methods were developed. The Consultation made particular reference to the heat stable nuclease test as a method for detecting S. aureus after thermal death.

99. Some delegations were of the opinion that, in view of the particular conditions prevalent in hot countries, the inclusion of moulds and yeasts should be considered. It was pointed out that these microorganisms were connected with the contamination of the packaging material rather than of the contents and, while a real problem in tropical climates, should not be included in product specifications.

100. The Committee noted that in all microbiological specifications there was a close correlation between the sampling method and the numerical limits for the microorganisms concerned and that an important factor which must be borne in mind in microbiological control was the cost/benefit of any methods proposed for microorganisms.

101. The Committee agreed that the specifications mentioned in the present document and these discussions should be brought to the notice of the Codex Committee on Food Hygiene and to the FAO/WHO Working Group on Microbiological Specifications for Foods which would meet in February 1979. This group was asked to pay particular attention to practical limitations in applying sampling techniques so that the costs of testing could be weighed against the benefits obtained in protecting the consumer.

102. With regard to specifications for Salmonella, it was noted that the ISO Method, which bore a close resemblance to the method for the detection and determination of Salmonella proposed by the First Joint Expert Consultation, had been adopted by the Codex Committee on Food Hygiene for inclusion in the microbiological specifications for Dried Egg Products.

103. It was agreed that the Codex Committee on Food Hygiene should be asked to consider whether already agreed upon methods should replace the present (AOAC) method in the specifications for Dried Milk Products.

Status of the Code

104. The Committee agreed to adopt the Code at Step 4 of the Procedure for the Elaboration of Milk and Milk Product Standards and to refer the Code for further development to the Codex Committee on Food Hygiene. The Committee expressed the wish to have the opportunity to review the Code before it was finalized.

HATTERS OF INTEREST ARISING PROM THE 12TH SESSION OF THE CODEX ALIMENTARIUS COMMISSION -FUTURE WORK OF THE COMMITTEE

105. The Committee had before it document MDS 78/3(0) which contained an extract of the Report of the 12th Session of the Codex Alimentarius Commission concerning the work of the Committee. In introducing the document, the Secretariat referred to the opening speech as contained in para 3 of this Report. With regard to the Commission's conclusion that the Committee should go into recess, the Secretariat stressed that while several Codex Committees had gone into recess, they had not been abolished but would be convened again in the light of new developments and the necessity for revisions. The Secretariat than briefly referred to a question concerning the Draft Standard for Low-Fat Spreads containing Milk Pat, which had been raised by the Codex Committee on Fats and Oils and the Codex Commission and to the request of the Commission that the Milk Committee should not start work on new items, including imitation milk products. The Committee agreed to deal separately with (i) its future work, (ii) the question of low-fat spreads containing milk fat, and (iii) imitation milk products.

Future Work

106. With regard to its future work the Committee discussed the following statement presented by the delegation of Denmark:

"Having been informed through MDS 78/3(o) about the Commission's discussion of the future work of the Expert Committee on the Code of Principles and about the Commission's request to the Committee, the Committee wishes to express the following views:

> The Committee, having first demonstrated that it was possible on a governmental basis to reach international agreement on principles for designations, definitions and standards for foods, finds that the work done so far has been of importance to the protection of the consumers and of assistance for fair and honest national and international trade in milk and milk products. The large number of governments having accepted the Code of Principles and the associated standards confirm to the Committee these findings. The Committee is further aware of the influence the Code and Standards have in national legislation in developing as well as developed countries.
>
> The Committee agrees with the view that it has reached a point where it has elaborated standards for the most important milk products and that consequently it could adjourn sine die. Realizing this situation the Committee has rearranged the agenda for its 19th Session and has finalized or nearly finalized the most important items on the agenda, including the revised General Standard for Cheese, the revised standards for processed cheese and processed cheese products and the standard for hard grating cheese.

"The Committee, having made every effort to finalize as much work as possible during the present meeting wishes, however, strongly to recommend that before it adjourns sine die it be given the opportunity to have at least one more scheduled session, in order not to leave unfinished the following major tasks which are already on the working programme of the Committee:

(a) Review of the food additive lists in the compositional and in the individual cheese standards;
(b) changes in the individual cheese standards which have been held for some years pending the finalization of the General Cheese Standard;
(c) Code of hygienic practice for dried milk products and microbiological specifications;
(d) work on methods of sampling and analysis. The Committee could invite the three organizations, IDF, ISO, AOAC, to continue their excellent cooperation in this field and could inform them as to which methods the Committee feels should be given high priority;
(e) eventually the revision of the Standard for Cream Cheese;
(f) paying attention to IDF in its advisory capacity as to which work the Committee would like to see done by IDF until the Committee is reconvened.

"The Committee, being aware, through experience, that developments may require amendments to and changes in existing standards as well as elaboration of standards for new products, expects to be reconvened as and when the developments so require.

"Especially in the field of milk and milk products national legislation is to a wide extent united to the Code and the standards and vice-versa and the work already done will easily lose its importance if it is not in harmony with the development.

"The Committee therefore finds that a procedure for reconvening the Committee would be useful and that IDF in this respect could play a role as to when, or about when, the developments may require the Committee to be reconvened."

107. The delegation of the Netherlands stated that in general it could agree with the view of the Codex Alimentarius Commission that for the most important milk products compositional standards had been elaborated and that there was no need to embark at present on the standardization of other milk products. It could also agree with the Danish view that standards already published in the Code of Principles needed revision periodically due to technological developments and consumer demands. Since the responsibility for the compositional milk and milk products standards rested with this Committee, it should decide when standards needed updating. The Committee had agreed some years ago to consider the revision of standards every five years. This decision had proved to be satisfactory. Taking into account that a number of standards had already been published or revised some years ago, the delegation of the Netherlands proposed that a meeting of the Committee be convened after a period of four years. This would give the IDF a better possibility to act in its advisory capacity as mentioned in the programme for the elaboration of milk and milk products standards. It further proposed that the Committee should ask the IDF to carry out the necessary preparatory work.

108. The statements of the delegations of Denmark and the Netherlands were supported by the delegations of Poland, New Zealand, Norway, Prance, Spain, Iran, Finland, USA, Federal Republic of Germany and Switzerland.

109. The representative of the IDF then made the following statement:

"The FAO/WHO Committee of Government Experts on the Code of Principles for Milk and Milk Products was created on the initiative of the International Dairy Federation and by decision of the FAO Conference in 1958 in order to protect the consumer and to assist the dairy industry by ensuring the precise use of the term "milk" and the terms used for the different milk products and by establishing definitions and designations and minimum standards for these products.

"The general principles of the Code were accepted by 72 governments. It is, therefore, understandable that IDF as the initiator of the Code and as a body having Specialized Consultative Status with FAO is concerned about developments in relation to the future of the Code as discussed during the 12th Session of the Codex Alimentarius Commission in April 1978. It had been recognized by the Commission at this Session that the Committee on the Code of Principles concerning Milk and Milk Products has done excellent work and that a whole range of useful standards, both composite and analytical, had been developed for a number of important dairy products. These standards meet international requirements as far as consumer aspects and conditions for fair trade are concerned.

"When now the question arises whether the work of the Committee on the Code of , Principles may be considered as completed, it should be pointed out that the Committee itself made it clear, that it is required periodically to revise the existing standards in the light of new developments since a standard;, however good, is not static but must be adapted to changing requirements from the angle of the consumer, of advanced technology and of new nutritional findings. Standards need also to be periodically adjusted to the changing requirements emanating from the work of the horizontal. Codex Committees.

"In due consideration of its consultative status with FAO and of its obligation as the dairy expert organization the IDF will continue to deal with the matter of the Code, irrespective of possible changes in FAO structures. Accordingly the respective technical bodies of IDF have already initiated and will continue to work on the following items:

(i) Hygienic requirements (end-product specifications) for each of the compositional standards of the Code;
(ii) Code of hygienic practice for the dairy industry;
(iii) compositional standards for further products (at present for whey powder);
(iv) principles for the labelling of milk and milk products (in the context of the Codex Standard for the Labelling of Prepackaged Foods);
(v) tentative classification of various types of dairy products according to the Cod© (composite and modified products);
(vi) general standard for substitute products (products similar to dairy products in which one or more ingredients are replaced by non-dairy ingredients).

"IDF is furthermore prepared:

- to continue in the joint work of AOAC, ISO and IDF on methods of sampling and analysis;
- to consider the applications for International Cheese Standards as requested in the Procedure for the Elaboration of International Individual Cheese Standards (Step 2);
- to review the additive lists in all Code Standards in accordance with decisions emanating from the work of the respective Codex Committee;

- to do the preparatory work for the revision of Code Standards as provided by the Code Committee.

"The IDF trusts that these activities, even at the preparatory stage, should encompass the legitimate views of all interested parties. The IDF therefore, wishes to invite all countries, irrespective of their membership, and FAO and WHO Representatives to take part in the work by participating in the respective working groups. The IDF will report to the Committee's Secretariat on the progress made with the aim to inform the governments accordingly."

110. During the discussion of the various statements, the date of the next session of the Committee was also considered and it was generally agreed that the 20th session should be convened in approximately three years time. This interval would allow an adequate preparation of the work to be finalized at the next meeting after which the Committee would adjourn sine die. It was further agreed that before the Committee went into recess, a clearly defined mechanism be established for reactivating it. The Secretariat was requested to ensure that conclusions from other Codex Committees of concern to the Milk Committee be circulated not only to member governments but also to delegates participating in the work of the Milk Committee.

111. In addition to the reasons given in the statement of the Danish delegation for convening another session before the Committee went into recess, the view was expressed that the session proposed above should also be held to encourage a high level of acceptance of the standards elaborated (and amended). It was agreed that the work would continue and that the Secretariat also in the future should ask governments to comment on matters in relation to the Committee's work.

112. The Committee gave its support to the opinions expressed and agreed to request-the Commission to give due consideration to the issues raised.

113. The Chairman speaking on behalf of the Committee thanked the representative of IDF for the work this Organization had carried out and for the intention of IDF to continue giving its support to the Committee's work as indicated in para 100.

LOW PAT SPREADS

114. The Committee noted that the Codex Committee on Fats and Oils (CCFO) had under preparation a standard for low fat spreads based on fats not mainly derived from milk. The Codex Alimentarius Commission had been asked by the Codex Committee on Fats and Oils to determine whose responsibilities it should be to develop standards for low fat spreads based solely or mainly on milk. The Commission decided that the Codex Committee on Fats and Oils should be asked to reconsider its decision in the light of the views of the Milk Committee on this matter. The Committee agreed that the standard for low fat spreads being elaborated by the CCFO should cover all products except those where the fat content was solely derived from milk.

115. The delegation of Denmark, supported by the delegations of France and Norway, wanted to put on record their views that if and when standards covering products containing mixtures of milk fat and non-milk fat were elaborated, the expertise of both the Milk Committee and the Codex Committee on Fats and Oils should be employed. The delegation of Sweden was of the opinion that the standard for low fat spreads under preparation in the CCFO should cover all types of products including those containing solely milk fat.

IMITATION MILKS

116. The Committee had before it document MDS 78/9 summarizing information received from a number of governments on the manufacture of imitation milks. The Committee, having agreed with the recommendation of the Codex Alimentarius Commission not to embark on standards for imitation milk, briefly considered a proposal by the delegation of Denmark for a "Decision No. 6" of the Committee dealing in more general terms with compositional, hygienic and food additive aspects of imitation milk. The Committee agreed to submit the draft to governments for comments. The delegation of Denmark underlined that the proposal was intended to be a decision of the Committee. The text of the proposed Decision No. 6 is contained in Appendix V to this Report.

INTERNATIONAL INDIVIDUAL CHEESE STANDARDS

117. The Committee had before it document MDS 78/6 listing the applications for international individual cheese standards not yet dealt with by the Committee. The Committee recalled the decision it had taken at its 14th Session that further work on applications for cheese standards should be deferred until the results of the work on classifying cheese could be more clearly evaluated. Having finished its work on the redraft of the General Standard for Cheese A-6 the Committee decided to take no action on the remaining applications until there was a demonstrated need in the future to continue this work.

118. The following delegations stated that their governments wanted to withdraw their applications, listed hereunder:

Denmark	Elbo Tybo Mycella
Norway	Ekte Geitost Nokkelost Gammelost
Sweden	Kaggost Västerbottenost Prästost
Italy	Taleggio

119. The delegations of France and Switzerland wanted to have put on record their objection against the use of the designations "Cantal" and "Sbrinz" respectively in the applications received from Turkey and Uruguay respectively. The designation "Cantal", the name of a French Department, was internationally registered as an appellation d'origine and the designation "Sbrinz" was protected by an appellation d'origine in Switzerland and by bilateral agreements. The delegation of New Zealand placed on record that it desired to maintain its application for Egmont cheese.

REVISION OF THE STANDARD FOR CREAM CHEESE

120. The Committee noted that the Secretariat had not received a revised text from the depositing countries and that therefore no action could be taken.

DRAFT STANDARD FOR CO-PRECIPITATED EDIBLE CASEIN

121. As agreed at its 18th Session (para 155) the Committee had before it a document prepared by IDF containing a proposed draft standard for co-precipitated

casein (MDS '78/10). The Committee was informed that the product was not of great significance in international trade and it was proposed that the elaboration of the standard be postponed. The Committee agreed to this. The delegation of France expressed the view that instead of co-precipitated edible casein, co-precipitated edible milk protein was a better description of the product. The Committee took no further action.

DEFINITIONS OF PASTEURIZATION, STERILIZATION, UHT-PROCESSES

122. The Committee agreed with the suggestion of the Secretariat that governments should be asked to provide information on national legislations on definitions for pasteurization, sterilization and UHT-processes. The Committee also agreed that the Secretariat should ask IDF to submit to the Committee the results of their work on these definitions for consideration at its 20th Session. The delegation of the Federal Republic of Germany stated that in its opinion pasteurization, sterilization and UHT-treatment were among the most important hygienic requirements and should be included in a code of hygienic practice for milk and milk products; minimum and maximum time/temperature coordinates would have to be specified.

IDF/ISO/AOAC COOPERATION IN THE FIELD OF METHODS OF SAMPLING AND ANALYSIS

123. The Committee was informed of the work in the field of sampling and analysis done by the representatives of IDF/ISO/AOAC, during their traditional meeting prior to the present session of the Committee, by the representative of IDF, Dr. H. Kay. The report of the meeting (MDS 78/12(b)) is contained in Appendix VII to this Report. The Committee noted that the report referred to the activities of more than 30 joint expert groups of the three organizations during the last two years and that the results of the work in the various expert groups were followed-up and evaluated by representatives of the three organizations during regular meetings.

124. In addition, the Rapporteur drew the Committee's special attention to the following items which were dealt with by the Committee during its 19th Session:

(i) In connection with the General Standard for Cheese (A-6) and the General Standard for Processed Cheese Products (A-8), a method for the determination of dry matter was in preparation by a joint working group of the three organizations which were revising the IDF Standard 4. A new draft of the method would be sent to the member countries of the three organizations in the course of this year.

(ii) As far as the analysis of phosphates in processed cheese products was concerned, a method for the calculation of the quantity of emulsifiers from the phosphorus content as determined by Standard B-12 was available in IDF. The method would be reconsidered and sent to AOAC and ISO with the aim of reaching agreement.

(iii) Work on analytical methods to determine heavy metals in dairy products was in progress. It was noted, however, due to the rather diverging procedures used in the various countries and the not too satisfying results of national and international comparative tests with these methods, that the finalization of internationally agreed methods would take some time. This would result in some delay in setting up maximum levels for heavy metals in the standards for dairy products.

(iv) Microbiological specifications for dried milk products as a part of the Code of Hygienic Practice. The joint groups of the three organizations were involved in the elaboration of the necessary methodology and sampling plans. Since sampling plans were closely related to the microbiological limits the joint AOAC/ISO/IDF groups would have to deal with these limits.

125. Dr. Kay thanked the representatives of AOAC and ISO for their constructive cooperation and on behalf of the three organizations expressed the willingness of IDF, ISO and AOAC to continue the joint work on international standardization of analytical methods for dairy products.

126. With reference to the FAO/WHO Standard Methods B-16 and B-17 - Detection of Vegetable Fat in Milk Products - the delegation of Saudi Arabia asked whether a method existed for the determination of lard in processed cheeses. The representative of IDF informed the Committee that these methods did not cover this aspect. However, he indicated that the three organizations would look into this matter.

127. The Committee adopted the report as contained in MDS 78/12(b) and the Chairman, on behalf of the Committee, expressed his appreciation for the excellent work done by the three organizations.

ACCEPTANCE OF MILK PRODUCT STANDARDS - THE MEANING OF SPECIFIED DEVIATIONS

128. The Committee recalled that during its 18th Session (1976) it had set up a drafting group to consider the meaning of specified deviations in accepting standards for milk and milk products under the Code of Principles and/or the General Principles of the Codex Alimentarius. There had been general agreement in the Committee that the proposed guidelines on this subject which had been drawn up by the drafting group should be sent to governments for consideration. The views of a number of governments were before the present session of the Committee in document MDS 78/3(b). In the proposed guidelines it had been recommended that governments should use the Codex acceptance forms to transmit notifications of acceptance. The proposed guidelines had appeared in the Report of the 18th Session of the Committee as Appendix VII and the Codex acceptance form as an Annex to Appendix VII.

129. The Committee, at its present session, was informed of the developments which had taken place in the Codex Committee on General Principles and in the Commission on the subject of "acceptance with specified deviations" and, in particular, on the question of whether there was a need to establish criteria for drawing a line of demarcation between meaningful acceptance and non-acceptance in relation to "acceptance with specified deviations". The Committee was informed that this was a question on which there were two schools of thought both in the Codex Committee on General Principles and in the Commission and that the matter would be considered further by the Codex Committee on General Principles at its next session. The Committee was also informed that there was general agreement in the Codex Committee on General Principles that any such criteria, if established, should be solely for the purpose of offering guidance to governments in choosing between acceptance with specified deviations and non-acceptance: it was not contemplated that such criteria "be used by the Commission, with the notion of the Commission pronouncing on a country's acceptance statement, in the case of "acceptance with specified deviations".

130. In the light of the above, the Secretariat proposed amendments to the proposed guidelines contained in Appendix VII to the Report of the Committee's 18th Session. It

was explained to the Committee that the sole purpose of the amendments was to remove from the text the words which implied that the Committee would, at some stage, exercise judgement on acceptance statements of governments choosing to notify "acceptance with specified deviations". The Committee adopted the following amended version of the guidelines?

"The Meaning of Specified Deviations in Accepting Standards for Milk and Milk Products under the Code of Principles and/or the Codex Procedure

Guidelines to Governments

"The Committee considered the effect of the new Codex Acceptance Procedure in relation to standards for milk products elaborated under the Code of Principles. Since the Code of Principles Preamble states in part: "The purpose of this Code of Principles is to protect the consumer of milk and milk products and to assist the dairy industry on both the national and international levels by:

> ENSURING the precise use of the term "milk" and the terms used for the different milk products; **...**
>
> ESTABLISHING (a) definitions and designations; (b) minimum standards of composition, **...**"

the Committee agreed to recommend to governments that, when considering the giving of acceptance to standards, they should look upon any acceptance statement with less than the minimum compositional requirements (less stringent requirements) for a standard as not being an acceptance under any qualification.

"The Committee also agreed to recommend to governments that when acceptance of standards under the Codex Procedure was being considered any deviation from the requirement in the standard relating to Definitions, Essential Composition and the provisions relating to the Name of the Food should not be considered as specified deviations relative to acceptance of the standards except in very special circumstances not in conflict with the Code of Principles.

"The Committee also recommended that governments use the forms for acceptance of recommended Codex standards provided by the FAO Secretariat to transmit notices of acceptance."

DATE AND PLACE OF NEXT SESSION

131. The Committee noted that the twentieth session would be held in approximately three years time. It was proposed to hold the session immediately following the annual IDF sessions to facilitate participation by overseas delegations. The Committee requested Secretariat to take this suggestion into account when drawing up the schedule of Codex meetings.

CX 5/70-19th Session
APPENDIX I

LIST OF PARTICIPANTS*
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LISTA DE PARTICIPANTES

* The Heads of delegations are listed first; Alternates, "Advisers and Consultants are listed in alphabetical order.

Les chefs de délégations figurant en tête; les suppléants, conseillers, consultants sont énumérés par ordre alphabetique.

Figuran en primer lugar los Jefes de las delegaciones; los Suplentes, Asesores y Consultores aparecen for orden alfabétioo.

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CX 5/70-19th Session
APPENDIX II

Submitted to Governments for acceptance

standard A-6
Step 6

RECOMMENDED GENERAL STANDARD FOR CHEESE

1. SCOPE

This standard applies to all products in conformity with the definition of cheese in paragraph 2 of this standard, including those varieties of cheese for which individual or group standards have been elaborated. Subject to the provisions of this standard, standards for individual varieties of cheese, or groups of varieties of cheese may contain provisions which are more specific than those in this standard and in such cases those more specific provisions shall apply to the individual variety or groups of varieties of cheese. The standard does not apply to whey cheeses.

2. DEFINITION

Cheese is the fresh or matured solid or semi-solid product obtained:

(a) by coagulating milk, skimmed milk, partly skimmed milk, cream, whey cream, or butter milk, or any combination of these materials, through the action of rennet or other suitable coagulating agents, and by partially draining the whey resulting from such coagulation; or

(b) by processing techniques involving coagulation of milk and/or materials obtained from milk which give an end-product which has the same essential physical, chemical and organoleptic characteristics as the product defined under (a).

3. ADDITIONS

3.1 Flavouring substances

For cheese for which there is an international individual or group standard only those additions permitted in the individual or group standard may be used.
Natural flavouring substances not derived from milk such as spices, may be added in such quantity that they can be considered only as flavouring substances, provided that such substances are not intended to take the place of any milk constituent and provided that the cheese remains the major constituent.

3.2 Other additions

For cheese for which there is no international individual or group standard only those additions may be used which are technologically necessary and which are permitted in an international individual or group standard for a similar type of cheese according to the characteristics classified in the Annex or, in the absence of a similar type of cheese, for the type of cheese nearest in character.

4. LABELLING

In addition to sections 1,2,4 and 6 of the Recommended International General Standard for the Labelling of Prepackaged Foods (Ref. No. CAC/RS 1-1969), the following specific provisions apply;

4.1 Name of the Food

4.1.1 All products designated cheese or with the name of a variety of cheese must conform to this standard.

4.1.2 Products conforming to the standard shall be designated cheese and shall include the name of the variety or fanciful name if there is one, except that where an international or group standard is applicable or the cheese is defined in national legislation only the specific designation may be required.

4.1.3 The designation shall be accompanied by a declaration oft

(a) the appropriate designation in accordance with the classification of cheese in the Annex; and

(b) the minimum fat in dry matter content, and/or the fat content expressed as a percentage by mass, except that such declarations need not be made in countries of retail sale in which:

(i) an international or group standard for the cheese is applicable, or
(ii) the composition of the cheese is specified in national legislation.

4.1.4 Where milk other than cows' milk is exclusively used for the manufacture of the product a word or words denoting the animal from which the milk has been obtained shall be inserted immediately before or after the designation of the product, and where milk from more than one species of animal is blended the milk from the different species shall be declared in descending order of proportion. Such declarations are not required if the consumer would not be misled by the omission.

4.1.5 An indication shall be given of the addition of spices or other natural flavouring substances except in the case of cheeses in which the presence of these substances is a traditional characteristic.

4.2 Ingredients

Ingredients other than those obtained from milk, starter cultures, rennet and coagulating enzymes shall be declared on the label in descending order of proportion.

4.3 Net weight

The net weight shall be declared in either the metric system ("Système international" units) or avoirdupois or both systems of measurement as required by the country in which the cheese is sold.

4.4 Name and address

The name and address of the manufacturer, packer, distributor, importer, exporter or vendor of the product shall be declared.

4.5 Country of manufacture

The country in which the cheese is manufactured shall be declared if its omission would mislead or deceive the consumer. In particular, cheese designated with the name of a variety and not manufactured in the country of origin of the variety must be marked with the country of manufacture even when sold on the home market.

4.6 Lot identification

The cheese shall be given a "lot" or "batch" number and inscription which will enable the origin and date of manufacture to be determined.

4.7 Cheese made from recombined or reconstituted milk

Cheese conforming with this standard and made from recombined or reconstituted milk, skimmed milk, partly skimmed milk, or cream may be designated cheese provided that there is a prominent label declaration "made from recombined (X)" or "made from reconstituted (x)", to be completed by inserting at (x) milk, skimmed milk, partly skimmed milk, or cream as appropriate. This labelling provision shall not apply to the use of reconstituted skimmed milk used for the preparation of starter cultures nor for standardizing the fat/ casein ratio.

4.8 Date marking

There shall be a clear indication of the date of minimum durability for:

- fresh cheese packaged by the manufacturer, and
- cut, sliced or grated prepackaged ripened cheese.

No date marking is required for whole cheese which is still ripening.

4.9 Cheese in bulk

In the case of cheese in bulk the Information required in 4.1-4.7 shall be given on the cheese or in the accompanying documents.

5. METHODS OF SAMPLING AND ANALYSIS

5.1 Sampling

According to FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products", paragraphs 2 and 7.

5.2 Fat content

According to FAO/WHO Standard B-3 "Determination of the Fat Content of Cheese and Processed Cheese Products".

5.3 Dry matter

According to FAO/WHO Standard B- (still to be developed).

ANNEX

Terminology for the Classification of Cheeses

1. Definitions

1.1 "Cured or ripened cheese" is cheese which is not ready for consumption shortly after manufacture but which must be held for such time, at such temperature, and under such other conditions as will result in the necessary biochemical and physical changes characterizing the cheese.

1.2 "Mould cured or mould ripened cheese" is a cured cheese in which the curing has been accomplished primarily by the development of characteristic mould growth throughout the interior and/or on the surface of the cheese.

1.3 "Uncured, unripened, or fresh cheese" is cheese which is ready for consumption shortly after manufacture.

2. Classification of cheese according to firmness, fat content and principal curing characteristics

The following classification shall be applicable to all cheeses covered by this standard. However, this classification shall not preclude the designation of more specific requirements in individual cheese standards.

	Term I		Term II	Term III
If the MFFB* is %	The first phrase in the designation shall be	If the FDB** is %	The second phrase in the designation shall be	Designation according to principal curing characteristics
< 51	Extra hard	> 60	High fat	1. Cured or ripened
49-56	Hard	45-60	Pull fat	a. mainly surface
54-63	Semi-hard	25-45	Medium fat	b. mainly interior
61-69	Semi-soft	10-25	Low fat	2. Mould cured or ripened
> 67	Soft	<10	Skim	a. mainly surface
				b. mainly interior
				3. Uncured or unripened

* MFFB equals percentage moisture on a fat-free basis, i.e.

$$\frac{\text{Weight of moisture in the cheese}}{\text{Total weight of cheese - weight of fat in the cheese}} \times 100$$

** FDB equals percentage fat on the dry basis, i.e.

$$\frac{\text{Fat content of the cheese}}{\text{Total weight of cheese - weight of moisture in the cheese}} \times 100$$

Example: The description of a cheese with moisture on a fat-free basis of 57% and fat on a dry basis of 53% which is cured in a manner similar to that in which roquefort is cured would be.

Semi-hard	Full fat	Interior mould cured cheese
(Term I)	(Term II)	(Term III)

CX 5/70-19th Session
APPENDIX III-A

Submitted to Governments for acceptance

Standard A-8(a)
Step 7

RECOMMENDED GENERAL STANDARD FOR NAMED VARIETY PROCESS(ED) CHEESE AND SPREADABLE PROCESS(ED) CHEESE

1. DEFINITION

Named variety process(ed) cheese and spreadable process(ed) cheese is made by grinding, mixing, melting and emulsifying with the aid of heat and emulsifying agents one or more varieties of cheese, with or without the addition of foodstuffs in accordance with para 2.

2. OPTIONAL INGREDIENTS

2.1 Cream, butter and butteroil may be added in quantities to ensure compliance with the minimum fat requirements.

2.2 Salt (sodium chloride).

2.3 Vinegar.

2.4 Spices and other vegetable seasonings in sufficient quantity to characterize the product.

2.5 For the purpose of flavouring the product, foods other than sugars, properly cooked or otherwise prepared, may be added in sufficient quantity to characterize the product provided these additions, calculated on the basis of dry matter, do not exceed one sixth of the weight of the total solids of the final product.

2.6 Cultures of harmless bacteria and enzymes.

3. FOOD ADDITIVES

		Maximum level in the final product
3.1	Emulsifiers	
	Sodium, sodium-aluminium, potassium and calcium salts of the mono-, di- and polyphosphoric acids Sodium, potassium and calcium salts of citric acid Citric acid and/or phosphoric acid with sodium hydrogen carbonate and/or calcium carbonate	40 g/kg singly or in combination, calculated as anhydrous substances, except that added phosphorus compounds should not exceed 9 g/kg, calculated as phosphorus
3.2	Acidifiers/pH controlling agents	
	Citric acid	
	Phosphoric acid	
	Acetic acid	
	Lactic acid	
	Sodium hydrogen carbonate and/or calcium carbonate	
3.3	Colours	
	Annatto [1/]	

	Beta-carotene Chlorophyll incl. copper chlorophyll (CI No. 75810) Riboflavin Oleoresin of paprika Curcumin [1/]	Limited by Good manufacturing Practices (GMP)
3.4	Preservatives	
3.4.1	Either sorbic acid and its sodium and potassium salts, or Propionic acid and its sodium and calcium salts	3000 mg/kg singly or in combination expressed as the acids
3.4.2	Nisin	12.5 mg of pure nisin per kg

[1/] Temporarily endorsed by the Codex Committee on Food Additives (CCFA).

4. HEAT TREATMENT

During their manufacture, products conforming to the definition of the standard shall be heated throughout to a temperature of 70° C for 30 seconds, or any other equivalent time/ temperature combination.

5. COMPOSITION AND DESIGNATION

5.1 Designation

5.1.1 When a variety name is used to describe Processed Cheese or Spredable Processed Cheese, the cheese blend from which the product is made must contain at least 75% of the cheese variety mentioned. The remaining cheese must be of similar type.

5.1.2 Where more variety names are used to describe a product, only those varieties may be used in the manufacture of the product.

5.1.3 In this connection, it should be noted that Gruyere and Emmental are interchangeable.

5.2 Composition of a Named Variety Process(ed) Cheese

5.2.1 The minimum milk fat content in the dry matter shall be not less than that prescribed in the international individual standard for the natural cheese of the variety mentioned and in the case where two or more varieties are mentioned not less than the arithmetic average of the fat contents in dry matter prescribed in the standards concerned.

5.2.2 The minimum dry matter content shall not be more than 4% lower than the minimum dry matter content prescribed in the international standard for the variety and in the case of two or more varieties shall not be more than 4% lower than the arithmetical average. Process(ed) Gruyere or Emmental cheese will be exempt from this requirement; in these cases the minimum dry matter content shall be 50%.

5.2.3 In the case of varieties for which no international standards exist the minimum dry matter content will be related to the fat in dry matter content as prescribed in the table below:

Milk Fat in Dry Matter %	Minimum Dry Matter %
65	53
60	52
55	51
50	50
45	48
40	46
35	44
30	42
25	40
20	38
15	37
10	36
leas than 10	34

If national legislation of the consuming country differs from the above, the national legislation prevails in the case of varieties for which no international standards exist.

5.3 Composition of a Named Variety Spreadable Process(ed) Cheese

5.3.1 The minimum milk fat content in the dry matter shall not be less than that prescribed for the variety in the international individual standard for the natural cheese.

5.3.2 The minimum dry matter content shall be in accordance with the following tablet

Milk Pat in Dry Matter %	Minimum Dry Matter %
65	45
60	44
55	44
50	43
45	41
40	39
35	36
30	33
25	31
20	29
15	29
10	29
less than 10	29

If national legislation of the consuming country differs from the above, the national legislation prevails in the case of varieties for which no international standards exist.

6. LABELLING

The following provisions in respect of the labelling of the products are subject to endorsement by the Codex Committee on Food Labelling. In addition to Sections 1, 2, 4 and 6 of the General Standard for the Labelling of Prepackaged Foods (Ref. No. CAC/RS 1-1969), the following specific provisions apply:

6.1 The Name of the Food *

* In some French and Spanish speaking countries the word "fromage" or "queso" (cheese) need not be included in the name of the product when a variety name is used to describe Process(ed) Cheese or Spreadable Process(ed) Cheese.

6.1.1 The name of a product made according to 5.1.1 shall be "Process(ed) _____Cheese" or "_____Process(ed) Cheese" or "Spreadable Process(ed) _____ Cheese" or "_____ Spreadable Process(ed) Cheese" (the blank being filled with the name of the variety of cheese used).

6.1.2 The name of a product made according to 5.1.2 shall be "Process(ed) _____ and _____ Cheese" or " and_____ Process(ed) Cheese" or "Spreadable Process(ed) and_____ Cheese" or "_____ and_____ Spreadable Process(ed) Cheese", in descending order of proportion.

6.1.3 In case the named variety process(ed) cheese or the named variety spreadable process(ed) cheese includes apices according to 2.3 or natural foodstuffs, according to 2.4, the name of the product shall be the one applicable according to 6.1.1 and 6.1.2 followed by the term "with ___", the blank being filled with the common or usual name or names of the spices or natural foodstuffs used, in order of predominance by weight.

6.1.4 The milk fat content shall be declared as fat in the dry matter in multiples of 5% (the figures used being that of the 5% multiple immediately below the actual composition) and/or as percentage by mass. Process(ed) cheese or spreadable process(ed) cheese which carries the name of a single variety of cheese covered by an international individual, natural cheese standard is exempt from the declaration of the fat content.

6.2 List of Ingredients

A complete list of ingredients shall be declared on the label in descending order of proportion, in accordance with para 3.2(c) of the General Standard for the Labelling of Prepackaged Foods (Ref. No. CAC/RS 1-1969).

6.3 Net Contents

The net contents, except on individual portions not intended for separate sale, shall be declared by weight in either the metric ("Système international" units) or avoirdupois or both systems of measurement as required by the country in which the food is sold.

6.4 Name and Address

The name and address of the manufacturer, packer, distributor, importer, exporter or vendor of the product shall be declared, except on individual portions not intended for separate sale, in which case the declaration may be replaced by a trademark or other indication of the manufacturer, or importer, or seller.

6.5 Country of Manufacture

The name of the producing country shall be declared (for export only).

6.6 Date Marking

There shall be a clear indication of the minimum durability date.

6.7 Lot Identification

Each container shall be permanently marked in code or in clear to identify the producing factory and the lot.

7. METHODS OF SAMPLING AND ANALYSIS

7.1 Samplings according to FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products", paragraphs 2 and 7.

7.2 Fat Contents according to FAO/WHO Standard B-3 "Determination of the Pat Content of Cheese and Processed Cheese Products".

7.3 Phosphorus Contents according to FAO/WHO Standard B-12 "Determination of the Phosphorus Content of Cheese and Processed Cheese Products".

7.4 Citric Acid Contents according to FAO/WHO Standard B-13 "Determination of the Citric Acid Content of Cheese and Processed Cheese Products".

7.5 Dry Matter Contents in preparation.

CX 5/70-19th Session
APPENDIX III-B

Submitted to Governments for acceptance

Standard A-8(b)
Step 7

RECOMMENDED GENERAL STANDARD FOR "PROCESS(ED) CHEESE" AND "SPREADABLE PROCESS(ED) CHEESE"

1. DEFINITION

Process(ed) cheese and spreadable process(ed) cheese are made by grinding, mixing, malting and emulsifying with the aid of heat and emulsifying agents one or more varieties of cheese, with or without the addition of milk components and/or other foodstuffs in accordance with paragraph 2.

2. OPTIONAL INGREDIENTS

2.1 Cream, butter and butteroil may be added.

2.2 Other milk products may be added to a maximum of % lactose content in the final product.

2.3 Salt '(sodium chloride).

2.4 Vinegar.

2.5 Spices and other vegetable seasonings in sufficient quantity to characterize the product.

2.6 For the purpose of flavouring the product, foods other than sugars, properly cooked or otherwise prepared, may be added in sufficient quantity to characterize the product provided these additions, calculated on the basis of dry matter, do not exceed one sixth of the weight of the total solids of the final product.

2.7 Cultures of harmless bacteria and enzymes.

3. FOOD ADDITIVES

		Maximum level in the final product
3.1	Emulsifiers	
	Sodium, sodium-aluminium, potassium and calcium salts of the mono-, di- and polyphosphoric acids Sodium, potassium and calcium salts of citric acid Citric acid and/or phosphoric acid with sodium hydrogen carbonate and/or calcium carbonate	40 g/kg singly or in combination, calculated as anhydrous substances, except that added phosphorus compounds should not exceed 9 g/kg, calculated as phosphorus
3.2	Acidifiers/pH controlling agents	
	Citric acid	
	Phosphoric acid	
	Acetic acid	
	Lactic acid	
	Sodium hydrogen carbonate and/or calcium carbonate	
3.3	Colours	

	Annatto [1/] Beta-carotene Chlorophyll incl. copper chlorophyll (CI No. 75810) Riboflavin Oleoresin of paprika Curcumin [1/]	Limited by Good manufacturing Practices (GMP)
3.4	Preservatives	
3.4.1	Either sorbic acid end its sodium and potassium salts, or Propionic acid and its sodium and calcium salts	3000 mg/kg singly or in combination expressed as the acids
3 4.2	Nisin	12.5 mg of pure nisin per kg

[1/] Temporarily endorsed by the Codex Committee on Food Additives (CCFA).

4. HEAT TREATMENT

During their manufacture, products conforming to the definition of the standard shall be heated throughout to a temperature of 70° C for 30 seconds, or any other equivalent or greater time/temperature combination.

5. COMPOSITION AND DESIGNATION

5.1 Products conforming to this standard may not be designated by a cheese variety name in connection with the names "Process(ed) Cheese" or "Spreadable Process(ed) Cheese" but mention may be made on the label of the name of a cheese variety which gives a characteristic flavour to the product (e.g. process(ed) cheese with _____).

5.2 Process(ed) cheese and Spreadable Process(ed) Cheese shall have a minimum dry matter content related to the declared minimum milk fat in dry matter content, as follows:

Milk fat in dry matter %	Minimum Dry Matter % Process(ed) Cheese	Minimum Dry Matter % Spreadable Process(ed) Cheese
65	53	45
60	52	44
55	51	44
50	50	43
45	48	41
40	46	39
35	44	36
30	42	33
25	40	31
20	38	29
15	37	29
10	36	29
less than 10	34	29

6. LABELLING

The following provisions in respect of the labelling of the products are subject to endorsement by the Codex Committee on Food Labelling. In addition to Sections 1, 2, 4

and 6 of the General Standard for the Labelling of Prepackaged Foods (Ref. No. CAC/RS 1-1969), the following specific provisions apply:

6.1 The Name of the Product

6.1.1 The name of the product shall be Process(ed) Cheese or Spreadable Process(ed) Cheese as applicable.

6.1.2 In case the Process(ed) Cheese or Spreadable Process(ed) Cheese above includes spices according to 2.4 or natural foodstuffs, according to 2.5 the name of the product shall be the one applicable above followed by the term "with ", the blank being filled with the common or usual names of the spices or natural foodstuffs used, in order of predominance by weight.

6.1.3 The milk fat content shall be declared as fat in dry matter on the label in multiples of 5% (the figure used to be that of the 5% multiple below the actual composition) and/or as percentage by mass.

6.2 List of Ingredients

A complete list of ingredients shall be declared on the label in descending order of proportion, in accordance with para 3.2(c) of the General Standard for the Labelling of Prepackaged Foods (Ref. No. CAC/RS 1-1969).

6.3 Net Contents

The net contents, except on individual portions not intended for separate sale, shall be declared by weight in either the metric ("Système international" units) or avoirdupois or both systems of measurement as required by the country in which the food is sold.

6.4 Name and Address

The name and address of the manufacturer, packer, distributor, importer, exporter or vendor of the product shall be declared, except on individual portions not intended for separate sale, in which case the declaration may be replaced by a trademark or other indication of the manufacturer, or importer, or seller.

6.5 Country of Manufacture

The name of the producing country shall be declared (for export only).

6.6 Date Marking

There shall be a clear indication of the minimum durability date.

6.7 Lot Identification

Each container shall be permanently marked in code or in clear to identify the producing factory and the lot.

7. METHODS OF SAMPLING AND ANALYSIS

7.1 Sampling: according to FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products" paragraphs 2 and 7.

7.2 Fat Content: according to FAO/WHO Standard B-3 "Determination of the Fat Content of Cheese and Processed Cheese Products".

7.3 Phosphorus Content: according to FAO/WHO Standard B-12 "Determination of the Phosphorus Content of Cheese and Processed Cheese Products".

7.4 Citric Acid Content: according to FAO/WHO Standard B-13 "Determination of the Citric Acid Content of Cheese and Processed Cheese Products".

7.5 Dry Matter Content: (in preparation).

CX 5/70-19th Session
APPENDIX III-C

Submitted to Governments for acceptance

standard A-8(c)
Step 7

RECOMMENDED GENERAL STANDARD FOR PROCESSS(ED) CHEESE PREPARATION (PROCESS(ED) CHEESE FOOD AND PROCESS(ED) CHEESE SPREAD)

1. DEFINITION

Process(ed) cheese food or process(ed) cheese spread is made by grinding, mixing, melting and emulsifying with the aid of heat and emulsifying agents one or more varieties of cheese with any selection of ingredients or additives mentioned in paragraphs 2 and 3 below.

2. INGREDIENTS

2.1 Cream, butter, butteroil and other dairy products may be added.

2.2 Salt (sodium chloride)

2.3 Spices and other vegetable seasonings in sufficient quantity to characterize the product.

2.4 Vinegar.

2.5 For the purposes of flavouring, the products, foods properly cooked or otherwise prepared, may be added in sufficient quantity to characterize the product provided these additions, calculated on the basis of dry matter, do not exceed one sixth of the weight of the total solids of the final product.

2.6 Sugars (any carbohydrate sweetening matters).

2.7 Cultures of harmless bacteria and enzymes.

3. FOOD ADDITIVES

	Maximum level in the final product
3.1 Emulsifiers	
Sodium, sodium-aluminium, potassium and calcium salts of the mono-, di- and polyphosphoric acids Sodium, potassium and calcium salts of citric acid Citric acid and/or phosphoric acid with sodium hydrogen carbonate and/or calcium carbonate	40 g/kg singly or in combination, calculated as anhydrous substances, except that added phosphorus compounds should not exceed 9 g/kg, calculated as phosphorus
3.2 Acidifiers /pH controlling agents	
Citric acid Phosphoric acid Acetic acid Lactic acid Sodium hydrogen carbonate and/or calcium carbonate	
3.3 Colours	
Annatto [1/]	

	Beta-carotene Chlorophyll incl. copper chlorophyll (CI No. 75810) Riboflavin Oleoresin of paprika Curcumin [1/]	Limited by Good manufacturing Practices ((IMP)
3.4	Preservatives	
3.4.1	Either sorbic acid and its sodium and potassium salts, or Propionic acid and its sodium and calcium salts	3000 mg/kg singly or in combination expressed as the acids
3.4.2	Nisin	12.5 mg of pure nisin per kg
3.5	Taste intensifiers	
	Sodium glutamate	Limited by GMP
3.6	Other additives	
	Arabic gum [2/] Locust (carob) bean gum [2/] Karaya gum [2/] Guar gum [1/] Oat gum [2/] Tragacanth gum [2/] Agar-agar Carrageenan Sodium carboxymethylcellulose (cellulose gum) Sodium, potassium, calcium and ammonium salts of alginic acid Propylene glycol ester of alginic acid [1/] Pectins Gelatine	8 g/kg singly or in combination

[1/] Temporarily endorsed by the Codex Committee on Food Additives (CCFA)
[2/] Not (yet) endorsed by the CCFA.

4. HEAT TREATMENT

During their manufacture products conforming to the definition of the standard shall be heated throughout to a temperature of 70°C for 30 seconds, or any other equivalent or greater time/temperature combination.

5. COMPOSITION AND DESIGNATION

5.1 Products conforming to this standard may not be designated by a cheese variety name in connection with the name processed cheese preparation (process(ed) cheese food or cheese spread) but mention may be made of the name of a cheese variety on the label in close proximity to the label declarations required under paragraph 6.2.

5.2 Processed cheese preparations (process(ed) cheese food and process(ed) cheese spread) shall have a minimum dry matter content related to the declared minimum milk fat in dry matter, as follows:

Milk fat in dry matter %	Minimum Dry Matter %
65	45
60	44
55	44
50	43
45	41
40	39
35	36
30	33
25	31
20	29
15	29
10	29
less than 10	29

6. LABELLING

The following provisions in respect of the labelling of the products are subject to endorsement Toy the Codex Committee on Food Labelling. In addition to Sections 1, 2, 4 and 6 of the General Standard for the Labelling of Prepackaged Foods (Ref. No. CAC/RS 1-1969), the following specific provisions apply:

6.1 The Name of the Product

6.1.1 The name of the product shall be "Processed cheese preparation" or where national regulations distinguish between "process(ed) cheese food" and "process(ed) cheese spread", these names will apply.

6.1.2 In case the products include spices and natural foodstuffs as provided for under 2.3 and 2.4, the name of the product shall be the one applicable above followed by the term "with _____", the blank being filled in with the common or usual name or names of the spices or foodstuffs used, in order of predominance by weight.

6.1.3 The milk fat content shall be declared as fat in the dry matter in multiples of 5% (the figure used to be that of the 5% multiple below the actual composition) and/or as percentage by mass.

6.2 List of Ingredients

A complete list of ingredients shall be declared on the label in descending order of proportion, in accordance with para 3.2(c) of the General Standard for the Labelling of Prepackaged Foods (Ref. No. CAC/RS 1-1969).

6.3 Net Contents

The net contents, except on individual portions not intended for separate sale, shall be declared by weight in either the metric ("Système international" units) or avoirdupois or both systems of measurement as required by the country in which the food is sold.

6.4 Name and Address

The name and address of the manufacturer, distributor, importer, exporter or vendor of the product shall be declared, except on individual portions not intended for separate sale, in which case the declaration may be replaced by a trademark or other indication of the manufacturer, or importer or seller.

6.5 Country of Manufacture

The name of the producing country shall be declared (for export only).

6.6 Date Marking

There shall be a clear indication of the minimum durability date.

6.7 Lot Identification

Each container shall be permanently marked in code or in clear to identify the producing factory and the lot.

7. METHODS OF SAMPLING AND ANALYSIS

7.1 Samplings according to FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products", paragraphs 2 and 7.

7.2 Pat Contents according to FAO/WHO Standard B-3 "Determination of the Pat Content of Cheese and Processed Cheese Products".

7.3 Phosphorus Contents according to FAO/WHO Standard B-12 "Determination of the Phosphorus Content of Cheese and Processed Cheese Products".

7.4 Citric Acid Contents according to FAO/WHO Standard B-13 "Determination of the Citric Acid Content of Cheese and Processed Cheese Products".

7.5 Dry Matter Content (in preparation).

APPENDIX IV

Standard C-35

INTERNATIONAL STANDARD FOR EXTRA HARD GRATING CHEESE

1. DESIGNATION OF CHEESE

Extra Hard Grating.

2. DEPOSITING COUNTRY

United States of America.

3. RAW MATERIALS

3.1 Kind of milk: cow's milk, goat's milk or sheep's milk and mixtures of these milks.

3.2 Authorized additions:

3.2.1 Necessary additions:

- cultures of harmless lactic acid producing bacteria (starter)
- rennet or other suitable coagulating enzymes
- sodium chloride

3.2.2 Optional additions:

- calcium chloride, maximum 200 mg anhydrous/kg of milk used
- harmless flavour producing bacteria
- harmless enzymes to assist 'in flavour development (solids of preparation not to exceed 0.1% of weight of milk used)
- chlorophylls, including copper chlorophyll (Colour Index No. 75810)
- sorbic acid or its sodium or potassium salts, maximum 1000 mg/kg calculated as sorbic acid in the final product.

4. PRINCIPAL CHARACTERISTICS OF THE CHEESE READY FOR CONSUMPTION

4.1 Type:

4.1.1 Consistency: extra hard, suitable for grating

4.1.2 Age of cure: minimum age 6 months

4.2 Shape: various

4.3 Dimensions and Height: 4.3.1 Dimensions: various 4.3.2 Weights: various

4.4 Rind, where present:

4.4.1 Consistency: extra hard

4.4.2 Appearance: dry, may be coated with vegetable oil, food grade wax or plastic materials.

4.4.3 Colour: amber

4.5 Body:

4.5.1 Texture: granular, slightly brittle

4.5.2 Colour: natural uncoloured to light cream colour.

4.6 Holes (when holes are a typical characteristic of the variety).

4.6.1 Number: few

4.6.2 Shape: small, round

4.6.3 Size: approximately 1-2 mm

4.6.4 Appearance: characteristic gas holes

4.7 Minimum fat: 32% fat in dry matter.

4.8 Maximum moisture: 36%.

4.9 Brief description: extra hard, dry, slightly brittle, suitable for grating.

5. METHOD OF MANUFACTURE

5.1 Method of coagulating: rennet or other suitable coagulating enzymes; with the possible addition of lactic acid starter.

5.2 Heat treatments milk may be raw or pasteurized. If pasteurized the milk is heated to not less than 72°C (161°F) for 15 seconds.

5.3 Fermentation procedures lactic acid fermentation or other flavour producing cultures and enzymes.

5.4 Maturation procedures after the curd which may be lightly salted is shaped into forms, the cheese may be salted again in brine, dry salted or both} held in a cool and well aerated or temperature controlled room for not less than 6 months.

6. SAMPLING AND ANALYSIS

6.1 Samplings according to FAO/WHO Standard B.1 "Sampling Methods for Milk and Milk Products" para 7 - Sampling Cheese»

6.2 Determination of fat contents according to FAO/WHO Standard B.3 "Determination of fat Content of Cheese and Processed Cheese Products".

6.3 Determination of dry matter: (under elaboration).

7. MARKING AND LABELLING

7.1 Cheese conforming to this standard may be designated Extra Hard Grating Cheese or any recognized variety name in the consuming country. A "coined" or "fanciful" name, however, may be used provided it is not misleading and is accompanied by the phrase "Extra Hard Orating Cheese".

7.2 It shall be labelled in conformity with the appropriate sections of Article of the FAO/WHO Standard A-6 "General Standard for Cheese".

CX 5/70-19-th Session
APPENDIX V

PROPOSAL BY THE DELEGATION OF DENMARK

"Decision No. 6

In countries in which it is not prohibited (is permitted) to manufacture and/or sell for human consumption imitation products which, in appearance, characteristics and intended use, are similar to milk or to milk products standardized under the Code of Principles and in which one or more milk ingredients are wholly or partly replaced by non-milk ingredients, such products shall meet the same requirement as to the qualitative composition of the milk and standardized milk product, apart from the nature of the ingredient being replaced» Such products shall not contain other additives than those provided for in the milk product standards except such additives necessary for the replacement.

Such products shall further be produced under hygienic conditions and shall conform to the hygienic quality standards and to such maximum levels for contaminants normally applicable for the corresponding milk product. They shall be labelled according to Article 4 of the Code of Principles and in other respects in conformity with the FAO/WHO Recommended International General Standard for the Labelling of Prepackaged Foods and with appropriate sections of the standard for the milk product in question."

DRAFT CODE OF HYGIENIC PRACTICE FOR PRIED MILK

This draft has been prepared by the delegation of Australia oh the basis of the Revised Proposed Draft Code of Practice - General Principles of Food Hygiene (ALINORM 78/13A, Appendix V).
For the convenience of the reader, those portions of the Revised General Principle® of Food Hygiene which are applicable to this Code are written full. Sidelined portions indicate material which is particular to this Code of Hygienic Practice.

SECTION I - SCOPE

1. The Code of Practice applies to dried milk products as defined. It recommends general hygiene and technological practices for use in the handling (including the production, preparation, processing, packaging, storage, transport and distribution) of dried milk products for human consumption to ensure safe, sound and wholesome dried milk products.

SECTION II - DEFINITIONS

2. For the purposes of this Code the following expressions have the meaning stated:

2.1	Adequate	sufficient to accomplish the intended purpose of this Code.
2.2	Cleaning	the removal of food residues, soil, dirt, grease or other objectionable matter.
2.3	Contamination	the addition of any objectionable matter, directly or indirectly, to the product or the presence of any such matter in the product. Contamination includes infestation by pests.
2.4	Disinfection	the reduction, without adversely affecting the food by means of hygienically satisfactory chemical agents and/or physical methods, of the number of micro-organisms to a level that will not lead to harmful contamination of food.
2.5	Dried milk	roller dried or spray dried milk products or composite milk products as defined in Articles 2 and 3 respectively of the Code of Principles concerning Milk and Milk Products, Seventh Edition (CAC/M 1-1973).
2.6	Establishment	any building(s) or area(s) in which dried milk products are prepared, processed, handled, packed or stored and the surroundings under the control of the same management.
2.7	Food handling	any operation in the production, preparation, processing, packaging, storage, transport and distribution and sale of food.
2.8	Liquid milk products	except for milk, the raw materials from which dried milk products are prepared, including intermediate evaporated or concentrated products used in the process of preparing dried milk products.
2.9	Pasteurization	heating:

		(i) milk, skimmed milk or whey to a minimum temperature of 72°C for 15 seconds; (ii) milk products which have a higher milk fat content than milk and/or contain added sweeteners to at least 72°C for 15 seconds; (iii) concentrated milk and concentrated milk products to at least 80 C for 25 seconds; or treating at a time/temperature relationship sufficient to ensure equivalent destruction of micro-organisms
2.10	Pests	any animals capable of directly or indirectly contaminating food.
2.11	Protective clothing	special garments intended to prevent the contamination of dried milk products and used as outer wear by persons in an establishment and includes head coverings and footwear.

SECTION III - HYGIENIC REQUIREMENTS IN PRODUCTION AREA

> Hygienic considerations in regard to milking practices are not covered in this Code.
>
> For Raw Material Requirements, see Section VII of this Code.

SECTION IV - ESTABLISHMENT: DESIGN AND FACILITIES

4.1 Location

Establishments should be located, in areas which are free from objectionable odours, smoke, dust or other contaminants and are not subject to flooding.

4.2 Roadways and Yards

Roadways and yards serving the establishment and which are within its boundaries or in its immediate vicinity should have a hard paved surface suitable for wheeled traffic. There should be adequate drainage and provision should be made to allow for cleaning.

4.3 Buildings and facilities

4.3.1 Buildings and facilities should be of sound construction and maintained in good repair.

4.3.2 Adequate working space should be provided to allow for satisfactory performance of all operations.

4.3.3 The design should be such as to permit easy and adequate cleaning and to facilitate proper supervision of food hygiene.

4.3.4 The buildings and facilities should be designed to prevent the entrance and harbouring of pests and the entry of environmental contaminants such as smoke and dust.

4.3.5 Buildings and facilities should be designed to provide separation, by partition, location or other effective means, between those operations which may cause cross-contamination.

4.3.6 Buildings and facilities should be designed to secure hygienic operations by means of a regulated flow in the process from the arrival of the raw material at the premises to the finished product, and should provide for appropriate temperature conditions for the process and the product.

4.3.7 In food handling areas:

- Floors, where appropriate, should be of water-proof, non-absorbent, washable, non-slip and non-toxic materials, without crevices, and should be easy to clean and disinfect. Where appropriate, floors should slope sufficiently for liquids to drain to trapped outlets.
- Walls, where appropriate, should be of water-proof, non-absorbent, washable and non-toxic materials and should be light coloured. Up to a height appropriate for the operation they should be smooth and without crevices, and should be easy to clean and disinfect.
- Ceilings should be so designed, constructed and finished as to prevent the accumulation of dirt and minimize condensation, mould development and flaking, and should be easy to clean»
- Windows and other openings should be so constructed as to avoid accumulation of dirt and those which open should be fitted with screens. Screens should be easily movable for cleaning and kept in good repair. Internal window sills, if present, should be sloped to prevent use as shelves. In rooms where products may be exposed or where there are air inlets to dryers and other equipment, windows should remain closed whenever such equipment is in use.
- Doors should have smooth, non-absorbent surfaces, and, where appropriate, be self-closing and close fitting.
- Stairs, lift cages and auxiliary structures such as platforms, ladders, chutes, should be so situated and constructed as not to cause contamination to food. Chutes should be constructed with inspection and cleaning hatches.

4.3.8 In food handling areas all overhead structures and fittings should be installed in such a manner as to avoid contamination directly or indirectly of food and raw materials by condensation and drip, and should not hamper cleaning operations. They should be insulated where appropriate and be so designed and finished as to prevent the accumulation of dirt and to minimize condensation, mould development and flaking. They should be easy to clean.

4.3.9 Living quarters and toilets should be completely separated from and should not open directly on to food handling areas.

4.3.10 Where appropriate, establishments should have facilities for control of access.

4.3.11 The use of material which cannot be adequately cleaned and disinfected, such as wood, should be avoided, unless its use would clearly not be a source of contamination.

4.3.12 Laboratory facilities should be readily available.

4.4 Sanitary Facilities

4.4.1 Water Supply

4.4.1.1 An ample supply of potable water under adequate pressure should be available with adequate facilities for its storage where necessary and distribution, and with adequate protection against contamination and pollution. The standard of potability should not be less than those contained in the latest edition of "International Standards of Drinking Water" (WHO).

4.4.1.2 Hot potable water should be in adequate supply at all times during the working day.

4.4.1.3 Non-potable water should be carried in completely separate lines, identifiable preferably by colour, and with no cross-connection with or back-siphonage into the system carrying potable water. It should not be possible to connect lines carrying non-potable water to any equipment or cleaning-disinfection apparatus used in handling food. The facilities for non-potable water should be approved by the official agency having jurisdiction.

4.4.2 Steam

4.4.2.1 An adequate supply of steam, or other heating medium, should be provided to ensure satisfactory operation of all heat treatment, evaporating and drying equipment during the production of dried milk products, and also provide the necessary heat for cleaning, disinfection and other operations.

4.4.2.2 Steam used in direct contact with food or food contact surfaces should contain no substances (including volatile boiler water compounds) which may be hazardous to health or may contaminate the food.

4.4.3 Refrigeration

Sufficient refrigeration capacity should be available to chill and maintain raw and pasteurized milk and liquid milk products at a temperature sufficiently low to ensure no adverse effect on the hygienic quality of the product. A maximum temperature of 4°C is considered desirable in this regard»

4.4.4 Air

An adequate supply of air should be provided for the drying, conveying, cooling or air-sweeping of the product. Such air should be drawn from a source which is free from contamination such as odours, smoke, dust or dirt. Precautions should be taken to remove oil, moisture, dirt or odours from such air. Compressed air which comes into contact with milk products or product contact surfaces should also conform to these requirements.

4.4.5 Effluent and waste disposal

Establishments should have an efficient effluent and waste disposal system which should at all times be maintained in good order and repair. All effluent lines (including sewer systems) should be large enough to carry peak loads and should be so constructed as to avoid contamination of potable water supplies»

4.4.6 Changing facilities and toilets

Adequate, suitable, and conveniently located changing facilities and toilets should be provided in all establishments. Toilets should be so designed as to ensure hygienic removal of waste matter. These areas should be well lit, ventilated and where appropriate heated and should not open directly onto food handling areas. Hand washing facilities with warm or hot and cold water, a suitable hand cleansing facility and with suitable hygienic means of hand drying should be provided adjacent to toilets;

where paper towels are used, a sufficient number of dispensers and receptacles should be provided near to each washing facility. Taps of a non-hand operable type are desirable. Notices should be posted directing personnel to wash their hands after using the toilet.

4.4.7 Hand washing facilities in processing areas

Adequate and conveniently located facilities for hand washing and drying should be provided wherever the process demands. Where appropriate, facilities for hand disinfection should also be provided. Harm or hot and cold water and a suitable hand-cleaning preparation should be provided. There (should be suitable hygienic means of drying hands. Where paper towels are used, a sufficient number of dispensers and receptacles should be provided adjacent to each washing facility. The facilities should be furnished with waste pipes leading to drains.

4.4.8 Disinfection facilities

Where appropriate, adequate facilities for cleaning and disinfection of working implements and equipment should be provided. These facilities should be constructed of corrosion-resistant materials, capable of being easily cleaned, and should be fitted with suitable means of supplying warm and cold water in sufficient quantities.

4.4.9 Lighting

Adequate natural or artificial lighting which does not alter colours should be provided throughout the establishment. Where appropriate, the intensity should not be less than:

540 lux (50 foot candles) at all inspection points

220 lux (20 foot candles) in work rooms

110 lux (10 foot candles) in other areas

Light bulbs and fixtures suspended over food materials in any stage of production should be of a safety type and protected to prevent contamination of food in case of breakage.

4.4.10 Ventilation

Adequate ventilation should be provided to prevent excessive heat, steam, condensation and dust and to remove contaminated air. The direction of the air flow should never be from a dirty area to a clean area. Ventilation openings should be provided with a screen or-other protecting enclosure of non-corrodible materials Screens should be easily removable) for cleanings

4.4.11 Facilities for storage and disposal of waste and inedible material

Facilities should be provided for the storage of waste (and inedible material prior to removal from the (establishment. These facilities should be designed to prevent access to waste or inedible material by pests and to avoid contamination of food, potable water, equipments buildings or roadways.4.5

Equipment and Utensils

4.5.1 Materials

All equipment and utensils used in food handling areas and which may contact food ¡should be made of material which does not transmit toxic substances, odour or taste, is non-absorbent , is resistant to corrosion and is capable of withstanding repeated cleaning and disinfection. Surfaces should be smooth and free from pits and crevices.

The use of wood and other materials which cannot be adequately cleaned and disinfected should be avoided except when their use would clearly not be a source of contamination.

4.5.2 Sanitary design, construction and installation

4.5.2.1 All equipment and utensils should be so designed and constructed an to prevent hygienic hazards and permit easy and thorough cleaning and disinfection and where practicable be visible for inspection. Stationary equipment should be installed in such a manner as to permit easy access and thorough cleaning. The use of different materials in such a way that contact corrosion can occur should be avoided.

Equipment should be designed to minimize build-up of moisture or dried product in dryers, lines, bins and packaging equipment.

4.5.2.2 Containers for inedible material and waste should be leak proof, constructed of metal or other suitable impervious material which should be easy to clean and be fitted with close-fitting lids.

4.5.2.3 The plant for pasteurizing or pre-heating of milk and liquid milk products should be provided with a thermometer and an automatic temperature recorder, a flow diversion valve or pump "cut out" as well as a positive pump or timing device to ensure that the proper time/temperature combination for pasteurization is maintained.

The following equipment should he used in association with the flow diversion valve or pump cut out:

(i) A device which will automatically shut off the steam supply to the evaporator when the flow diversion valve in the pre-heating section moves to the divert position.

(ii) A means whereby clean water or condensate may be introduced to the product side of the evaporator automatically when a diversion occurs in the pre-heating section.

(iii) The incorporation of a vacuum break device on evaporators which are fitted with spray condensers to prevent water being sucked back into the evaporator on emergency shutdown*

4.5.2.4 Instruments should be so positioned as to indicate the temperature of the milk or milk products on the completion of the holding section of the pasteurizing or pre-heating process.

4.5.2.5 Facilities for the convenient withdrawal of samples for the purpose of control of effective pasteurizing or heat-treatment should be provided,

4.5.2.6 Refrigeration equipment should be equipped with thermometers.

4.5.3 Thermometers

4.5.3.1 Thermometers which include glass in their construction should not be used in any application where glass may come into contact with milk or milk products»

4.5.3.2 Thermometers, temperature recorders and similar instruments should be calibrated against a reference instrument upon installation and periodically at adequate intervals to ensure efficient operation.

4.5.4 Spray dryers

4.5.4.1 Spray dryers should be equipped with adequate air intake filters. Air which is drawn into the dryer should comply with the requirements of Section 4.4.4. In direct gas-fired dryers, precautions should be taken to prevent contamination of the product.

4.5.4.2 Exhaust air from dryers should be treated to remove milk solids which may otherwise contaminate factory buildings and surroundings.

4.5.5 Equipment identification

Equipment and utensils used for inedible or discarded materials should be so identified and should not be used for edible products.

SECTION Y - ESTABLISHMENT; HYGIENIC REQUIREMENTS

5.1 Maintenance

The buildings, equipment, utensils and all other physical facilities of the establishment, including drains, should be maintained in good repair and in an orderly condition. As far as practicable, rooms should be kept free from steam, vapour and surplus. Storage rooms should be kept dry.

Special attention should be paid to the maintenance of roofs, guttering and drainage in the area surrounding the exhausts of dryers to prevent the accumulation of milk solids and the subsequent contamination of the area.

5.2 Cleaning and Disinfection

5.2.1 Cleaning and disinfection should meet the requirements of this Code. For further information on cleaning and disinfect ion procedures see Annex 1 to the Recommended Code of Practice - Revised General Principles of Food Hygiene (ALINORM 78/13A, Appendix V).

5.2.2 To prevent contamination of food, all equipment and utensils should be cleaned as frequently as necessary and disinfected whenever circumstances demand.

All wet product contact surfaces should be cleaned immediately after use. Dry product contact surfaces should be dry-cleaned by a technique appropriate to the equipment concerned immediately after use, and should be wet-cleaned as often as is necessary to maintain the quality of the end product. Where necessary equipment should be disassembled for cleaning. Chambers of the spray dryers should preferably be equipped with automatic cleaning devices.

5.2.3 Steel wool or metal sponges should not be used in the cleaning of dairy equipment or utensils.

5.2.4 Equipment and pipelines which are cleaned in place should first be rinsed with water at a temperature of 40°to 45°C to remove product residues. Spray nozzles should be examined periodically to ensure efficient distribution of detergent and disinfectant.

5.2.5 Equipment and utensils should be disinfected immediately before use, by the use of steam, hot water or chemical agents appropriate to the equipment concerned. Where chemical agents are used, the equipment shall be drained and then rinsed with cold, clean, potable water.

5.2.6 Special clean protective clothing and shoe covers should be used by any person entering the chamber of the spray dryer for the purpose of cleaning or maintenance.

5.2.7 Adequate precautions should be taken to prevent food from being contaminated during cleaning or disinfection of rooms, equipment or utensils by water and detergents or by disinfectants and their solutions. Detergents and disinfectants should be suitable for the purpose intended and should conform to public health requirements. Any residues of these agents on a surface which may come in contact with food should be removed by thorough rinsing with potable water before commencing work.

5.2.8 Either immediately after cessation of work for the day or at such other times as may be appropriate, floors, including drains, auxiliary (structures and walls of food handling areas should be thoroughly cleaned.

5.2.9 Changing facilities and toilets should be kept clean at all times,,

5.2.10 Roadways and yards in the immediate vicinity of and serving the premises should be kept clean.

5.2.11 Detergents and disinfectants should be stored in covered, labelled containers in a dry place.

5.3 Hygiene Control Programme

A permanent cleaning and disinfection schedule should be drawn up for each establishment to ensure that all areas are appropriately cleaned and that critical areas,, equipment and material are designated for special attention» A single individual who should be a permanent member of the staff of the establishment and whose duties should The divorced from production, should be appointed to be responsible for the cleanliness of the establishment,. He should have a thorough understanding of the significance of contamination and the hazards involved. All cleaning personnel should be adequately trained in cleaning techniques.

5.4 Storage and Disposal of Waste

Waste material should be handled in such a manner as to avoid contamination of food or potable water. Care should be taken to prevent access to waste by pests. Waste should be removed from the food handling and other working areas as often as necessary and at least daily. Immediately after disposal of the waste, receptacles used for storage and any equipment which has come into contact with the waste should be cleaned and disinfected. The waste storage area should also be cleaned and disinfected.

5.5 Exclusion of Domestic Animals

Dogs, cats and other domestic animals should be excluded from establishments.

5.6 Pest Control

5.6.1 There should be an effective and continuous programme for the control of pests. Establishments and surrounding areas should be regularly examined for evidence of infestation.

5.6.2 Should pests gain entrance to the establishment, eradication measures should "be instituted. Control measures involving treatment with chemical, physical or biological agents should only be undertaken by or under direct supervision of personnel who have a thorough understanding of the potential hazards to health resulting from the use of these agents, including those which may arise from residues retained in the product® Such measures should only be carried out in accordance with the recommendations of the official agency having jurisdiction.

5.6.3 Pesticides should only be used if other precautionary measures cannot be used effectively. Before pesticides are applied, care should be taken to safeguard all food, equipment and. utensils from contamination. After application, contaminated equipment and utensils should be thoroughly cleaned to remove residues prior to being used again.

5.7 Storage of Hazardous Substances

5.7.1 Pesticides or other substances which may represent a hazard to health should be labelled with a warning about their toxicity and use. They should be stored in looked rooms or cabinets used only for that purpose and dispensed and handled only by authorized and properly trained personnel or by persons under strict supervision of trained personnel. Extreme care should be taken to avoid contaminating food.

5.7.2 Except when necessary for hygienic or processing purposes, no substance which could contaminate food should be used or stored in food handling areas.

5.8 Personal Effects and Clothing

Personal effects and clothing should not he deposited in processing areas.

SECTION VI - PERSONNEL: HYGIEN AND HEALTH REQUIREMENTS

6.1 Hygiene Training

Managers of establishments should arrange for adequate and continuing training of every food handler in hygienic handling of food and in personal hygiene so that they understand the precautions necessary to prevent contamination of food. Instruction should include relevant parts of this Code.

6.2 Medical Examination

Persons who come in contact with food in the course of their work should have a medical examination prior to their employment if the official agency having jurisdiction, acting on medical advice, considers that this is necessary, either because of epidemiological considerations, the nature of the food prepared in a particular establishment or the medical history of the prospective food handler. Medical examination of a food handler should be carried out at other times when clinically or epidemiologically indicated.

6.3 Communicable Diseases

The management should take care to ensure that no person, while known or suspected to be suffering from, or to be a carrier of a disease likely to he transmitted through food or while afflicted with infected wounds, skin infections, sores or with diarrhoea, is permitted to work in any food handling area in any capacity in which there is any likelihood of such a person directly or indirectly contaminating food with pathogenic micro-organisms. Any person so affected should immediately report to the management that he is ill.

6.4 Injuries

Any person who has a out or wound should not continue to handle food or food contact surfaces until the injury is completely protected by a waterproof covering which is firmly secured, and which is conspicuous in colour. Adequate first-aid facilities should be provided for this purpose.

6.5 Washing of Hands

Every person engaged in a food handling area should wash his hands frequently and thoroughly with soap or other detergent under running warm, potable water while on duty. Hands should always be washed before commencing work, immediately after using the toilet, after handling contaminated material and whenever else necessary. After handling any material which might be capable of transmitting disease, hands should be washed and disinfected immediately. Notices requiring hand-washing should be displayed. There should be adequate supervision to ensure compliance with this requirement.

6.6 Personal Cleanliness

Every person engaged in a food handling area should maintain a high degree of personal cleanliness while on duty, and should at all times while so engaged wear suitable protective clothing including head covering and footwear, all of which articles should be cleanable unless designed to he disposed of and should be maintained in a clean condition consistent with the nature of the work in which the person is engaged. Aprons and similar items should not be washed on the floor.

6.7 Personal Behaviour

Any behaviour which could result in contamination of food, such as eating, use of tobacco, chewing (eg.: gum, sticks, betel nuts, etc.) or unhygienic practices such as spitting, should be prohibited in food handling areas.

6.8 Gloves

Gloves, if used in the handling of food products, should be maintained in a sound, clean and sanitary condition. The wearing of gloves does not exempt the operator from having thoroughly washed hands. Cloves should he made of an impermeable material except where their usage would be inappropriate or incompatible with the work involved.

6.9 Visitors

Precautions should be taken to prevent visitors to food handling areas from contaminating food. These may include the use of protective clothing. Visitors should observe the provisions recommended in paragraphs 5.8 to 6.8.

6.10 Supervision

Responsibility for ensuring compliance with all requirements of paragraphs 5.8-6.9 inclusive should be specifically allocated to competent supervisory personnel.

SECTION VII - ESTABLISHMENT; HYGIENIC PROCESSING REQUIREMENTS

7.1 Raw Material Requirements

7.1.1 All milk used in the manufacture of dried milk products should have been produced under hygienic conditions in compliance with the provisions of the official agency having jurisdiction.

7.1.2 No milk or liquid milk product should be accepted for processing unless it is suitable for human consumption and has not been contaminated, processed, handled, or subjected to the addition of any harmful substance which renders it unfit for human consumption. It should be unadulterated and be of good hygienic quality, odour and appearance.

7.1.3 No milk or liquid milk product should be accepted by an establishment unless it has been derived from healthy animals. Milk from animals which have been treated with antibiotics and other drugs should be excluded for a period adequate to prevent contamination of the milk.

7.1.4 Tests should be carried out on incoming milk and milk products to ensure that unsatisfactory raw materials are withheld from processing.

7.1.5 Where necessary, laboratory tests should be made of the ingredients prior to their use.

7.1.6 Raw materials and ingredients stored on the premises of the establishment should be maintained under conditions that will prevent spoilage, protect against contamination and minimize damage. Stocks of raw materials and ingredients should be properly rotated.

7.2 Prevention of Cross-Contamination

7.2.1 Effective measures should be taken to prevent contamination of pasteurized products by direct or indirect contact with material at an earlier stage of the process.

7.2.2 Persons who have come into contact with raw milk or other raw materials should not handle any product which has been pasteurized unless and until they discard all protective clothing which may have been contaminated with raw materials.

7.2.3 If there is a likelihood of contamination, hands should be washed thoroughly between handling products at different stages of processing.

7.2.4 All equipment which has been in contact with raw materials or contaminated material should be thoroughly cleaned and disinfected before being used for contact with pasteurized products.

7.2.5 Every department in which any dried milk product is prepared, processed or stored should be used at that time only for that purpose or for the preparation of other dried milk products or products subject to the same hygienic requirements. If the department is used for processing of products requiring lesser hygienic standards the arrangements should be such that there is no resultant contamination of the dried milk products subject to more stringent hygienic requirements.

7.3 Use of Water

7.3.1 As a general principle only potable water as defined in the latest edition of "International Standards of Drinking Water" (WHO) should be used in food handling.

7.3.2 Non-potable water may be used with the acceptance of the official agency having jurisdiction for steam production, refrigeration, fire control and other similar purposes not connected with food. However, non-potable water may, with specific acceptance by the official agency having jurisdiction, be used in certain food handling areas provided this does not constitute a hazard to health.

7.3.3 Water recirculated for re-use within an establishment should be treated and maintained in a condition so that no health hazard can result from its use. The treatment process should be kept under constant surveillance. Alternatively, recirculated water which has received no further treatment may be used in conditions where its use would not constitute a health hazard and will not contaminate either the raw material or the end product. Recirculated water should have a separate distribution system which can be readily identified. The acceptance of the official agency having jurisdiction should be

required for any treatment process and for the use of recirculated water in any food process.

7.4 Processing

7.4.1 Processing should be supervised by technically competent personnel.

7.4.2 All steps in the production process, including packaging, should be performed without unnecessary delay and under conditions which will prevent the possibility of contamination, deterioration, or the development of pathogenic and spoilage micro-organisms.

7.4.3 After inspection and testing, incoming milk or liquid milk products should be processed quickly or, if this is not possible, cooled to [4°C] or less and held at this temperature until processing. Milk which is in cans should be transferred to bulk holding tanks without delay.

7.4.4 Adequate heat-treatment facilities should be provided. All milk and liquid milk products should be pasteurized prior to concentrating.

7.4.5 The concentrated product should be dried as soon as possible after production to avoid contamination, deterioration or spoilage by the growth of micro-organisms and the production of toxins during the holding period,

7.4.6 The concentrated product leaving the evaporator should be fed directly to the dryer. If this is not possible for technical reasons it should be stored under such conditions of time and temperature as will prevent development of micro-organisms and toxins during storage. Twin feed-balance tanks should be used alternatively and all feed-balance tanks should be cleaned and sterilized at intervals not exceeding two hours.

7.4.7 Concentrated products may be transported to the drying plant, provided that they are further heat-treated prior to drying, at a minimum temperature of 75°C for 15 seconds, or a heat-treatment equivalent in destruction of micro-organisms.

7.4.8 It should be recognized that the heating of the concentrated product for technological reasons may reduce viable microbiological numbers but may not remove some microbiological toxins.

7.4.9 A continuous chart recording should be made of all pasteurization and heat-treatment steps, and these charts should be dated and kept available for inspection for a period of at least 1 year.

7.4.10 Dried milk products should be continuously removed from the drying chamber or rollers. Immediately following removal from the dryer the dry product should be cooled to a temperature not exceeding 44 C, except in the case of intermediate storage in bulk bins where the temperature of the dry product should not exceed 40°C, and should preferably be reduced to approximately 35°C.

7.4.11 When breakdowns or unplanned discontinuities in processing occur which disrupt the normal flow of the product, the batch should not be released for human consumption unless special precautions are taken to ensure acceptable hygienic quality of the batch. Re-processing, diversion to non-human use or additional testing may be required.

7.4.12 Dried milk products recovered from equipment which is not obtained as part of the normal continuous process should not be included in the end-product. This includes powder from dust collectors and hand-swept from dryers.

7.5 Packaging

7.5.1 All packaging material should be stored in a clean and sanitary manner. The material should be appropriate for the product to be packed and for the expected conditions of storage and should not transmit to the product objectionable substances beyond the limits acceptable to the official agency having jurisdiction. The packaging material should be sound and should provide appropriate protection from contamination.

7.5.2 Product containers should not have been used for any purpose which may lead to contamination of the product. Where practicable containers should be inspected immediately before use to ensure that they are in a satisfactory condition and where necessary cleaned and/or disinfected; when washed they should be well drained before filling. Only packaging material required for immediate use should be kept in the packing or filling area.

7.5.3 Precaution should be taken to minimise product dust and spillage. The packages should be closed immediately after filling or gassing, and the exteriors should be brushed or cleaned where necessary to remove any product dust.

7.5.4 Packaging should be done under conditions that preclude the introduction of contamination into the product.

7.5.5 Product coding

Products sold or otherwise distributed from a manufacturing, processing, packing, or repacking establishment should be coded to enable identification of lots and when necessary, segregation of specific food lots which may have become contaminated or otherwise unfit for their intended use. Records, adequate to identify the processing history of each lot, should be retained for a period that exceeds the shelf life of the product, except that unless a specific need exists they need not be retained more than two years.

7.6 Storage and Transport of the End Product

7.6.1 The end product should be stored and transported under such conditions as will preclude contamination with and/or proliferation of micro-organisms and protect against deterioration of the product or damage to the container.

7.6.2 Storage should be in such a manner and in such containers as to prevent moisture absorption. Dried milk products should not be stored on the floor. During storage, periodic inspection of the product should take place to ensure that only food which is fit for human consumption is despatched and that end-product specifications should be complied with. The product should be despatched in the sequence of lot numbers.

7.7 Sampling and Laboratory Control Procedures

7.7.1 The establishment should be provided with adequate laboratory facilities to guarantee products of approved quality and to carryout the testing routine needed to guarantee continuous and effective control of operations. specifications should be provided for raw materials, ingredients and ancillary materials (packaging, chemicals, etc.) and all testing and quality control procedures should be adequately documented.

7.7.2 The laboratory should monitor:

(i) Incoming milk and liquid milk products.
(ii) Ingredients.

(iii) Processing and manufacturing stages.
(iv) Cleaning and disinfection in the plant.
(v) Finished products.
(vi) Water quality.
(vii) Calibration of instruments, for example, gauges, thermometers, etc.
(viii) Packaging materials.
(ix) Air quality.

7.7.3 The laboratory should be of adequate size and construction and be well equipped and have suitable lighting. It should be staffed with adequately trained personnel.

7.7.4 Analytical procedures should preferably follow recognized or standard methods in order that the results may be readily interpreted.

7.7.5 Care should be taken that arrangements are provided for the hygienic factory control of the processes of manufacture. This should include phosphatase assays on pasteurized milk, liquid milk products, and coliforms, aerobic plate counts, salmonella tests and phosphatase assays on one sample of the finished product, representing each storage tank or batch. Monitoring of the microbiological quality of concentrate is desirable, particularly where there is external access to the concentrate balance tank. Testing for salmonella should be done within the confines of the establishment only when adequate precautions have been taken to ensure that no contamination of the product arising from the laboratory is possible.

7.7.6 if 7.7.5 is not possible, at least five samples should least taken from the daily out-put from each plant. The first sample should be taken immediately after the start of the run, another in the middle of the run and the final one before the plant is closed for cleaning, with two other samples at intermediate stages.

7.7.7 The results of the daily microbiological examinations should be consistently monitored and in the event of a significant deviation from the normal characteristics of the product occurring, appropriate action, including more detailed investigation, should be undertaken immediately.

7.7.8 The records of the microbiological examinations should be maintained at each plant, for a period of at least one year. It would also be appropriate to retain the records of microbiological examinations relating to the various manufacturing processes. All records should be available for inspection if so required. Means of identifying batches with samples should also be provided.

7.7.9 The person in charge of the quality control programme should have authority commensurate with the responsibilities associated with planning, coordinating, executing and maintaining the plant quality control programme and he should have a thorough understanding of the significance of contamination and the hazards involved.

SECTION VIII - END PRODUCT SPECIFICATIONS

8.1 General Requirements

Appropriate methods should be used for sampling and examination to determine the compliance with the following specifications:

A. The products should be, to the extent possible in good manufacturing practice, free from objectionable matter.

B. The products should be free from micro-organisms in amounts harmful to humans and should not contain any substances originating from micro-organisms in amounts which may represent a hazard to health.

C. The products should be free from chemical pollutants in amounts which may represent a hazard to health.

D. The products should comply with any requirements set forth by the Codex Alimentarius Commission on pesticide residues and food additives as contained in permitted lists of Codex Commodity standards, or should comply with the requirements on pesticide residues and food additives of the country in which the product will be sold.

8.2 Microbiological Specifications (See Annex I).

ANNEX I

DRAFT MICROBIOLOGICAL SPECIFICATIONS FOR DRIED MILK PRODUCTS

This draft proposal for microbiological specifications for dried milk products contains:

(1) Microbiological sampling plans and limits

(2) Number of field samples from a lot

(3) Sampling methods

(4) Reference methods for the detection of salmonellae, and for the enumeration of mesophilic aerobic bacteria and coliform bacteria

(5) Routine microbiological sampling plans and limits.

Note: This proposal does not apply to dried milk products intended for use by high risk populations such as infants and children, invalids and geriatrics. These are special dietary foods and therefore are outside the terms of reference of the Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products.

A Proposed Draft Code of Hygienic Practice for Foods for Infants and Children which contains microbiological specifications was advanced to Step 6 by the 1?th Session of the Commission (see ALINORM 78/13A, Appendix VII).

1. Sampling Plans and Microbiological Limits [1/]

[1/] These sampling plans and microbiological limits are intended for use in oases of dispute or investigation. Routine examination of dried milk products would usually require less extensive testing. See also Section 5.

Salmonellae: Salmonella organisms should not be recovered from any of *[*15*]* sample units examined when the test is carried out according to the method described [2/]. (n = 15, c = 0, m = 0).

[2/] The method described requires sample units of 25 grammes.

Mesophilic aerobic bacteria: Mesophilic aerobic bacteria should not be recovered from any of the five sample units examined when the test is carried out according to the method described in a number exceeding *[*200 000*]* per gramme, nor in a number exceeding *[*50 000*]* per gramme from three or more of the five sample units tested. (n=5, c=2, m=50 000, M = 200 000).

Coliform bacteria: Coliforms should not be recovered from any of the five sample units examined when the test is carried out according to the method described in a number exceeding *[*100*]* per gramme, nor in a number exceeding *[*10*]* per gramme from two or more of the five sample units examined. (n = 5, c = 1, m = 10, M = 100).

2. Number of Field Samples from a Lot [3/]

Take *[*15*]* [4/] field samples, all of which are used for detection of salmonellae, and select at random 5 of these field samples to be examined also for mesophilic aerobic bacteria and coliform bacteria.

[3/] A lot is a quantity of food produced under identical conditions, all packages of Which should bear a lot number that identifies the production during a particular time interval and usually from a particular "line" or other critical processing unit.

[4/] Routine sampling will require different numbers of field samples: see Section 5

3. Sampling Methods

For all dried milk products take field samples of at least *[*200*]* grammes.

Equipment: Sterile trier long enough to reach to the bottom of containers to be sampled. Sterile sample containers with tight closures, sterile spoon, alcohol lamp or other burner, cotton, clean cloth or towel and water pail.

Methods: For small packages, randomly take one unopened package for each of the field samples required. If the net weight of the package is less than 200 g, take as many unopened packages as required to make at least 200 g for each field sample. For larger containers, such as boxes, bags, etc., remove top layer with sterile spoon or other sterile implement, and with a sterile trier remove at least 3 cores from the centre, midway between the centre and the periphery, and from the periphery respectively. Aseptically transfer the cores to a sterile container. Samples should be stored in a refrigerated or cool place until analysis takes place.

4. Reference Methods

4.1 Detection of Salmonellae

Dried whole milk, dried skim milk, and similar products. The method is, that of the AOAC except that a 25 g sample is dispersed in 225 ml of water [1/]. Proceed as described in AOAC Methods, 12th Edition, 1975, 46.013.

[1/] A number of 25 g samples may be composited and proportional amounts of diluent may added.

4.2 Enumeration of mesophilic aerobic bacteria

Dried whole milk, dried skim milk, dried whey, and similar products. The method is the reference method of the International Dairy Federation; ref. FIL-IDF 49:1970.

4.3 Enumeration of coliform bacteria

Dried whole milk, dried skim milk, dried whey, and similar products. The method is the reference" method of the International Dairy Federation; ref. FIL-IDF 64:1971.

5. Routine Sampling Plans and Microbiological Limits

The following routine sampling plans and microbiological limits are intended for use by the manufacturer as an indicator that the good manufacturing processes described in this Code have generally been complied with: see Section 7.7.5 and Section 7.7.6 of the Code. Should any batch fail to pass these routine tests the action recommended in Section 7.7.7 of this Code should be taken. The sampling plans and microbiological limits in Section 1 of this Annex are appropriate for use in further investigations.

5.1 Field samples and sampling methods

For all dried milk products covered by this Code, take 5 field samples, each of *[*200*]* grammes from each lot or day's production (See also Sections 7.7.5 and 7.7.6 of this Code). Take samples in accordance with Section 3 of this Annex,

5.2 Methods

The reference methods given in Section 4 of this Annex may be used, except that a modified test for the presence or absence of coliform bacteria is preferred.

5.3 Sampling plans and microbiological limits

Five equal field samples taken from the batch should be comingled aseptically. The resulting composited sample should conform to the following limits:

Salmonellae: Salmonella should not be recovered from *[*100*]* grammes of the composited sample when the test is carried out according to the method described.

Coliform bacteria: Coliform bacteria should not be recovered from *[*0.1 g*]* of the composited sample when tested by an appropriate method.

Mesophilic aerobic bacteria: Mesophilic aerobic bacteria should not be recovered from the sample unit examined in a number exceeding *[*50 00*]* per gramme when the test is carried out by the method described.

IDF/ISO/AOAC Cooperation in the field of methods of sampling and analysis

1. Representatives of the IDF, ISO and AOAC met in Rome on 10 June 1978 to discuss progress on collaboration between IDF, ISO and AOAC especially in connection with analytical standards for the Code of Principles concerning Milk and Milk Products.

Present

Dr. R. Demeter (Chairman)	IDF
Dr. H. Kay	IDF
Mr. p. Staal	IDF
Dr. J.B. Roos	ISO
Mr. S. Boelsma	ISO
Mrs. M. Tuinstra-Lauwaars	AOAC
Dr. R.W. Weik [1/]	AOAC
Mr. T.L. Hall [1/]	Chairman, Committee of Government Experts
Mr. K.P. Andersen	1st Vice Chairman, Committee of Government Experts
Dr. F. Winkelmann [1/]	FAO
Mr. W.L. de Haas [1/]	FAO
Mr. G. Vos (Observer)	EEC

[1/] Present for part of the session only.

2. Joint IDF/ISO/AOAC standards submitted to the 19th Session of the Committee of Government Experts.

Submitted to the Committee at step (c)

2.1 Caseins and caseinates - Lactose

Submitted to the Committee at step (g) (after comments of governments on methods at step (d) as contained in MDS 78/12(a) were considered and revised texts were prepared:

2.2 Caseins and caseinates - Water

2.3 Rennet caseins and caseinates - Ash

2.4 Caseins - "Fixed" ash

2.5 Caseins and caseinates - Protein

2.6 Casein - Free acidity

Note - It is recommended to the Committee to delete in Standard A12, in clause 2.6 the following words: "extracted at 20°c"

2.7 Caseins - ph

2.8 Milk and milk products - Lactose in the presence of other reducing substances

2.9 Dried Milk - Titratable acidity

Submitted to Governments for acceptance at step (h)

2.10 Cheese - Nitrate and nitrite

2.11 Anhydrous milk fat - Peroxide value

2.12 Butter - Water, solids-non-fat (s.n.f.) and fat on the same test portion

3. Work is in progress on:

3.1 Milk and milk products - Water

3.2 Dried milk - Nitrate and nitrite

3.3 Cheese - Chloride (potentiometric method)

3.4 Milk fat - Detection of foreign fat

3.5 Casein and caseinates - Scorched particles and extraneous matter

3.6 Dairy products - Heavy metals

3.7 Milk - Protein (dye-binding method)

3.3 Milk and milk products - Fat (Roese-Gottlieb)

3.9 Mycotoxins

3.10 Butter - Water dispersion

3.11 Milk - Freezing point

3.12 Milk - Free fatty acids

3.13 Cheese and cheese products - Sorbic acid

3.14 Dried milk - Vitamin A

3.15 Cheese - Phosphorus (revision of Standard B-12)

3.16 Selection of samples (governments' comments received are still under consideration)

3.17 Sampling techniques (revision of Standard B-I)

3.18 Colony count

3.19 Coliforms

3.20 Psychrotrophs

3.21 Yeasts and moulds

3.22 Coagulase positive staphylococci (including thermonuclease test)

3.23 Milk and milk products - Pesticide residues - The existing IDF Standard 75 shall be considered as provisional. The joint IDF/ISO/AOAC group of experts is considering comments and new developments with the aim of submitting a new draft standard including PCBs and PBB.
Mr. W.L. de Haas (FAO) drew attention to the matter that the MRL for fat-soluble pesticides will be revised by the Codex Committee on Pesticide Residues and be expressed in the case of low-fat products, on a product rather than on fat-basis. The distinction would be made at the 8% fat-content level. Document E-Doc 89/1978 allows to express the results either on fat or on product basis. For low-

fat products (under 1% level) the expression on product basis is recommended. The standpoint of the mentioned Codex Committee will be transmitted to the Joint Group for consideration.

Other items under joint IDF/ISO/AOAC consideration

4.1 Dried milk - Lactic acid/lactates

4.2 Dried milk - Characterization according to heat treatment

4.3 Identification of rennet and of rennet substitutes

4.4 Repeatability and reproducibility

4.5 Fermented milks - identification and enumeration of characteristic micro-organisms

4.6 Analysis of edible ices (on request of the Codex Committee on Edible Ices)

5. Other business

5.1 IDF/ISO/AOAC will continue its work, irrespective of structural changes within the FAO/WHO Food Standards Programme of the Codex Alimentarius Commission.

6. The next meeting of the three organizations will be held during next autumn on the occasion of the IDF Permanent Committee of Commission E meeting (20 October 1978).

(6th Ed. of Code of Principles concerning Milk and Milk Products and Associated Standards CX 8/6-May 1968)

STATUS OF ACCEPTANCES

STANDARD A. 1 -BUTTER

The following countries have communicated their acceptance of this Standard as a minimum standard:

Australia
Belgium
Canada
Congo
Democratic
Kampuchea
Denmark
Ecuador
Ethiopia
Finland
Franca
Germany,
Fed. Rep.
Greece
Guatemala
Guyana
India
Ireland
Jordan
Kenya
Kuwait
Luxembourg
Madagascar
Malawi
Malaysia
Malta
Netherlands
New Zealand
Niger
Nigeria
Norway
Pakistan
Panama
Poland
Portugal
Rhodesia
Saudi Arabia
South Africa
Spain
Sweden
Switzerland
Syria
Tanzania
Thailand
Trinidad and Tobago
Tunisia
United Kingdom
Viet-Nam
Zaire

STANDARD A. 2 - BUTTER-OIL

The following countries have communicated their acceptance of this Standard as a minimum standard:

Belgium
Canada
Democratic
Kampuchea
Denmark
Ecuador
Ethiopia
Fiji
Finland
France
Germany,
Fed. Rep.
Greece
Guatemala
Guyana
India
Ireland
Italy
Jordan
Kuwait
Luxembourg
Madagascar
Malta
Netherlands
New Zealand
Niger
Nigeria
Norway
Pakistan
Panama
Poland
Rhodesia
Saudi Arabia
Spain
Sri Lanka
Sweden
Switzerland
Syria
Tanzania
Thailand
Togo
Trinidad and Tobago
United Kingdom
United States of America
Viet-Nam
Zaire

STANDARD A. 3 - EVAPORATED MILK, EVAPORATED SKIMMED MILK

The following countries nave communicated their acceptance of this Standard as a minimum standard;

Belgium
Canada
Congo
Democratic
Kampuchea
Denmark
Ecuador
Ethiopia
Fiji
Finland
Franco
Germany,
Fed. Rep.
Guatemala
Guyana
India
Ireland
Italy
Jordan
Kenya
Kuwait
Luxembourg
Madagascar
Malaysia
Malta
Netherlands
New Zealand
Niger
Nigeria
Norway
Poland
Portugal
Rhodesia
Saudi Arabia
South Africa
Spain
Sweden
Switzerland
Syria
Tanzania
Thailand
Trinidad and Tobago
Tunisia
United Kingdom
United States of America
Viet-Nam
Zaire

STANDARD A.4 - SWEETENED CONDENSED MILK, SWEETENED SKIMMED CONDENSED MILK

The following countries have communicated their acceptance of this Standard as a minimum standard:

Belgium
Canada
Congo
Democratic
Kampuchea
Denmark
Ecuador
Ethiopia
Fiji
Finland
France
Germany,
Fed. Rep.
Guatemala
Guyana
India
Ireland
Italy
Jamaica
Jordan
Kenya
Kuwait
Luxembourg
Madagascar
Malaysia
Malta
Netherlands
New Zealand
Niger
Nigeria
Norway
Pakistan
Poland
Portugal
Saudi Arabia
South Africa
Spain
Sweden
Switzerland
Syria
Tanzania
Thailand
Trinidad and Tobago
Tunisia
United Kingdom
United States of America
Viet-Nam
Zaire

STANDARD A.5 - WHOLE MILK POWDER, PARTLY SKIMMED MILK POWDER AND SKIMMED MILK POWDER

The following countries nave communicated their acceptance of this Standard as a minimum standard:

Belgium
Bolivia
Burma
Cameroun
Canada
Congo
Cuba
Cyprus
Democratic Kampuchea
Denmark
Ecuador
El Salvador
Ethiopia
Fiji
Finland
Franco
Ghana
Guatemala
Guyana
Hong Kong
India
Iran
Iraq
Ireland
Italy
Jordan
Kenya
Korea
Kuwait
Lao
Liberia
Luxembourg
Madagascar
Malaysia
Malta
Mauritius
Nepal
Netherlands
New Zealand
Niger
Nigeria
Norway
Pakistan
Philippines
Poland
Portugal
Rhodesia
Rumania
Saudi Arabia
Senegal
Sierra Leone
Spain
Sweden
Switzerland
Syria
Tanzania
Thailand
Togo
Trinidad and Tobago
Tunisia
United Kingdom
United States of America
Viet-Nam
Zaire

STANDARD A.6 - GENERAL STANDARD FOR CHEESE

The following countries nave communicated their acceptance of this Standard as a minimum standard:

Belgium
Canada
Congo
Democratic Kampuchea
Denmark
Ecuador
Finland
France
Germany, Fed. Rep.
Guatemala
Guyana
Hong Kong
Ireland
Kuwait
Luxembourg
Madagascar
Malawi
Mauritius
Netherlands
New Zealand
Norway
Philippines
Poland
Portugal
Rhodesia
Saudi Arabia
Spain
Sri Lanka
Sweden
Switzerland
Tanzania
Trinidad and Tobago
United Kingdom
United States of America
Zaire

STANDARD A. 7 - WHEY CHEESES

The following countries have communicated their acceptance of the Standard as a minimum standard:

Belgium
Canada
Denmark
Finland
Franca
Germany, Fed. Rep.
India
Ireland
Madagascar
Netherlands
Norway
Poland
Spain
Sweden
Switzerland
Trinidad and Tobago
United Kingdom
United States of America

JOINT IDF/ISO/AOAC PROPOSAL

CASEINS AND CASEINATES - DETEHMINATION OF LACTOSE CONTENT - SPECTROPHOTOMETRIC METHOD

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method for the determination of the lactose content of caseins and caseinates.

2 REFERENCE

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

3 DEFINITION

lactose content of caseins and caseinates: The content of lactose, expressed as a percentage by mass, as determined by the procedure described in this International Standard.

4 PRINCIPLE

Dissolution of a test portion

a) in hot water in the case of caseinates;
b) in hot water with the addition of sodium hydrogen carbonate in the case of acid caseins;
c) in hot water with the addition of pentasodium triphosphate in the case of rennet casein.

Precipitation of the casein with acetic acid and sodium acetate solution. Filtration, addition of phenol solution and concentrated sulphuric acid to an aliquot portion of the filtrate, and spectrophotometric measurement at a wavelength of 490 nm.

5 REAGENTS

All reagents shall be of recognized analytical quality. The water used shall be glass-distilled water.

5.1 Sodium hydrogen carbonate ($NaHCO_3$).

5.2 Pentasodiura triphosphate ($Na_5P_3O_{10}$).

5.3 Hydrochloric or sulphuric acid, 0,1 N solution.

5.4 Acetic acid, 100 g/1 solution.

5.5 Sodium acetate, I N solution.

5.6 Phenol reagent, 80% (m/m).

Heat a mixture of 8 g phenol and 2 g of distilled water, until the crystals are dissolved.

5.7 Sulphuric acid, concentrated (p 20 1,84 g/ml).

5.8 Lactose monohydrate, 20 g/1 solution.

Dissolve 2 g of lactose monohydrate, weighed to the nearest I mg, in water in a 100 mi volumetric flask. Make up to volume with water and mix well. Store the solution at 0° C.

6 APPARATUS

6.1 Analytical balance.

6.2 Beakers, 100 or 200 ml capacity.

6.3 One-mark pipettes, 1, 2 and 10 ml capacity.

6.4 Micropipettes, 0,2 ml capacity, with 0,001 ml divisions.

6.5 Graduated pipette, 25 ml capacity.

6.6 Test tubes, about 40 ml capacity, with ground neck and ground glass stopper.

6.7 Automatic dispenser capable of dispensing 5 ml of concentrated sulphuric acid within 1 s.

6.8 Water bath, capable of being controlled at a temperature of 60 to 70° C.

6.9 Spectrophotometer.

6.10 Mixer, suitable for mixing inside the test tubes (6.6).

6.11 Grinding device, for grinding the laboratory sample, if necessary (see 8.1.4), without development of undue heat and without loss of moisture. A hammer-mill shall not be used.

6.12 Test sieve, wire cloth, diameter 200 mm, nominal size of aperture 500 µm, with receiver, complying with ISO 565.

7 SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

8 PROCEDURE

8.1 Preparation of the test sample.

8.1.1 Thoroughly mix the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an air-tight container of sufficient capacity to allow this operation to be carried out).

8.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (6.12).

8.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 8.1.1.

8.1.4 Otherwise, grind the 50 g portion, using the grinding device (6.11), until it passes the sieve. Immediately transfer all the sieved sample to an air-tight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

8.1.5 After the test sample has been prepared, the determination (8.4) should be proceeded with as soon as possible.

8.2 Preparation of blank solution

Prepare a blank solution using 25 ml of distilled water, the same apparatus, the same reagents in the same amounts and the same procedure as described in 8.4.1 to 8.4.6 inclusive.

NOTE - For the most accurate results, prepare the blank solution, the test solution and the standard solutions for the calibration graph (see 8.5) simultaneously.

8.3 Test portion

Weigh, to the nearest 1 mg, about 1 g of casein or caseinate in a beaker (6.2).

8.4 Determination

8.4.1 In the case of acid casein, add 0,1 ± 0,001 g of sodium hydrogen carbonate (5.1).

In the case of rennet casein, add 0,1 ± 0,001 g of pentasodium triphosphate (5.2).

8.4.2 Add 25 ml of distilled water and warm to 60 to 70° C in the water bath (6.8), mixing occasionally by shaking.

8.4.3 When the test portion is completely dissolved - generally this takes about 10 to 15 min - cool and add successively:

- 15 ml of distilled water;
- 8 ml of the hydrochloric or sulphuric acid solution (5.3);
- 1 ml of the acetic acid solution (5.4); mixing the contents by shaking after each addition.

8.4.4 Wait 5 min and then add 1 ml of the sodium acetate solution (5.5). Mix by shaking.

8.4.5 Allow the casein precipitate to settle, then filter through a dry filter paper. Discard the first few millilitres of the filtrate.

8.4.6 Pipette 2 ml of the filtrate (8.4.5) into a test tube (6.6), add 0,2 ml of the phenol reagent (5.6) by means of a micropipette (6.4) and mix by shaking. Then add from the automatic dispenser, in less than a second (6.7), 5 ml of concentrated sulphuric acid (5.7), directing the stream of acid against the liquid surface rather than against the side of the test tube in order to obtain good mixing. Immediately mix, using the mixer (6.10) and allow to stand for 15 min. Cool for 5 min in a water bath at 20°C. Wipe the tube and proceed immediately to step 8.4.7.

8.4.7 Measure the absorbance of the solution (8.4.6) at 490 nm against the blank solution (8.2) as reference.

8.4.8 If the absorbance is above the upper limit of the calibration graph (see 8.5), repeat steps 8.4.6 and 8.4.7, using 2 ml of a suitable dilution of the filtrate (8.4.5) instead of 2 ml of the filtrate itself.

NOTE - If such a dilution is made, the formula given in 9.1 must be modified accordingly.

8.5 Calibration graph

Pipette 10 ml of the lactose monohydrate solution (5.8) into a 100 ml volumetric flask and dilute to the mark with water (solution A); 1 ml of solution A corresponds to 2 mg of lactose monohydrate.
Prepare three standard solutions by pipetting 1, 2 and 3 ml of solution A into three 100 ml volumetric flasks and diluting to the mark with water.
The lactose monohydrate concentrations of the standard solutions obtained are respectively 20, 40 and 60 µg/ml.
Introduce respectively into a series of four test tubes (6.6) 2 ml of water and 2 ml of each of the three standard solutions and proceed according to 7.4.6 and 7.4.7, measuring the

absorbances of the three standards against the solution obtained by taking 2 ml of water through the procedure as reference.
Construct a calibration graph by plotting the absorbances of the standards against their lactose monohydrate concentrations in micrograms per millilitre Draw the best-fitting line through the calibration points.

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula

Read the lactose monohydrate concentration of the test solution from the calibration graph (8.5).
The lactose content of the sample, expressed as a percentage by mass, is equal to

$$\frac{0,950 \times \frac{c}{10^6} \times 50}{m} \times 100 = 0,00475 \times \frac{c}{m}$$

where

c is the concentration, in micrograms per millilitre, of lactose monohydrate in the test solution;

m it is the mass, in grams, of the test portion (8.3);

0,950 is the factor for conversion of lactose monohydrate to lactose.

9.2 Repeatability

For lactose contents equal to or less than 0,2% (m/m), the difference between two single results found on identical test material by one analyst using the same apparatus within a short time-interval will exceed 0,03 g of lactose per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

9.3 Reproducibility

For lactose contents equal to or less than 0,2% (m/m), the difference between two single and independent results found by two operators working in different laboratories on identical test material will exceed 0,04 g of lactose per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

10 TEST REPORT

The test report shall show the method used and the result obtained; it shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details necessary for complete identification of the sample.

Submitted to the Committee for Approval

JOINT IFD/ISO/AOAC PROPOSAL

CASEINS AND CASEINATES - DETERMINATION OF WATER CONTENT (Reference Method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the water content of all types of casein and caseinates.

2 REFERENCE

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

3 DEFINITION

water content of casein and caseinates : The loss of mass determined by the procedure described in this International Standard and expressed as a percentage by mass.

4 PRINCIPLE

Drying of a test portion at 102 ± 10 C and weighing to determine the loss of mass.

5 APPARATUS

5.1 Analytical balance.

5.2 Drying oven, well ventilated, capable of being controlled at 102 ± 1° C.

5.3 Flat-bottomed dishes of material non-corrodible under the conditions of the test (for example glass with ground-glass cover, aluminium or stainless steel equipped with tight-fitting lid which can readily be removed) of at least 50 mm (preferably 75 mm) diameter and at least 25 mm deep.

5.4 Desiccator, containing an effective desiccant. If silica gel is used it should be changed daily.

5.5 Grinding device, for grinding the laboratory sample, if necessary (see 7.1.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

5.6 Test sieve, wire cloth, diameter 200 mm, nominal size of aperture 500 µm, with receiver, complying with ISO 565.

5.7 Suitable device for handling dishes, e.g. laboratory tongs.

6. SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

7. PROCEDURE

7.1 Preparation of the test sample

7.1.1 Thoroughly mix the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an air-tight container of sufficient capacity to allow this operation to be carried out).

7.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (5.6).

7.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 7.1.1.

7.1.4 Otherwise, grind the 50 g portion, using the grinding device (5.5), until it passes the sieve. Immediately transfer all the sieved sample to an air-tight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

7.1.5 After the test sample has been prepared, the determination (7.4) should be proceeded with as soon as possible.

7.2 Preparation of the dish

7.2.1 Heat the uncovered dish and its lid (5.3) in the oven (5.2), controlled at 102 ± 1° C, for at least 1 h.

7.2.2 Place the lid on the dish, transfer the covered dish to the desiccator (5.9), allow to cool to the temperature of the balance room and weigh to the nearest 0,1 mg.

7.3 Test portion

Put 3 to 5 g of the test sample (7.1) into the dish, cover with the lid and weigh to the nearest 0,1 mg.

7.4 Determination

7.4.1 Uncover the dish and place it with its lid in the oven (5.2) for 4 h.

7.4.2 Replace the lid on the dish, transfer to the desiccator, allow to cool to the temperature of the balance room and weigh to the nearest 0,1 mg.

7.4.3 Uncover the dish and heat it again, with its lid, in the oven for 1 h. Then repeat operation 7.4.2.

7.4.4 If the mass obtained in 7.4.3 is less than the mass obtained in 7.4.2 by more than 1 mg, repeat operation 7.4.3.

In the event of an increase of mass, take for the calculation the lowest mass recorded.

The total drying time should not normally exceed 6 h.

8 EXPRESSION OF RESULTS

8.1 Method of calculation and formula

The water content of the sample, as a percentage by mass, is equal to

$$\frac{m_1 - m_2}{m_1 - m_0} \times 100$$

where

m_0 is the mass, in grams, of the dish and the lid (7.2.2);

m_1 is the mass, in grams, of the dish, the lid and the test portion before drying (7.3);

m_2 is the mass, in grams, of the dish, the lid and the test portion after drying (7.4.3 or 7.4.4).

Calculate the water content to the nearest 0,01%.

8.2 Repeatability

The difference between two single results obtained on identical test material by one analyst using the same apparatus within a short time-interval will exceed 0,10 g of water per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

9 TEST REPORT

The test report shall show the method used and the result obtained; it shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details necessary for complete Identification of the sample.

Submitted to the Committee for Approval

JOINT IDF/ISO/AOAC PROPOSAL

Rennet caseins and caseinates - Determination of ash

(Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the ash of caseins obtained by rennet precipitation and of caseinates, with the exception of ammonium caseinate.

NOTE - For the determination of ash ("fixed ash") of acid caseins, of ammonium caseinates, o' their mixtures with rennet casein and with caseinates, and of caseins of unknown type, see ISO 5544.

2 REFERENCES

See FAO//WHO Standard B-1 "Sampling Methods for Milk and Milk Products",
ISO 3310/1, *Test sieves - Technical requirements and testing -- Part 1* : *Metal wire cloth.*

ISO 5550, Caseins and caseinates - Determination of water content (Reference method).[1)]

3 DEFINITION

ash of rennet caseins or of caseinates: The substances determined by the procedure described in this International Standard and expressed as a percentage by mass.

4 PRINCIPLE

Incineration of a test portion at 825 ± 25 °C. Weighing of the residue.

5 APPARATUS

5.1 Analytical balance.

5.2 Silica or platinum dish, about 70 mm diameter and 25 to 50 mm deep.

5.3 Muffle furnace, electrically heated, with air circulation, capable of being controlled at 825 ± 25°C.

5.4 Desiccator, containing an effective desiccant.

5.5 Grinding device, for grinding the laboratory sample, if necessary (see 7.1.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

5.6 Test sieve, wire cloth, diameter 200 mm, nominal size of aperture 500 µm, with receiver, complying with ISO 3310/1.

6 SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

7 PROCEDURE

7.1 Preparation of the test sample

7.1.1 Thoroughly mix the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an air-tight container of sufficient capacity to allow this operation to be carried out).

7.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (5.6).

7.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 7.1.1.

7.1.4 Otherwise, grind the 50 g portion, using the grinding device (5.5), until it passes the sieve. Immediately

transfer all the sieved sample to an air-tight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

7.1.5 After the test sample has been prepared, the determination (7.4) should be proceeded with as soon as possible.

7.2 Preparation of the dish

Heat the dish (5.2) in the muffle furnace (5.3), controlled at 825±25°C, for 30 min. Allow the dish to cool in the desiccator (5.4) to the temperature of the balance room and weigh to the nearest 0,1 mg.

7.3 Test portion

Weigh, to the nearest 0,1 mg, directly in or by difference into the prepared dish, approximately 3 g of the test sample (7.1).

7.4 Determination

Heat the dish with its contents on a low flame until the test portion is completely charred, taking care that it does not burst into flame.

Transfer the dish to the electrical furnace (5.3), controlled at 825 ± 25 °C, and heat for at least 1 h until all carbon has disappeared from the dish. Al low the dish to cool in the desiccator (5.4) to the temperature of the balance room and weigh to the nearest 0,1 mg.

Repeat the operations of heating in the electrical furnace (5.3), cooling and weighing, until the mass remains constant to within 1 mg or begins to increase. Record the minimum mass.

8 EXPRESSIONOF RESULTS

8.1 Method of calculation and formula

8.1.1 The ash of the sample, as a percentage by mass, is equal to

$$\frac{m_1 - m_2}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the dish and residue;

m_2 is the mass, in grams, of the prepared dish.

Calculate the ash to the nearest 0,01 % and report the final result to the nearest 0,1 %.

8.1.2 To calculate the ash of the sample on the dry basis, as a percentage by mass, multiply the result obtained in accordance with 8.1.1 by

$$\frac{100}{100 - M}$$

where M is the water content of the sample determined according to ISO 5550.

8.2 Precision

8.2.1 *Repeatability*

The difference between two single results obtained on identical test material by one analyst using the same apparatus within a short time interval will exceed 0,15 g of ash per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

8.2.2 *Reproducibility*

The difference between two single and independent results obtained by two operators working in different laboratories on identical test material will exceed 0,25 g of ash per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

9 TEST REPORT

The test report shall show the method used and the result obtained; it shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any

circumstances that may have influenced the result.

The report shall include all details necessary for complete identification of the sample.

Submitted to the Committee for Approval

JOINT IDF/ISO/AOAC PROPOSAL

Caseins - Determination of "fixed ash" (Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the "fixed ash" of caseins obtained by acid precipitation or lactic fermentation, of ammonium caseinates, of their mixtures with rennet casein and with caseinates, and of caseins of unknown type.

NOTE- For the determination of ash of rennet caseins and caseinates (except ammonium caseinates), see ISO 5545.

2 REFERENCES

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".
ISO 3310/I, *Test sieves - Technical requirements testing - Part I: Metal wire cloth.*

ISO 5550, *Caseins and caseinates - Determination of water content (Reference method)*.[1)]

3 DEFINITION

"fixed ash" of caseins : The substances determined by the procedure described in this International Standard and expressed as a percentage by mass.

NOTE - The designation "fixed ash" is used to indicate that the phosphorus of organic origin is retained in the ash.

4 PRINCIPLE

Incineration of a test portion at 825 ± 25 °C in the presence of magnesium acetate to bind all phosphorus of organic origin. Weighing of the residue and subtraction of the mass of ash originating from the magnesium acetate.

5 REAGENT

The reagent shall be of recognized analytical quality. The water used shall be distilled water or water of at least equivalent purity.

5.1 Magnesium acetate tetrahydrate

$[Mg(CH_3CO_2)_2.4H_2O)$, 120 g/l solution.

6 APPARATUS

6.1 Analytical balance.

6.2 One-mark pipette, 5 ml.

6.3 Silica or platinum dishes, about 70 mm diameter and 25 to 50 mm deep.

6.4 Drying oven, capable of being controlled at 102 ± 2 °C.

6.5 Muffle furnaces electrically heated, with air circulation, capable of being controlled at 825± 25° C.

6.6 Boiling water bath.

6.7 Desiccator, containing an effective desiccant.

6.8 Grinding device, for grinding the laboratory sample, if necessary (see 8.1.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

6.9 Test sieve, wire cloth, diameter 200 mm, nominal size of aperture 500 µm, with receiver, complying with ISO 3310/1.

7 SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

8 PROCEDURE

8.1 Preparation of the test sample

8.1.1 Thoroughly mix the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an air-tight container of sufficient capacity to allow this operation to be carried out).

8.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (6.9).

8.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 8.1.1.

8.1.4 Otherwise, grind the 50 g portion, using the grinding device (6.8). until it passes the sieve. Immediately transfer all the sieved sample to an air-tight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

8.1.5 After the test sample has been prepared, the determination (8.4) should be proceeded with as soon as possible.

8.2 Preparation of the dishes

Meat two dishes (6.3) in the muffle furnace (6.5), controlled a; 825 ± 25 °C, for 30 min. Allow the dishes to cool in the desiccator (6.7) to the temperature of the balance room and weigh to the nearest 0,1 mg.

8.3 Test portion

Weigh, to the nearest 0,1 mg, directly in or by difference into one of the prepared dishes (A), approximately 3 g of the test sample (8.1).

8.4 Determination

Using the pipette (6.2), add to the dish (A) exactly 5 ml of the magnesium acetate solution (5.1) so as to wet all of the test portion, and allow to stand for 20 min.

To the other prepared dish (B), add with the pipette (6.2) exactly 5 ml of the magnesium acetate solution (5.1).

Evaporate the contents of both dishes (A and B) to dryness on the boiling water bath (6.6).

Place both dishes in the oven (6.4), controlled at 102 ± 2 °C, for 30 min.

Meat dish A with its contents on a low flame until the test portion is completely charred, taking care that it does not burst into flame.

Transfer both dishes (A and B) to the electrical furnace (6.5), controlled at 825 ± 25 °C, and heat for at least 1 h until all carbon has disappeared from dish A. Allow both dishes to cool in the desiccator (6.7) to the temperature of the balance room and weigh to the nearest 0,1 mg.

Repeat the operations of heating in the electrical furnace (6.5), cooling and weighing, until the mass remains constant to within 1 mg or begins to increase. Record the minimum mass.

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula

9.1.1 The "fixed ash" of the sample, including phosphorus, as a percentage by mass, is equal to

$$\frac{(m_1 - m_2) - (m_3 - m_4)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of dish A and residue;

m_2 is the mass, in grams, of the prepared dish A;

m_3 is the mass, in grams, of dish B and residue;

m_4 is the mass, in grams, of the prepared dish B.

Calculate the "fixed ash" to the nearest 0,01 % and report the final result to the nearest 0,1 %.

9.1.2 To calculate the "fixed ash" of the sample on the dry basis, as a percentage by mass, multiply the result obtained in accordance with 9.1.1 by

$$\frac{100}{100 - M}$$

where M is the water content of the sample determined according to ISO 5550.

9.2 Precision

9.2.1 *Repeatability*

The difference between two single results obtained on identical test material by one analyst using the same apparatus within a short time interval will exceed 0,1 g of "fixed ash" per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

9.2.2 *Reproducibility*

The difference between two single and independent results obtained by two operators working in different laboratories on identical test material will exceed 0,2 g of "fixed ash" per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

10 TEST REPORT

The test report shall show the method used and the result obtained; it shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details necessary for complete identification of the sample.

Submitted to the Committee for Approval

JOINT IDF/ISO/AOAC PROPOSAL

Caseins and caseinates - Determination of protein content (Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the protein content of caseins and caseinates, excluding those containing ammonium caseinate or other ammonium compounds, or other, nitrogenous non-protein compounds.

2 REFERENCES

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".
ISO 3310/1, *Test sieves - Technical requirements and testing Part I: Metal wire cloth.*

ISO 5550, *Caseins and caseinates - Determination of water content (Reference method)*.[1)]

3 DEFINITION

protein content of caseins and caseinates : The nitrogen content as determined by the procedure described in this International Standard, multiplied by 6,38 and expressed as a percentage by mass.

4 PRINCIPLE

Digestion of a test portion with a mixture of potassium sulphate and sulphuric acid, in the presence of copper (II) sulphate as catalyst, to convert organic nitrogen into ammoniacal nitrogen. Distillation and absorption of the ammonia in boric acid solution. Titration with standard volumetric hydrochloric acid solution. Multiplication of the result by the factor 6,38.

5 REAGENTS

All reagents shall be of recognized analytical quality. The water used shall be distilled water or water of at least equivalent purity.

5.1 Sulphuric acid, concentrated, ρ20 1,84 g/ml.

5.2 Potassium sulphate, anhydrous (K_2SO_4).

5.3 Copper (II) sulphate pentahydrate ($CuSO_4.5H_2O$).

5.4 Sucrose ($C_{12}H_{22}O_{11}$).

5.5 Boric acid, 40 g/l solution.

5.6 Sodium hydroxide, concentrated aqueous solution, 30% (m/m).

5.7 Hydrochloric acid, approximately 0,1 N standard volumetric solution, standardized against sodium tetra-borate decahydrate ($Na_2B_4O_7.10H_2O$) or anhydrous sodium carbonate (Na_2CO_3).

5.8 Mixed indicator

Mix equal volumes of a 2 g/l solution of methyl red in at least 95% (V/V) ethanol and a 1 g/l solution of methylene blue in at least 95 % (V/V) ethanol.

6 APPARATUS

6.1 Analytical balance.

6.2 Kjeldahl flask, 500 ml capacity.

6.3 Digestion apparatus to hold the Kjeldahl flask (6.2) in an inclined position and with a heating device which will not heat the part of the flask above the surface of the liquid contents.

6.4 Condenser with straight inner tube.

6.5 Outlet tube with safety bulb connected to the lower end of the condenser (6.4) by a ground glass joint or a rubber tube. If rubber tubing is

used, the glass ends must be near one another.

6.6 Splash head connected to the Kjeldahl flask (6.2) and to the condenser (6.4) by soft, close-fitting rubber stoppers.

6.7 Conical flask, 500 ml capacity.

6.8 Graduated cylinders, 50 ml and 100 ml capacity.

6.9 Burette, 50 ml capacity, graduated in 0,1 ml.

6.10 Boiling aids :

6.10.1 For the digestion : small pieces of hard porcelain, or glass heads.

6.10.2 For the distillation : freshly calcined pieces of pumice.

6.11 Grinding device, for grinding the laboratory sample, if necessary (see 8 1 4). without development of undue heat and without loss or absorption of moisture A hammer-mill shall not be used

6 12 Test sieve, wire cloth, diameter 200 mm, nominal size of aperture 500 µm, with receiver, complying with ISO 3310/1

7 SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

8 PROCEDURE

8.1 Preparation of the test sample

8.1.1 Thoroughly mix the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an air tight container of sufficient capacity to allow this operation to be carried out)

8.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (6.12)

8.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 8.1.1.

8.1.4 Otherwise, grind the 50 g portion, using the grinding device (6 11), until it passes the sieve. Immediately transfer all the sieved sample to an air-tight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

8.1.5 After the test sample has been prepared, the determination should be proceeded with as soon as possible.

8.2 Test for presence of non-protein nitrogen

If the presence of ammonium caseinate or other ammonium compounds is suspected, carry out the following test. Add to 1 g of sample in a small conical flask, 10 ml of water and 100 mg of magnesium oxide. Rinse down any magnesium oxide adhering to the walls and close the flask with a cork stopper, inserting a piece of red litmus paper between the stopper and the neck of the flask. Mix the contents of the flask carefully and heat the flask in a water bath at 60 to

65 C. If the litmus paper colours blue within 15 min. ammonia is present, and the method is not applicable (see clause 1).

8.3 Blank test

At the same time as the determination of the nitrogen content of the sample, perform a blank determination using 0,5 g of the sucrose (5.4) instead of the test portion, using the same apparatus, the same quantities of all reagents and the same procedure as described in 8.5, If the result of the blank determination exceeds 0.5 ml of 0,1 N acid, the reagents shall be checked and the impure reagent or reagents purified or replaced

8.4 Test portion

Transfer to the Kjeldahl flask (6.2) 0,3 to 0,4 g of the test sample (8.11, weighed to the nearest 0,1 g

8.5 Determination

8.5.1 Transfer to the flask a few pieces of porcelain of a few glass beads (6.10.1) and about 15 g of the anhydrous potassium sulphate (5.2).

Add 0,2 g of the copper (II) sulphate (5.3) and wash down the neck of the flask with a little water Add 20 ml of the concentrated sulphuric acid (5.1) Mix the contents of the flask

Heat gently on the digestion apparatus (6 3) until any frothing has ceased. Boil gently until the solution is clear and a pale green-blue colour persists. During heating, shake the flask from time to time

Continue the boiling, regulating the heating so as to condense the vapours in the middle of the flask neck Continue the heating for 90 min, avoiding local overheating

Allow to cool to room temperature. Carefully add about 200 ml of water and a few pieces of pumice (6.10.2). Mix and cool again.

8.5.2 Transfer into the conical flask (6.7) 50 ml of the boric acid solution (5.5) and 4 drops of the indicator (5 8) Mix. Place the conical flask under the condenser (6 4) so that the tip of the outlet tube (6.5) is immersed in the boric acid solution Using a graduated cylinder (6.8), add to the Kjeldahl flask 80 ml of the sodium hydroxide solution (5 6) During this operation, hold the flask in an inclined position so that the sodium hydroxide solution runs down the side of the flask to form a bottom layer.

Immediately connect the Kjeldahl flask to the condenser by means of the splash-head (6.61.

Gently rotate the Kjeldahl flask to mix its contents Boil gently at first, avoiding any frothing. Continue the distillation so that 150 ml of distillate are collected in approximately 30 min. The distillate should have a temperature below 25°C. About 2 min before the end of the distillation, lower the conical flask so that the tip of the outlet tube is no longer immersed in the acid solution, and

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APPENDIX IX-F

Submitted to the Committee for Approval

JOINT IDF/ISO/AOAC PROPOSAL

Caseins - Determination of free acidity (Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the free acidity of caseins obtained by acid precipitation or lactic fermentation and of rennet caseins.

2 REFERENCES

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".
ISO 3310/1, *Test sieves - Technical requirements and testing Part I: Metal wire cloth.*

ISO 5550, *Caseins and caseinates - Determination of water content (Reference method).* [1)]

[1)] At present at the stage of draft.

3 DEFINITION

free acidity of caseins : Volume, in millilitres, of a 0,1 N standard volumetric sodium hydroxide solution required lo titrate an aqueous extract of 1 g of the product.

4 PRINCIPLE

Aqueous extraction of a test portion at 60 °C. Filtration. Titration of the filtrate with a standard volumetric sodium hydroxide solution, using phenolphthalein as indicator.

5 REAGENTS

All reagents shall be of recognized analytical quality. The water used shall be distilled or deionized water, freed from carbon dioxide by boiling for 10 min before use.

5.1 Sodium hydroxide, approximately 0,1 N standard volumetric solution.

5.2 Phenolphthalein, 10 g/l ethanolic solution.

6 APPARATUS

6.1 Analytical balance.

6.2 Conical flask, 500 ml capacity, with ground neck and fitted with a ground glass stopper.

6.3 One-mark pipette, 100 ml capacity.

6.4 Pipette, suitable for measuring 0,5 ml of indicator solution (5.2).

6.5 Corneal flask, 250 ml capacity.

6.6 Measuring cylinder, 250 ml capacity.

6.7 Burette, graduated in 0,1 ml.

6.8 Water bath, capable of being controlled at a temperature of 60± 2 °C.

6.9 Appropriate filter.

6.10 Grinding device, for grinding the laboratory sample, if necessary (see 8.1.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

6.11 Test sieve, wire cloth, diameter 200 mm, nominal size of aperture 500 µm, with receiver, complying with ISO 3310/I.

7 SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

8 PROCEDURE

8.1 Preparation of the test sample

8.1.1 Thoroughly mix the laboratory sample by repeatedly shaking and

inverting the container (if necessary, after having transferred all of the laboratory sample to an air-tight container of sufficient capacity to allow this operation to be carried out).

8.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (6.11).

8.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 8.1.1. rinse the tip with a little water. Stop heating, remove the outlet tube and rinse its outer and inner walls with a little water, collecting the washings in the conical flask.

8.5.3 Titrate the distillate in the conical flask, using the standard volumetric hydrochloric acid solution (5.7).

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula

9.1.1 The protein content of the sample, expressed as a percentage by mass, is equal to

$$\frac{(V_1 - V_2) \times T \times 1,4}{m} \times 6,38$$

$$= \frac{8,932\ (V_1 - V_2) \times T}{m}$$

where

V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (5.7) used in the determination (8 4);

V_2 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (5.7) used in the blank test (8.3);

T is the normality of the standard volumetric hydrochloric acid solution (5 7);

M is the mass, in grams, of the test portion.

Calculate the protein content to the nearest 0.1 %

9.1.2 To calculate the protein content of the sample on the dry basis, as a percentage by mass, multiply the result obtained in accordance with 9.1.1 by

$$\frac{100}{100 - M}$$

where M is the water content of the sample determined according to ISO 5550

9.2 Precision

9.2.1 *Repeatability*

The difference between two single results obtained v identical test material by one analyst using the same apparatus within a short time interval will exceed 0,5 g of protein per 100 g of product on average not more Than once in 20 cases in the normal and correct operation of the method.

9.2.2 *Reproducibility*

The difference between two single and independent results obtained by two operators working in different laboratories on identical test material will exceed 1,0 g of protein per 100 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

10 TEST REPORT

The test report shall show the method used and the result obtained; it shall also mention all operating conditions not specified in this International Standard, or regarded a? optional, as well as any circumstances that may have influenced the result.

The report shall include all details necessary for complete identification of the sample.

8.1.4 Otherwise, grind the 50 g portion, using the grinding device (6.10), until it passes the sieve. Immediately transfer all the sieved sample to an air-tight container of sufficient capacity. and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

8.1.5 After the test sample has been prepared, the determination (8.3) should be proceeded with as soon as possible.

8.2 Test portion

Weigh about 10 g of the test sample (8.1) to the nearest 10 mg and transfer it to the conical flask (6.2).

8.3 Determination

Using the 250 ml measuring cylinder (6.6), add 200 ml of freshly boiled water, previously heated to 60 °C. Stopper the flask, mix by swirling and place in the water bath at 60 °C (6.8) for 30 min. Shake the flask at intervals of about 10 min.

Filter, and cool the filtrate to about 20 °C. The filtrate must be clear.

Transfer 100 ml of the cooled filtrate into the conical flask (6.5), using the pipette (6.3). Add 0,5 ml of the ethanolic phenolphthalein solution (5.2), using the pipette (6.4). Titrate with the standard volumetric sodium hydroxide solution (5.1), until the appearance of a faint pink colour, persisting for at least 30 s. Record the volume used to the nearest 0,01 ml.

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula

9.1.1 The free acidity of the casein is equal to

$$\frac{20 \times V \times T}{m}$$

where

V is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (5.1) used;

T is the normality of the standard volumetric sodium hydroxide solution (5.1);

m is the mass, in grams, of the test portion.

Calculate the free acidity to the nearest 0,01.

9.1.2 To calculate the free acidity of the sample on the dry basis, multiply the result obtained in accordance with 9.1.1 by

$$\frac{100}{100 - M}$$

where M is the water content of the sample determined according to ISO 5550.

9.2 Precision

9.2.1 *Repeatability*

The difference between two single results obtained on identical test material by one analyst using the same apparatus within a short time interval will exceed 0,02 ml of 0,1 N sodium hydroxide solution per 1 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

9.2.2 *Reproducibility*

The difference between two single and independent results obtained by two operators working ¡n different laboratories on identical test material will exceed 0,04 ml of 0,1 N sodium hydroxide solution per 1 g of product on average not more than once in 20 cases in the normal and correct operation of the method.

10 TEST REPORT

The test report shall show the method used and the result obtained; it shall also mention all operating conditions not

specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details necessary for complete identification of the sample.

Submitted to the Committee for Approval

JOINT IDF/ISO/AOAC PROPOSAL

MILK AND MILK PRODUCTS - DETERMINATION OF LACTOSE IN THE PRESENCE OF OTHER REDUCING SUBSTANCES *

(*) This standard was developed by a joint IDF/ISO/AOAC Group (Group E6 - Chairman: Dr B. Lindqvist of Sweden) after duo consideration of views submitted by member countries (Questionnaire 276/E) and was approved for publication at the IDF Sessions in Stockholm, Sweden, in June 1977 (report E-Doc 80). Before publication, editorial amendments were introduced by the Chairman in close consultation with the members of his Group. It is expected that the method will also be Issued by ISO in 1078 as a Draft International Standard.

In accordance with a decision of the Permanent Committee of IDF Commission E, the present standard should be considered as a "Provisional Standard" (hence the green paper) indicating that the method, although considered as the most suitable one for the time being, is still subject to change or nullification in the light of more experience to be gained by the responsible Group of Experts. Information on results obtained by interested laboratories in testing the present method, would be appreciated. Such information should be sent to the IDF General Secretariat at the above address, preferably by 16 June 1979.

1 SCOPE AND FIELD OF APPLICATION

This standard describes an enzymatic method for the determination of lactose in the presence of other reducing substances.

The method is applicable to milk, milk products and to many foodstuffs containing added milk products. Results may be uncertain when the method is applied to products containing consider-ably more glucose than lactose.

2. DFFINITION

Lactose content: The content of lactose expressed as a percentage by mass, that is obtained when using the method specified below.

3. PRINCIPLE (**)

(**) This method is mainly based on the following publications:

- Bahl R.K., "An Enzymic Method for the Determination of Skimmed milk powder in Raw Sausages". Analyst 96 (1971). 88 92.
- Bahl. R.K., "An Enzymic Method for the Determination of Lactose in Milk including Human Milk". Analyst. 96 (1972). 559-561.

Treatment of a purified extract of the sample with the following enzymes and biochemical substances, added simultaneously but acting in sequence:

- β - galactosidase (EC 3.2.1.23) (***) to split lactose into glucose and galactose;
- hexokinase (EC 2.7.1.1) and adenosine tri-phosphate (ATP) to phosphorylate glucose, both that originally present and that liberated by the β - galactosidase, to glucose-6-phosphate (G-6-P);
- glucose-6-phosphate dehydrogenase (G-6-P-D, EC 1.1.1.49) in the presence of nicotinamide-adenine dinucleotide phosphate (NADP) to oxidixe G-6-P to 6-phosphogluconate (6-GP) and to convert NADP to its reduced form (NADPH).

(***) The EC number refers to the Enzyme Classification number at given in:

- The International Union of Biochemistry, "Enzyme Nomenclature", Elsevier Publ. Co Amsterdam 1966.

Determination of the amount of NADPH by reading the extinction of the test solution at 340 nm. The lactose content is proportional to the amount of NADPH if a correction is made for the glucose present from the beginning of the analysis.

4. REAGENTS

Where not otherwise specified, the reagents shall be of analytical grade. The water used in the preparation of the enzyme solutions shall be of at least doubly glass-distilled purity and the water used for other purposes shall be glass-distilled or of at least equal purity.

4.1 Iron solution

Dissolve 162 g of iron (III) chloride hexahydrate ($FeCl_3$. $6H_2O$) in 500 ml of water. Adjust the pH to 5,0 by addition of sodium hydroxide solution and make up to 1000 ml with water.

Note. Commercially available "dialysed iron solution" containing 5% Fe_2O_3 may be used for convenience.

4.2 Sodium sulphate solution

Dissolve 200 g of sodium sulphate decahydrate (Na_2SO_4 .10 H_2O) in water and make up to 1000 ml.

4.3 β-galactosidase suspension

The specific activity of the β - galactosidase suspension should amount to at least 150 units/ ml (***) (Substrate lactose, pH 7,0, 25 °C). The suspension will keep for about 12 months in a refrigerator. When the suspension is in use, The vessel should be kept immersed in crushed ice.

Note: The β- galactosidase should not contain more than 0.01 % each of galactose dehydrogenase. α - galactosidase, glucose dehydrogenase, α-glucosidase or invertase and not more than 0,1% of lactate dehydrogenase calculated in terms of the specific activity of the enzyme.

(***) Unit (often called International or Standard unit) is defined as the amount of enzyme which will catalyze the transformation of one micromole of substrate per minute under standard conditions.

4.4 Sodium phosphate buffer

0.2 M sodium phosphate, pH 7.5, 0,001 M Mg SO_4. Dissolve 4,2 g sodium dihydrogen phosphate monohydrate (NaH_2PO_4. H_2O), 30,2 g disodium-hydrogen phosphate dihydrate (Na_2HPO_4. $2H_2O$) and 0,25 g magnesium sulfate heptahydrate (Mg SO_4. $7H_2O$) in ca 700 ml water. Check the pH (desired pH 7,5). Dilute to 1000 ml with water.

The solution should be stored at 4 °C.

4.5 Sulphuric acid, analytical grade, ρ 1,84 g/ml, 95-97% (m/m).

4.6 NA DP, 0,012 M aqueous solution.

This solution will keep for 3 weeks in a refrigerator. When the solution is in use, the vessel should be kept immersed in crushed ice.

Note see under 4. 7.

4.7 ATP, 0,080 M aqueous solution

This solution will keep for 3 weeks in a refrigerator. When the solution is in use, the vessel

should be kept immersed in crushed ice.

Note: NADP and ATP may be purchased in a variety of forms, like free acid, monosodium salt, disodium salt, monopotassium salt and dipotassium salt, and with various assay values. Depending on supply any type with sufficient purity may he used. Each laboratory can then easily calculate current weight corresponding to the specified number of millimoles:

4.8 Hexokinase, G-6-PD solution

A solution of hexokinase from baker's yeast (crystallized and lyophilized) and glucose-6-phosphate dehydrogenase from baker's yeast (crystallized, lyophilized, sulphate free) containing at least 100 units (*) of hexokinase and 50 units (*) of glucose-6-phosphate dehydrogenase per millilitre. The solution will keep for at least 12 months in a refrigerator. When the solution is in use, the vessel should be kept immersed in crushed ice.

Notes; This reagent may be obtained commercially as a ready-made mixture, where the ratio of hexokinase to G-6-P-D activity is 2:1.

The reagents 4.4, 4.6, 4.7 and 4.8 may be obtained commercially in kits.

(*) For the definition of units. see par. 4.3.

4.9 Mixed enzyme reagents (see note below):

100 parts of sodium phosphate buffer solution (4.4)

5 parts of NADP solution (4.6)

5 parts of ATP solution (4.7)

1 part of hexokinase-G-6-P-D solution (4.8)

This reagent is stable for 1 2 h at 25 °C and for 4 days at 4 C.

Note: This reagent may be used for convenience in routine determinations. The use of the individual reagents 4.4, 4.6, 4.7 and 4.8 is preferable in non-routine work.

5. APPARATUS

5.1 Analytical balance.

5.2 Pipettes of 2 ml, 1 ml, 100 µl and 20 µl capacity.

5.3 Graduated cylinders 250 ml and 25 ml.

5.4 Filter paper, 15 cm diameter, medium grade.

5.5 Filter funnels, 10 cm diameter.

5.6 Spectrophotometer permitting the measurement of the extinction at 340 nm, and equipped with spectrophotometric cells, of 1 cm optical path length.

5.7 Test tubes suitable for mixing sample and reagents and for subsequent incubation. 100 mm x 10 mm tubes are suggested as suitable.

5.8 High-speed macerating equipment or other suitable mixing device.

5.9 Centrifuge capable of handling 50 ml vessels and producing at least 500 g.

5.10 Water bath, maintained at 30 ± 0.5 °C, for incubation of the sample-enzyme mixtures in the test tubes.

6. SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

7. PROCEDURE

7.1 Test to check the function of the reagents.

A test for the recovery of lactose from a solution of known content of pure lactose monohydrate should be performed according to the par. 7.4 to 7.4.9 and calculated according to par. 8.1.

7.2 Preparation of the test sample.

If necessary, mix the sample before analysis so that the test portion is representative of the sample taken.

7.3 Water content of the test sample.

To permit accurate addition of water at step 7.4.2 the water content of the test sample must he known.

7.4 Determination.

7.4.1 Weigh accurately a test portion that contains from 0,2 to 0,6 g of lactose. If the sample is sticky weigh the test portion on a piece of wax paper or filter paper, and treat the test portion and paper together in step 7.4.2.

7.4.2 Add the test portion to the macerating equipment (5 8) together with 20 ml of the iron solution (4.1), 20 ml of the sodium sulphate solution (4.2), and an amount of water that together with the water present in the test portion makes 210 ml and together with the reagents makes a total of 250 ml of liquids. (The actual total volume is higher due to the volume of the precipitated fat, protein etc. in the sample).

7.4.3 Macerate the test portion avoiding excessive foam formation. Check the pH of the suspension. If it is above 5.0 add a few drops of concentrated sulphuric acid (4.5) to bring the pH into the range 4.8 to 5.0.

Note: As usually not more than 2 to 4 drops of acid are required, the volume added is negligible in comparison with the total volume.

7.4.4 Centrifuge 50 ml of the dispersion for 15 min. at at least 500 g. If necessary, filter the supernatant layer.

7.4.5 Depending on the expected lactose content, use in 7.4.6.1 1 ml each of the supernatant layer or filtrate for the determination and for the blank test, or dilute a suitable aliquot to 100 ml in a volumetric flask and use 1 ml each of that dilution for the determination, and for the blank test.

7.4.6 Carry out the enzyme addition as follows:

7.4.6.1 Where there is a single sample or

In sequence, transfer by pipette to each of two test tubes:

- 1 ml of diluted supernatant layer or filtrate (7.4.5);
- 2 ml of sodium phosphate buffer solution (4.4);
- 100 µl of NADP solution (4.6);
- 100 µl of ATP solution (4.7);
- 20 µl of hexokinase-G-6-P-D solution (4.8).

Mix the contents of each test tube.

7.4.6.2 Where there is a number of samples

Transfer by pipette to each of two test tubes:

- 1 ml of diluted supernatant layer or filtrate (7.4.5);

- 2 ml of the mixed enzyme reagent (4.9).

Mix the contents of each test tube.

7.4.7 Add 20 µl of β - galactosidase suspension (4.3) to one test tube and 20 µl of water to the other tube.

The tube to which water has been added is the blank and will be used as reference in the spectrophotometric determination. It will compensate for the glucose initially present in the sample and for the optical properties of the reagents.

7.4.8 Incubate the test tubes at 30 °C for 30 min. and transfer the contents to 2 spectrophotometer cells. (5.6).

7.4.9 Using the blank solution as a reference, measure the extinction at 340 nm.

8. EXPRESSION OF RFSULTS

8.1 Method of calculation

The lactose content of the sample, expressed as a percentage by mass (L), is given by the formula:

$$L = \frac{E \times MW}{\varepsilon \times d \times m} \times \frac{V_1 \times V_4 \times V_5}{V_2 \times V_3} \cdot 10^{-4}$$

where

E is the extinction at 340 nm measured in accordance with 7.4.9;

ε is the molar extinction coefficient of NADPH at 340 nm =6.22 cm2. µ mol

d is the optical pathlength, in centimetres, of the spectrophotometer cells, d - 1 cm;

MW is the mass, in grams, of one mole of lactose; for anhydrous lactose, MW - 342,30; for α - lactose monohydrate, MW = 360,31; m is the mass, in grams, of the test portion (7.4.1);

V_1 is the total volume of liquid, in millilitres, in the test tube at step 7.4.7.

(V1 = 3,24 using the method 7.4.6.1 and V1 = 3,02 using the method 7.4.6.2);

V_2 is the volume of diluted supernatant layer or filtrate (7.4.5); in millilitres, in the test tube at step 7.4.7.
(V2 is 1 ml in both 7.4.6.1 and 7.4.6.2);

V_3 is the volume, in millilitres, of supernatant layer or filtrate (7.4.4) taken for dilution in step 7.4.5;

V_4 is the volume, in millilitres, of the liquid in the dispersion of the test portion at stop 7.4.2.
(V_4 is 250ml);

V_5 is the volume, in millilitres, of the supernatant layer or the filtrate after dilution in step 7.4.5.
(V_5 is 100 ml.).

8.2 Repeatability

The difference between the results of 2 determinations, carried out simultaneously or in rapid succession by the same analyst, using the same apparatus, should not exceed 1,5% of the assayed lactose content of the sample.

9. TEST REPORT

The test report should state the method used and the result obtained. It shall also mention any operating conditions not specified in this Standard, or regarded as optional, as well as any circumstances that may have influenced the result. The report shall include all details required for the complete Identification of the sample.

Submitted to the Committee for Approval

JOINT IDF/ISO/AOAC PROPOSAL

DRIED MILK - DETERMINATION OF TITRATABLE ACIDITY (Reference Method) •

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the titratable acidity of all types of dried milk.

2 REFERENCES

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products". ISO/R 1736, Dried milk - Determination of fat content (Reference method). IDF Standard 26, Determination of the water content of dried milk. [1)]

1) An ISO standard for the determination of the moisture content of dried milk is under consideration.

3 DEFINITION

titratable acidity of dried milk : The number of millilitres of a 0,1 N sodium hydroxide solution required to titrate a quantity of the reconstituted product corresponding to 10 g of solids-not-fat to the pH of 8,3.

4 PRINCIPLE

Preparation of reconstituted milk by addition of water to a test portion of dried milk corresponding to accurately 5 g of solids-not-fat. Titration with 0,1 N sodium hydroxide solution to the pH of 8,3. Multiplication of the number of millilitres used in the titration by the factor 2, in order to obtain the number of millilitres in terms of 10 g of solids-not-fat. The amount of sodium hydroxide solution required is a function of the amount of natural buffering substances present in the product, and of developed or added acid or alkaline substances.

5 REAGENTS

All reagents shall be of recognized analytical quality. Water shall be distilled or deionized water, freed from carbon dioxide by boiling for 10 min before use.

5.1 Sodium hydroxide, 0,1 ± 0,0002 N standard volumetric solution, Protect this solution against absorption of carbon dioxide.

5.2 Nitrogen.

6 APPARATUS

6.1 Analytical balance.

6.2 pH meter, with a glass electrode and a suitable reference electrode, calibrated using buffers with pH of about 6 and 9, known to within 0,01 pH unit.

6.3 Magnetic stirrer.

6.4 Burette, graduated in 0,1 ml and with an accuracy of 0,05 ml.

6.5 Measuring cylinder of 50 ml capacity.

6.6 Conical flask, 100 ml or 150 ml capacity, with ground glass stopper, and having a neck sufficiently wide to accommodate the electrodes, the burette tip and a nitrogen inlet tube.

7 SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

8 PROCEDURE

8.1 Preparation of the test sample

Transfer the sample to a clean, dry container (provided with an air-tight lid) of a capacity about twice the volume of the sample. Close the container immediately and thoroughly mix the contents by repeatedly shaking and inverting the container. During these operations, exposure of the sample to the atmosphere should be avoided as far as possible, to minimize absorption of water.

8.2 Determination

8.2.1 Weigh $\frac{500}{a}$ ± 0,01 g of the test sample (8.1) into the conical flask (6.6), a being the solids-not-fat content of the sample, expressed as a percentage by mass.

NOTE - The solids-not-fat content of the sample may be calculated by subtracting the fat content (determined in accordance with ISO/R 1736) and the moisture content (determined in accordance with IDF Standard 26) from 100.

8.2.2 Reconstitute the test portion (8.2.1) with 50 ml of water at about 20 C, agitating vigorously, and allow to stand for about 20 min.

8.2.3 Titrate the contents of the conical flask by adding the sodium hydroxide solution (5.1) from the burette (6.4) until the pH has reached 8,3, measured with the pH meter (6.2); during the titration, the solution should be stirred using the magnetic stirrer (6.3), and absorption of carbon dioxide from the air should be avoided by flushing the conical flask with nitrogen. The titration should be completed within 1 min.

Record the volume, in millilitres, of sodium hydroxide solution used, to the nearest 0,05 ml.

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula The titratable acidity is equal to

2 x V

where V is the volume, in millilitres, of the sodium hydroxide solution used for the titration (8.2.3). Express the result to one decimal place.

9.2 Repeatibility

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,4 ml of 0,1 N sodium hydroxide solution per H 10 g. of solids-not-fat.

10 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the

result. The report shall include all details required for the complete identification of the sample.

APPENDIX IX-I

Submitted to Governments for Acceptance

JOINT IDF/ISO/AOAC PROPOSAL

CHEESE - DETERMINATION OF NITRATE AND NITRITE CONTENTS METHOD BY CADMIUM REDUCTION AND PHOTOMETRY

I SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the nitrate and nitrite contents of cheese.

The method is suitable for hard, semi-hard and soft cheeses of various ages and for processed cheese.

2 REFERENCE

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

3 DEFINITIONS

nitrate and nitrite contents of cheese : The contents of substances determined by the procedure specified in this International Standard and expressed respectively as milligrams of nitrate ion'(NO_3^-) and the nitrite ion (NO_2^-) per kilogram (parts per million).

4 PRINCIPLE

Extraction of the cheese with warm water, precipitation of the fat and proteins, and filtration.

Reduction to nitrite of the nitrate in a portion of the filtrate by means of copperized cadmium.

Development of a red colour, in portions of both unreduced filtrate and of the reduced solution, by addition of sylphanilamide and N-1-naphthyl-ethylenediamine dihydrochloride, and photometric measurement at a wavelength of 538 nm.

Calculation of the nitrite content of the sample and of the total nitrite content after reduction of nitrate, by comparing the measured absorbances with those of a series of standard sodium nitrite solutions; calculation of the nitrate content from the difference between these two contents.

5 REAGENTS

All reagents shall be of analytical quality. The water used shall be distilled or deionized, free from nitrite and nitrate.

NOTE - In order to avoid possible inclusion of small gas bubbles in the copperized cadmium column (6.10), the distilled or deionized water used for the preparation of the column (8.1), for checking the reducing capacity of the column (8.2), and for regeneration of the column (8.3) should preferably be freshly boiled and afterwards cooled to room temperature.

5.1 Cadmium granules, diameter 0,3 to 0,8 mm.

If cadmium granules are not available commercially, they may be prepared as follows:

Place a suitable number of zinc rods in a beaker and cover with a 40 g/l solution of cadmium sulphate. From time to time, scrape the cadmium sponge from the rods over a period of 24 h. Remove the zinc rods and decant the liquid until only sufficient remains to cover the cadmium. Wash the sponge two or three times with distilled water. Transfer the cadmium to a laboratory blender together with 400 ml of 0,1 N hydrochloric acid solution and blend for a few seconds to obtain granules of the required size. Return the contents of the blender to the beaker and leave to stand for several hours, occasionally stirring to remove bubbles. Decant most of the liquid and immediately copperize as described in 8.1.1 to 8.1.5.

5.2 Copper (II) sulphate solution.

Dissolve 20 g of copper (II) sulphate pentahydrate ($CuSO_4.5H_2O$) in water and dilute to 1000 ml.

5.3 Buffer solution, pH 9,6 to 9,7.

Dilute 50 ml of concentrated hydrochloric acid [ρ 20 1,19 g/ml; about 38% (m/m) HCl] with 600 ml of water. After mixing, add 100 ml of concentrated ammonia solution [ρ 20 0,88 g/ml; about 35% (m/m) NH_3]. Dilute to 1000 ml with water and mix.

Adjust the pH to 9,6 to 9,7 if necessary.

5.4 Hydrochloric acid solution, about 2 N.

Dilute 160 ml of concentrated hydrochloric acid (ρ20 1,19g/ml) to 1 000 ml with water.

5.5 Hydrochloric acid solution, about 0 1 N.

Dilute 50 ml of 2 N hydrochloric acid solution (b.4) to 1 000 ml with water.

5.6 Solutions for precipitation of proteins and fat.

5.6.1 Zinc sulphate solution.

Dissolve 53,5 g of zinc sulphate heptahydrate ($ZnSO_4$ $7H_2O$) in water and dilute to 100 ml.

5.6.2 Potassium hexacyanoferrate (ll) solution.

Dissolve 17,2 g of potassium hexacyanoferrate (ll) trihydrate [$K_4Fe(CN)_6.3H_2O$] in water and dilute to 100 ml

5.7 EDTA solution
Dissolve 33.5 g of disodium ethylenedinitrilotetraacetate (disodium ethylenedianinetetraacetate) dihydrate ($Na_2C_{10}H_{14}N_2O_8 2H_2O$) in water and dilute to 1 000 ml.

5.8 Solutions for colour development;

5.8.1 Solution I.

Dissolve, by heating on a water bath, 0,5 g of sulphanilamide ($NH_2C_6H_4SO_2NH_2$) in a mixture of 75 ml of water and 5 ml of concentrated hydrochloric acid ($_{\rho 20}$ 1,19 g/ml). Cool to room temperature and dilute to 100 ml with water. Filter if necessary

5.8.2 Solution II.

Dilute 450 ml of concentrated hydrochloric acid ($_{\rho 20}$ 1.19 g/ml) to 1 000 ml with water.

5.8.3 Solution III.

Dissolve 0,1 g of N.1 naphthyi ethylenediamine dihydrochloride ($C_{10}H_7NHCH_2CH_2NH_22HCl$) in water. Dilute to 100 ml with water. Filter if necessary.

The solution may be stored for up to 1 week in a well-stoppered brown bottle in a refrigerator.

6.9 Sodium nitrite, standard solution.

Dissolve in water 0,150 g of sodium nitrite ($_{NaN02),}$ dried to constant mass at 110 to 120°C, dilute to 1 000 ml with water in a one-mark volumetric flask, and mix.

On the day of use, dilute 10 ml of this solution with 20 ml of the buffer solution (5.3) and dilute further to 1 000 ml with water in a one mark volumetric flask. Mix.

1 ml of this final dilution contains 1,00µg of NO_2^-

5.10 Potassium nitrate, standard solution.

Dissolve in water 1,468 g of potassium nitrate (KNO_3), dried to constant mass at 110 to 120 °C, and dilute to 100) ml with water in a one mark volumetric flask.

On the day of use, dilute 5 ml of this solution with 20 ml of the buffer solution (5.3) and dilute further to 1 000 ml with water in a one mark volumetric flask. Mix.

1 ml of this final dilution contains 4.50 µg of NO_3^-.

6 APPARATUS

All glassware shall be thoroughly cleaned and rinsed with distilled water to ensure that it is free from nitrate and nitrite.

6.1 Analytical balance.

6.2 Appropriate grinding device.

6.3 Suitable laboratory mixer/homogenizer with glass containers of 250 or 400 ml capacity.

6.4 Conical flasks of 250 ml capacity.

6.5 Volumetric flasks of 100 , 500 and 1 000 ml capacity, complying with ISO 1042, class B.6.6

6.6 Pipettes, to deliver 2 - 4 - 5 - 6 - 8 - 10 - 12 - 20 - 25 and 50 ml, complying with ISO/R648: class A, or ISO/R 835.

NOTE - Where appropriate, burettes may be used instead of pipettes.

6.7 Graduated cylinders of 5 - 10 - 25 - 100 - 250 - 500 and 1 000 ml capacity

6.8 Glass funnels, diameter about 7 cm, with short stem.

6.9 Filter paper, medium grade, diameter about 15 cm, nitrate and nitrite free.

6.10 Reduction column (for example, as shown in the figure).

6.11 Photoelectric: colorimeter or spectrophotometer, suitable for making readings at a wavelength of 538 nm, with cells of 1 to 2 cm optical path length.

7 SAMPLING

7.1 See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

7.2 Store the sample in such a way that deterioration and change in composition are prevented.

8 PROCEDURE

8.1 Preparation of the copperized cadmium column

8.1.1 Transfer the cadmium granules (5.1) (approximately 40 to 60 g for each column) into a conical flask (6.4)

8.1.2 Add sufficient 2 N hydrochloric acid solution (5.4) to cover the cadmium. Swirl for a few minutes.

8.1.3 Decant the solution and wash the cadmium in the flask with water, until it is free from chloride.

8.1.4 Copperize the cadmium granules by adding copper (ll) sulphate solution (5.2) (about 2,5 ml pet gram of cadmium) and swirling for 1 min.

8.1.5 Decant the solution and wash the copperized cadmium immediately with water, taking care that the cadmium is continuously covered with water. Terminate the washing when the wash water is free from precipitated copper.

8.1.6 Fit a glass wool plug to the bottom of the glass column intended to contain the copperized cadmium (see figure) Fill the glass column with water.

8.1.7 Transfer the copperized cadmium into the glass column with minimum exposure to air. The height of the copperized cadmium should be 15 to 20 cm.

NOTES

1 Avoid trapping air bubbles between the copperized cadmium granule!.

2 Take care not to allow the level of the liquid to fall below the top of the copperized codmium.

8.1.8 Condition the newly prepared column by running through it a mixture of 750 ml of water, 225 ml of standard potassium nitrate solution (5 10). 20 ml of buffer solution (5.3) and 20 ml of EDTA solution (5.7), at a flow rate not exceeding 6 ml/min, then wish the column with 50 ml of water.

8.2 Checking the reducing capacity of the column

Carry out this check at least twice a day, at the beginning and at the end of a series of determinations.

8.2.1 Pipette 20 ml of standard potassium nitrate solution (5.10) into the reservoir on top of the column Immediately add 5 ml of buffer solution (5 3) to the contents of the reservoir. Collect the eluate in a 100 ml Volumetric flask.
The flow rate shall not exceed 6 ml/min.

8.2.2 When the reservoir has nearly run empty, wash the walls of the reservoir with about 15 ml of water and, when this has run off, repeat the same treatment with another 15 ml portion of water. After this second portion of water has run into the column as well, completely fill the reservoir with water and allow it to pass through the column at maximum flow rate.

8 2.3 After nearly 100 ml of eluate has been collected, remove the volumetric flask, make up to the mark with water and mix well.

8 2 4 Pipette 10 ml of the eluate into a 100 ml volumetric flask. Add water to obtain a volume of about 60 ml. Proceed as specified in 8 9.2, 8.9.3 and 8.9.4.

8 2.5 If the nitrite concentration of the diluted eluate (8.2.4), as determined from the calibration curve (8.101. is below 0.063 µg of NO_2 per millilitre (i.e. 95% of theoretical value), the column should be regenerated.

8.3 Regeneration of the column

Regenerate the column as follows, at the end of each day after use, or more frequently if the check (8.2) indicates a loss of efficiency.

8.3.1 Add about 5 ml of EDTA solution (5 7) and 2 ml of 0.1 N hydrochloric acid solution (5.5) to 100 ml of water. Run the mixture through the column at a flow rate of about 10 ml/min.

8 3.2 When the reservoir has run empty, wash the column with water, 0,1 N hydrochloric acid solution and water successively

8.3.3 If the column still does not show a satisfactory efficiency. repeat the procedure specified in 8.1.1.

8.4 Preparation of the test sample

Prior to analysis. remove the rind or mouldy surface layer of the cheese, in such a way as to provide a sample representative of the cheese as it is usually consumed. Grind the sample by means of an appropriate device: mix the ground mass quickly, and if possible grind a second time and again mix thoroughly. If the sample cannot be ground, mix it thoroughly by intensive stirring and kneading.

Transfer the test sample to an air-tight container to await analysis. which should be carried out as soon as possible after grinding If delay is unavoidable, take all precautions to ensure proper preservation of the sample and to prevent condersation moisture on the made under of the container. Ground cheese showing unwanted mould growth or begging to detuned are should not be examined.

Clean the device after grinding each sample.

8.5 Test portion

Weigh 10 g of the test sample, to the nearest 1 mg. and transfer it quantitatively into the glass container of the mixer homogenizer (6.3).

8.6 Extraction and deproteination

8.6.1 Add gradually 164 ml of warm water (50 to 55° C) to the test portion. Mix in the mixer/homogenizer until the cheese is well suspended.

8.6.2 Add, in the following order, 6 ml of zinc sulphate solution (5.6.1), 6 ml of potassium hexacyanoferrate (II) solution (5.6.2) and 20 ml of buffer solution (5.3) to the cheese suspension, swirling thoroughly after each addition.

8.6.3 After 3 min, filter through a filter paper (6.9), collecting the filtrate in a 250 ml conical flask.

NOT E - It is necessary to obtain a clear filtrate. For this purpose, if well-matured cheese are analysed, it might be necessary to use a larger quantity of clarification reagents.

8.7 Reduction of nitrate to nitrite

8.7 1 Pipette 20 ml of the filtrate (8,6.3) into the reservoir on top of the reduction column. Add 5 ml of buffet solution (5.3) to the content of the reservoir. Collect the eluate in a 100 ml volumetric flask. The low rate shall not exceed 6 ml/min.
8.7.2 When the reservoir has nearly run empty, wash the walls of the reservoir with about 15 ml of water and , when this has run oft, repeat the same treatment with another 15 ml portion of water. After this second portion of water has me into the column as well, completely fill the reservoir with water and allow it to flow through the column at maximum flow rate.

8.7.3 After nearly 100 ml of eluate has been collected, remove the volumetric flask, make up to mark with water and mix well.

8.8 Preparation of solution for determination of nitrite in sample

Pipette 20 ml of the filtrate (8.6.3) into a 100 ml volumetric flask, make up to the mark with water and mix well.

8.9 Determination

8.9.1 Pipette equal aliquots (for example 25 ml) of the diluted filtrate (8.8) and of the eluate (8.7.3) into separate 100 ml volumetric flasks. Add water to each to obtain a volume of about 60 ml. Then treat the contents of each flask as m 8.9.2, 8 9 3 and 8.9.4.

8.9.2 Add 6 ml of solution II (5.8.2) and then 5. ml of solution I (5.8.1). Mix carefully and leave the solution for 5 min at room temperature, protected from direct sunlight.

8.9.3 Add 2 ml of solution III (5 8 3) Mix carefully and leave the solution for 5 min at room temperature, protected from direct sunlight Make up to the mark with water and mix well.

8.9.4 Measure within 1°, min the absorbance of the solution against that of a reagents. blank (8. 10) at a wave length of 538 nm.

8.9.5 Carry out two determinations on the same diluted filtrate (8.8) and two determinations on the same eluate (8.7.3).

8.10 Blank test

Carry out a reagents blank test using all reagents and 4 ml of water instead of the test portion.

8.11 Calibration curve

8.11.1 Pipette 0-2-4-6-8 10 - 12 16 and 20 ml of the standard sodium nitrite solution (6.9) into separate 100 ml volumetric flasks. Add water to each volumetric flask to obtain volumes of about 60 ml.

8.11.2 Carry out the procedure described in 0.9.2 and 8.9.3.

8.11.3 Measure within 1 5 min the absorbances of the solutions against that of the first solution (containing no sodium nitrite) at a wavelength of 638 nm.

8.11.4 Plot the absorbances obtained in 8.11.3 against the nitrite concentrate, in micrograms per millilitre, calculated from the amounts of standard sodium nitrate solution added (see 8.11.1).

9 EXPRESSION OF RESULTS

9.1 Nitrite content

9.1.1 Method of calculation and formula

Calculate the nitrite content of the sample, expressed as milligrams of nitrition (NO_2^-) per kilogram f using the formula:

$$NO_2^- = \frac{100000 \times \underline{c}1}{\underline{m} \times \underline{V}}$$

where

$\underline{c}1$ is the concentration, in micrograms of NO_2^- per millilitre, read from the calibration curve, that corresponds with the measured absorbance (8.9.4) of the solution obtained using the diluted filtrate (8.8);

$\underline{m}$ is the mass, in grams, of the test portion;

$\underline{V}$ is the volume, in millilitres, of the aliquot taken (8.9.1) from the diluted filtrate (8.8).

Take as the result the arithmetic mean of the two determinations '8.9.5).

Report the result to the nearest 0,1 mg/kg.

9.1.2 Repeatability

The difference between the results of a determination in duplicate (results obtained almost simultaneously or in rapid succession by the same analyst) shall not exceed 1 mg/kg.

9.2 Nitrate content

9.2.1 Method of calculation and formula

Calculate the nitrate content of the sample, expressed as milligrams of nitrition (NO_3^-) per kilogram, using the formula:

$$NO_3^- = 1,35\left(\frac{100000 \times \underline{c}_2}{\underline{m} \times \underline{V}} - NO_2^-\right)$$

where

$\underline{C}_2$ is the concentration, in micrograms of NO_2 per millilitre, read from the calibration curve, that corresponds with the measured absorbance (8 9.4) of the solution obtained using the eluate (8.7.3);

NO_2^- is the nitrite content of the sample, expressed as milligrams per kilogram, calculated described in 9.1.1:

$\underline{m}$ is the mass, in grams, of the test portion;

$\underline{V}$ is the volume, in millilitres, of the aliquot taken (8.9.1) from the eluate (8.7.3).

Take as the result the arithmetic mean of the two determinations (8.9.5). Report the result to the nearest 1 mg/kg.

9.2.2 Repeatability

The difference between the results of a determination in duplicate (results obtained almost simultaneously or in rapid succession by the same analyst) shall not exceed 3 mg/kg if the nitrate content is lower than 30 mg/kg and shall not exceed 10 % of the arithmetic mean of the results if the nitrate content exceeds 30 mg/kg.

10 TEST REPORT

The test report shall show the method used and the results obtained. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the results.

The report shall include all details necessary (or complete identification of the sample

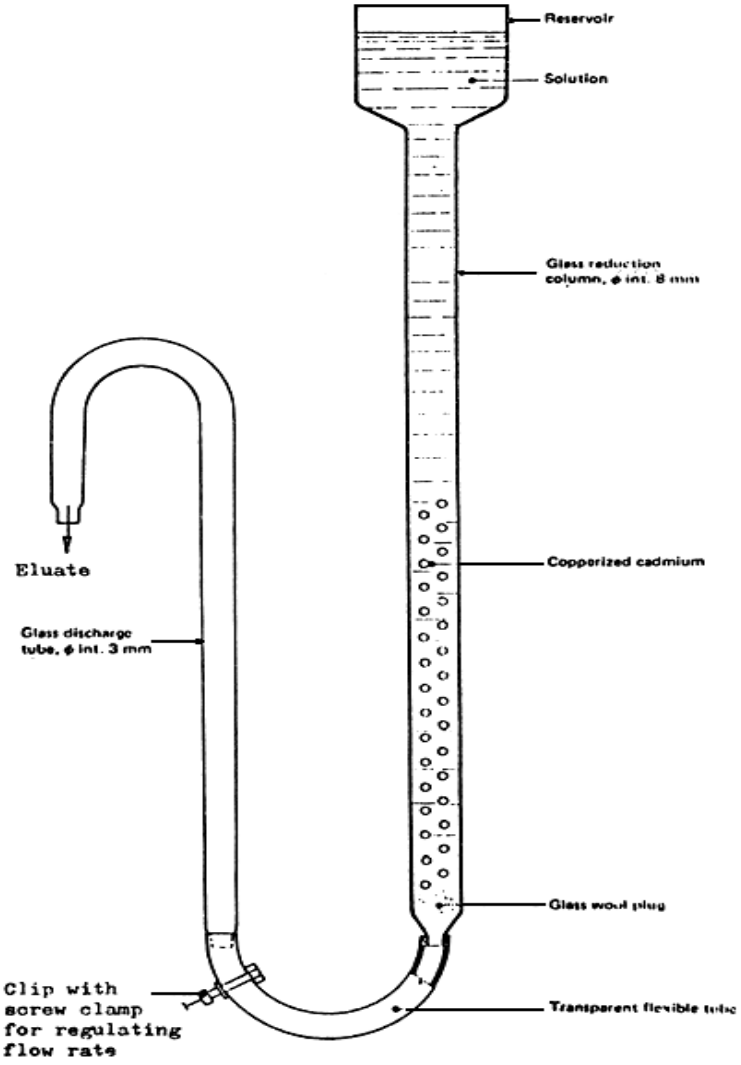

FIGURE – Apparatus for nitrate reduction

Submitted to Governments for Acceptance

JOINT IDF/ISO/AOAC PROPOSAL

Anhydrous milk fat - Determination of peroxide value (Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the peroxide value of anhydrous milk fat and related products.

The method is applicable to anhydrous milk fat, anhydrous butter oil (anhydrous butterfat), butter oil (butterfat) or ghee having peroxide values not exceeding 1,0.

NOTE These products are defined in IDF Standard 68 : 1971.

It is not applicable to products containing gallates as antioxidants.

2 REFERENCE

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

3 DEFINITION

peroxide value : The number of milliequivalents of oxygen per kilogram of anhydrous milk fat, determined by the procedure described.

4 PRINCIPLE

Dissolution of a test portion in a mixture of chloroform and methanol and addition of iron (II) chloride and ammonium thiocyanate. After a fixed reaction time, photometric determination of the red iron (III) complex.

5 REAGENTS

All reagents shall be of analytical reagent quality. The water used shall be distilled water or water of at least equivalent purity.

5.1 Chloroform/methanol mixture.

Mix 70 volumes of chloroform (trichloromethane) and 30 volumes of anhydrous methanol.

5.2 Iron (II) chloride solution.

This solution shall be prepared in indirect, dimmed light.

Dissolve approximately 0,4 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in about 50 ml of water.

Dissolve approximately 0,5 g of iron (II) sulphate heptahydrate ($FeSO_4.7H_2O$) in about 50 ml of water.

Slowly pour the barium chloride solution, with constant stirring, into the iron (II) sulphate solution and add about 2 ml of approximately 10 N hydrochloric acid.

Allow the precipitate of barium sulphate to settle or centrifuge the mixture until the upper liquid layer is clear. Decant the clear solution into a brown bottle. Do not store the solution for more than 1 week.

NOTE - The iron (II) chloride solution can also be prepared by dissolving approximately 0.35 g of iron (II) chloride tetrahydrate ($FeCl_2.4H_2O$) in about 100 ml of water and adding 2 ml of approximately 10 N hydrochloric acid.

5.3 Ammonium thiocyanate solution.

Dissolve approximately 30 g of ammonium thiocyanate (NH_4SCN) in water and dilute to 100 ml. If the solution is not colourless, remove the colour by extracting the solution several times with small amounts (for example 5 ml portions) of iso amyl alcohol (3-methyl butan 1 of).

5.4 Iron (lll) chloride, standard solution corresponding to 10 µg of Fe per millilitre.

Dissolve 0,500 g of iron powder or iron wire in about 50 ml of 10 N hydrochloric acid and 1 to 2 ml of about 30 % (m/m) hydrogen peroxide solution.

Remove the excess of hydrogen peroxide by boiling for 5 min. Cool to room temperature and dilute with water to 500 ml in a volumetric flask. Transfer, by means of a pipette, 1 ml of this solution to a 100 ml volumetric flask, dilute to the mark with the mixture of chloroform and methanol (5.1) and mix.

5.5 Hydrochloric acid, approximately 0,2 N solution.

Dilute 2 ml of approximately 10 N hydrochloric acid with water to 100 ml.

6 APPARATUS

6.1 Analytical balance.

6.2 Burettes, of 10 ml capacity, graduated in 0.02 ml. complying with class A of ISO/R 385.

6.3 Graduated pipettes, of 1 ml capacity, graduated in 0.05 ml, complying with class A of ISO/R 835.

NOTE: Alternatively pipettes of smaller capacity (not covered by ISO, R 835) may be used.

6.4 Photometer, suitable for measuring at a wavelength of 500 nm and with appropriate (preferably round) cells with an optical path length of at least 15 mm and a capacity of at least 15 ml.

7. SAMPLING

FAO/WHO Standard B-1 "Sampling Methods as Milk and Milk Products". the laboratory sample should be received in a securely closed air tight container at least three-quarters filled and protected from light. Note and report any laboratory sample not complying with these requirements.

8 PROCEDURE

8.1 Preparation of the test sample

Carry out the preparation as far as practicable ¡n indirect subdued light.

Completely liquefy the laboratory sample, if necessary, by warming the unopened container at the lowest temperature necessary to achieve liquefaction. Mix the liquefied sample, taking care to avoid the inclusion of air in the sample as far as possible. Proceed with the determination without delay and while the test sample is still liquid.

8.2 Precautions

In order to eliminate lipid oxidation, the following precautions shall be observed.

8.2.1 Avoid exposure of the sample to light.

8.2.2 Take care that the procedure, from 8.3.1 to 8.3.5 inclusive, with inclusion of a reaction time of 5 min, is completed within 10 min.

8 2.3 Carry out the test in indirect light, subdued as much as is practicable.

8.3 Determination

8.3.1 Weigh, to the nearest 0,001 g, in a photometer cell (see 6.4) approximately 0,3 g of the prepared test sample (8.1). Note the time (see 8.3.5).

8.3.2 Without delay, add 9,60 ml of the mixture of chloroform and methanol (5.1) to the cell by means of a burette (6.2); mix gently to dissolve the test portion.

NOTE - For a number of simultaneous determinations it may be advantageous to carry out the analysis in cylindrical photometer cells fitted with ground glass stoppers.

8.3.3 Add from a graduated pipette (6.3) 0,05 ml of the ammonium thiocyanate solution (5.3) and mix.

8.3.4 Measure the extinction (fat blank extinction E_0) at 500 nm against the mixture of chloroform and methanol container) in a similar cell.

8.3.5 Add from a graduated pipette (6.3) 0,05 ml of the iron (ll) chloride solution (5.2), mix and start an alarm clock or stop-watch for a waiting time of 5 min. Then measure the extinction (E_2) at 500 nm against the mixture of chloroform and methanol. This operation shall be completed within 10 min of the time noted in 8.3.1.

8.3.6 Carry out a reagents blank test by transferring 9,90 ml of the mixture of chloroform and methanol to a photometer cell (but omitting the test portion) and proceeding as described in 8.3.3 and 8.3.5.

(The extinction observed is the reagents blank extinction E_1)

8.4 Calibration curve

Transfer from a burette (6.2) to four cells 0,25 - 0,5 1 and 2 ml respectively of the standard iron (lll) chloride solution (5.4) so as to obtain a series containing 2,5 - 5 10 and 20µg of Fe^{3+}.

Add from a burette (6.2) to these four cells 9,65 9.4 8,9 and 7,9 ml respectively of the mixture of chloroform and methanol (5.1). Add from a graduated pipette (6.3) to each of the four cells 0,05 ml of the ammonium thiocyanate solution (5,3) and from another graduated pipette 0,05 ml of the hydrochloric acid solution (5.5) and mix. Note for each cell the time at which this stage is reached.

After a reaction time of 5 min for each cell, measure the extinction at 500 nm against the chloroform and methanol mixture contained in a similar cell.

Plot the measured extinctions against the masses of Fe^{3+} expressed in micrograms.

Construct the best-fitting straight line through the points.

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formulas

9.1.1 Calculate from the difference of extinction

$$E_2 - (E_0+E_1)$$

by means of the calibration curve, or by means of the factor calculated from the calibration curve, the content (m) of Fe^{3+}, in micrograms.

E_0 is the extinction measured as described in 8.3.4.

E_1 is the extinction measured as described in 8.3.6.

E_2 is the extinction measured as described in 8.3.5.

9.1.2 The peroxide value of the fat, expressed as milliequivalents of oxygen per kilogram, is equal to

$$\frac{m}{55{,}84\ m_0}$$

where

m is the mass, in micrograms, of Fe^{3+} calculated as described in 9.1.1;

m_0 is the mass, in grams, of the test portion.

Express the result to the nearest 0,01 unit of peroxide value.

9.2 Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession by the same analyst, using the same apparatus, shall not exceed 0,05 unit of peroxide value.

10 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International

Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The test report shall include all details required for the complete identification of the sample.

CX 5/70-19th Session
APPENDIX IX-K

Submitted to Government for Acceptance

JOINT IDF/ISO/AOAC PROPOSAL

Butter - Determination of water, solids-not-fat and fat contents on the same test portion (Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the water, solids-not-fat (including salt), and fat contents on the same test portion of butter.

2. REFERENCE

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

3 DEFINITIONS

3.1 water content of butter : The loss of mass, expressed as a percentage, as determined by the procedure specified.

3.2 solids-not-fat content of butter : The percentage by mass of substances as determined by the procedure specified.

3.3 fat content of butter : The percentage by mass obtained by subtracting the water content and the solids-not fat content from 100.

4 PRINCIPLE

4.1 Determination of water content

Drying of a known mass of butter at 102±2°C and weighing to determine the loss of mass.

4.2 Determination of solids-not-fat content

Extraction of the fat from the dried butter (4.1) with light petroleum or n-hexane and weighing of the residue.

4.3 Determination of fat content

Calculation of the fat content by difference (see 3.3).

5 REAGENT

n- Hexane or, alternatively, light petroleum (petroleum spirit) with any boiling range between 30 and 60 °C. The reagent shall not leave more than 1 mg of residue after evaporation of 100 ml.

6. APPARATUS

Usual laboratory equipment and in particular :

6.1 Analytical balance.

6.2 Drying oven, well ventilated and capable of being controlled at 102 ± 2 °C.

6.3 Dishes, of glass, porcelain or metal resistant to corrosion under the conditions of the test, at east 25 mm high and at least 50 mm in diameter.

6.4 Filter crucibles, sintered glass, porosity grade P 40 (pore diameters 16 to 40 µm), with suction false:

6.5 Stirrer with end piece of flexible, inert material.

6.6 Desiccator containing a suitable drying agent, for example silica gel containing an indicator.

7 SAMPLING

See FAO/WHO Standard B-1 "Sampling Method s for Milk and Milk Products".

8 PROCEDURE

8.1 Preparation of the test sample

Warm the laboratory sample in the origin. I unopened container, which should be from one-half to two thirds full, to a temperature at which the sample will be soft enough to facilitate a

thorough mixing to a homogeneous state (either by a mechanical shaker or by hand) without any rupture of emulsion. The temperature of mixing should normally not exceed 35° C

Cool the sample to ambient temperature, continuing to mix until cooling is completed. As soon as possible after cooling. open the sample container and stir briefly (not longer than 10 s) with a suitable device, fo example a spoon or spatula, before weighing.

8.2 Determination of water content

8.2.1 Dry a dish (6.3) in the oven (6.2) at 102 ± 2 °C for at least 1 h.

8.2.2 Allow the dish to cool in the desiccator (6.6) to the temperature of the balance room and weigh to the nearest 0,1 mg.

8.2.3 Weigh in the dish, to the nearest 1 mg, a test portion of between 2 and 6 g of the test sample. (8.1). (Test portions shall be between 5 and 6 g for unsalted butter.)

8.2.4 Place the dish in the oven at 102 ± 2 °C and leave it for 2 h.

8.2.5 Allow the dish to cool in the desiccator to the temperature of the balance room and weigh to the nearest 0,1 mg.

8.2.6 Repeat the drying process for 1 h and then for additional 30 min periods, cooling and weighing each time as specified in 8.2.5, until constant mass (mass change not exceeding 0,5 mg) is reached. In the event of an increase in mass, take for the calculation the lowest mass recorded.

8.3 Determination of solids-not-fat content

8.3.1 Dry a filter crucible (6.4) in the oven (6.2) at 102 ± 2 °C for at least 1 h.

8.3.2 Allow the crucible to cool in the desiccator (6.6) to the temperature of the balance room and weigh to the nearest 0,1 mg.

8.3.3 Add 10 to 15 ml of warm (see note) *n*-hexane or light petroleum (clause 5) to the dish containing the dry matter left from the water determination (8.2), to dissolve the fat.

NOTE - In the case of *n*-hexane or of light petroleum having an initial boiling point of 40 °C or above, use a temperature of 35 °C; in the case of light petroleum having an initial boiling point below 40 °C, use a temperature of 25 °C.

8.3.4 Detach as much as possible of the sediment adhering to the dish by using the stirrer (6.5), and transfer the contents quantitatively into the weighed crucible (8.3:2) with the aid of the stirrer tip.

8.3.5 Repeat operations 8.3.3 and 8.3.4 five times.

8.3.6 Wash the sediment in the crucible with 25 ml of warm (see note in 8.3.3) *n*-hexane or light petroleum (clause 5).

8.3.7 Dry the dish and crucible in the oven at 102 ± 2 °C for 30 min.

8.3.8 Allow the dish and crucible to cool in the desiccator to the temperature of the balance room and weigh to the nearest 0,1 mg.

8.3.9 Repeat operations 8.3.7 and 8.3.8 until constant mass (mass change not exceeding 0,5 mg) is reached.

8.4 Number of determinations

Carry out the procedure specified in 8.2 and 8.3 on duplicate test portions taken from the same prepared test sample.

9 EXPRESSION OF RESULTS

9.1 Method of calculation of water content

For each of the duplicate test portions, calculate the water content, *E*, as a percentage by mass, using the following formula :

$$E = \frac{m_1 - m_2}{m_1 - m_0} \times 100$$

where

m_0 is the mass, in grams, of the empty dish (8.2.2);

m_1 is the mass, in grams, of the test portion and dish before drying (8.2.3);

m_2 is the mass, in grams, of the test portion and dish after drying (8.2.6).

Provided that the requirement for repeatability (9.4.1) is satisfied, take as the result the arithmetic mean, *E,* of the values obtained, expressed to the first decimal place,

9.2 Method of calculation of solids not-fat content

For each of the duplicate test portions, calculate the solids-not-fat content, S, as a percentage by mass, using the following formula :

$$S = \frac{(m_4 - m_3) + (m_5 - m_0)}{m_1 - m_0} \times 100$$

where

m_0 and m_1 are as defined in 9.1;

m_3 is the mass, in grams, of the empty crucible (8.3.2);

m_4 is the mass, in grams, of the crucible containing sediment (8.3.9);

m_5 is the final mass, in grams, of the dish (83 9).

Provided that the requirement for repeatability (9.4.2) is satisfied, take as the result the arithmetic mean, *S,* of the values obtained, expressed to the first decimal place.

9.3 Method of calculation of fat content

The percentage, by mass, of fat is equal to :

$$100 - (\overline{E} + \overline{S})$$

where

$\overline{E}$ is the percentage, by mass, of water (9.1);

$\overline{S}$ is the percentage, by mass, of solids-not-fat (9.2).

Express the result to the first decimal place.

9.4 Repeatability

9.4.1 *Water content*

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0.1 g of water per 100 g of the product.

9.4.2 *Solids-not-fat content*

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,1 g of solids not fat per 100 g of the product.

10 TESTREPORT

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may nave influenced the results.

The report shall include all details required for the complete identification of the sample.

FAO/WHO STANDARD METHOD No. B-16

MILK FAT-DETECTION OF VEGETABLE FAT BY THE PHYTOSTERYL ACETATE TEST

1. SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the detection in milk fat of the presence of the more common vegetable fats, using the phytosteryl acetate test.

2. REFERENCES

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products", and B-17 "Milk Fat - Detection of Vegetable Fat by Gas-liquid Chromatography of Sterols (Reference Method).

3. DEFINITION

Sterols Content of Fat: The content of compounds precipitable as digitonides, expressed as a percentage by mass, as determined by the procedure described (see note under 8.2.9).

4. PRINCIPLE

4.1 Saponification of the fat and precipitation of the sterols as sterol digitonides by addition of an ethanolic digitonin solution.

4.2 Determination of the melting point of the steryl acetate after acetylation of the steryl digitonides with acetic anhydride.

4.3 Microscopical examination of the crystal form of the sterols after conversion of the steryl acetates into the sterols by saponification with a potassium hydroxide solution.

5. REAGENTS

All reagents shall be of analytical quality. Water shall be distilled water or water of at least equivalent purity.

5.1 Potassium hydroxide solution. Dissolve 400 g of potassium hydroxide in 600 ml of water.

5.2 Digitonin, 10 g/l ethanolic solution. Dissolve 10 g of digitonin in 1 l of ethanol (5.3).

5.3 Ethanol, 95 to 96% (v/v).

5.4 Ethanol. 80% (v/v).

5.5 Diethyl ether.

5.6 Acetic anhydride.

5. 7 Pentane or light petroleum, boiling range 40 to 60°C.

5.8 Copper (II) sulphate, 70 g/l solution. Dissolve 70 g of copper (II) sulphate pentahydrate ($CuSO_4.5H_2O$) in 1 l of water.

5.9 Sodium sulphate, anhydrous.

6. APPARATUS

Usual laboratory equipment and

6.1 Analytical balance.

6.2 Conical flasks, capacity 500 ml, with ground joints and fitted with air condensers.

6.3 Glass micro-filtering device, as shown in figure 1. (Sea al so P.C. den Herder, Neth. Milk and Dairy J., 9 (1955), P. 261).

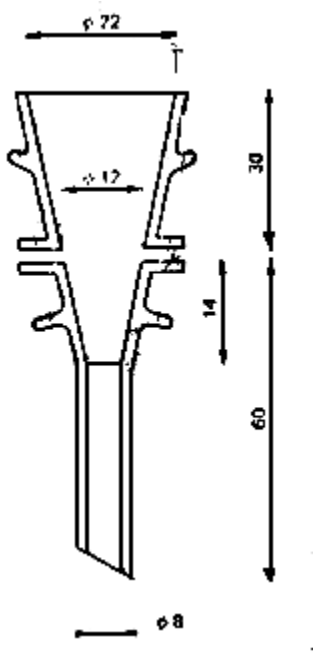

FIGURE 1 Micro filtering device

6.4 Melting point apparatus.

6.5 Melting point tubes, internal diameter 0,8 to 1,0 mm, length 50 nm.

6.6 Teat tubes, of heat-resistant glass, diameter 12 nm, length 35 mm.

6.7 Microscope slides and cover slips.

6.8 Ordinary or polarizing microscope, linear magnification 200X.

6.9 Drying oven, capable of being controlled at 102±2°C.

6.0 Glycerol bath, capable of being controlled at 130 to 145°C.

7. SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

8. PROCEDURE

8.1 Preparation of test sample

8.1.1 Butter - Melt about 50 g of the laboratory sample at a temperature below 50°C until the fat and water layers separate. Remove the fat layer by decantation and clarify the fat at a temperature of about 40°C by filtering it through a dry filter paper, taking care that no water passes on to the filter.

8.1.2 Milk and cream - Centrifuge the laboratory sample to obtain a cream having a fat content of about 40%. Churn the cream in a laboratory churn. Collect the butter lumps and proceed as described in 8.1.1.

8.1.3 Cheese - Grind the laboratory sample in a mortar with anhydrous sodium sulphate (5.9) until a granular mass is produced.
Extract the mass with pentane or light petroleum (5.7) (a continuous extraction apparatus may be used) and evaporate the solvent in a boiling water bath.

8.1.4 Condensed milk, evaporated milk and ice-cream - Add to the laboratory sample twice its volume of boiling water and heat the mixture on a boiling: water bath to a temperature of 75°C. Add an amount of copper (ll) sulphate solution (5.8) equal to one-tenth of the volume of the mixture and continue heating until the precipitate coagulates. Filter the precipitate through a filter paper and wash it with warm water until the filtrate is colourless. Carefully drain the precipitate, grind it in a mortar with anhydrous sodium sulphate (5.9) and proceed as described in 8.1.3.8.1.5 Dried milk - Grind the laboratory sample in a mortar with some water so as to obtain a clotted mass. Allow it to stand for about 15 min. Then add anhydrous sodium sulphate (5.9), grind again until a granular mass is produced and proceed as described in 8.1.3.

8.2 Preparation of the sterol digitonides

8.2.1 Weigh, to the nearest 0.1 g. about 15 g of the test sample (8.1) and transfer this test portion into a conical flask (6.2).

8.2.2 Add to the test portion 10 ml of potassium hydroxide solution (5.1) and 20 ml of ethanol (5.3).

8.2.3 Place the air condenser on the flask, heat the flask on a boiling water bath, swirling until the solution has become clear, and continue boiling for 30 min.

8.2.4 Add 60 ml of water and then 180 ml of ethanol (5.3) and raise the temperature to about 40°C.

8.2.5 Add 30 ml of the ethanolic digitonin solution (5.2), swirl and allow to cool. Place the flask in a refrigerator at about 5 C for about 12 h (conveniently overnight).

8.2.6 Collect the precipitate of sterol digitonide by filtration through a medium speed filter paper in a Büchner funnel (diameter 80 mm).

8.2.7 Wash the precipitate with water at about 5°C until the filtrate stops foaming, then once with 25 to 50 ml of ethanol (5.3) and once with 25 to 50 ml of diethyl ether (5.5).

8.2.8 Dry the filter paper and precipitate on a watch-glass in the drying oven (6.9), controlled at 102±2°C, for 10 to 15 min.

8.2.9 Fold the filter paper in two so that the precipitate comes off as a film and transfer the precipitate into a weighing bottle.

Note: If it is desired to know the content of sterols, weigh the precipitate in the weighing bottle to the nearest 0,001 g and calcultate this content, as a percentage by mass, using the formulas

$$\frac{0,25\ m_1}{m_0} \times 100 = \frac{25\ m_1}{m_0}$$

where:

m_0 is the mass, in grams, of the test portion (8.2.1);

m_1 is the mass, in grams, of sterol digitonide precipitate.

Express the result rounded off to the second decimal place.

8.3 Preparation of the steryl acetates and determination of the melting point

8.3.1 Transfer 0,1± 0,005 g of the sterol digitonide (8.2.9) to a test tube (6.6), add 1 ml of acetic anhydride (5.6) and heat the tube in the glycerol bath (6.10) between 130 and 145°C until the precipitate has dissulved. Do not use direct heat, since spattering may occur. Continue heating for 2 min and allow to cool to about 80 C.

8.3.2 Add 4 ml of ethanol (5.3), mix and heat slightly to dissolve any steryl acetate which may tend to crystallize out.

8.3.3 Filter the solution while still warm through a small medium speed filter paper impregnated with ethanol, and collect the filtrate in another test tube.

8.3.4 Heat the filtrate in the test tube carefully until it boils gently.

8.3.5 Keep the solution boiling and, while shaking the test tube vigorously, add carefully 1 to 1,5 ml of water drop by drop from a pipette until the steryl acetate is just about to precipitate yet remains in solution. Avoid superheating.

8.3.6 Add a few drops of ethanol (5.3) to redissolve any precipitated steryl acetates.

8.3.7 Allow to cool in air for 2 h and finally in ice water for 30 min.

8.3.8 Filter the crystallized steryl acetates on a small disk of hardened fast filter paper by using suction and the micro-filtering device (6.3), and rinse the crystals with 1 ml of ethanol (5.4).

8.3.9 Redissolve the crystal cake by heating it over a micro-burner in a test tube (6.6) with 1 ml of ethanol (5.3).

8.3.10 Allow to cool first in air for 15 min and then in ice water for 5 rain. Filter the crystallized steryl acetates as described in 8.3.8.

8.3.11 Repeat the operations described in 8.3.9 and 8.3.10. If necessary (see 9.3), repeat these operations twice more.

8.3.12 Dry the crystal cake on the paper first at about 30 C and then in the drying oven (6.9), controlled at 102±2°C, for 10 to 15 min.

8.3.13 Disintegrate the crystal cake, mix the crystals on a watch-glass and fill a melting point tube (6.5) to a height of about 3 mm. Determine the melting point in the melting point apparatus (6.4), raising the temperature in the last phase of the melting process at a rate of 0,5°c/min. Record as the melting point the reading on the thermometer, to 0,1 C, at the moment when the last crystal has just disappeared.

8.4 Microscopical examination of the sterols

Note; This examination will only be necessary when the melting point of the steryl acetate is found to be 115,5 °C or higher, but lower than 117,0 C (see 9.3.2).

8.4.1 Dissolve about 0,01 g of the steryl acetates (8.3.13) in 1 ml of ethanol (5.3) in a small test tube and add 1 or 2 drops of potassium hydroxide solution (5.1).

8.4.2 Heat on a boiling water bath until boiling begins and the steryl acetates dissolve.

8.4.3 Add 10 ml of water, transfer the solution to a 125 ml separating funnel and shake with 25 ml of diethyl ether (5.5).

8.4.4 After separation, drain and discard the aqueous layer.

8.4.5 Hash the ether layer with three 5 ml portions of water.

8.4.6 Transfer the ether layer to a 50 ml beaker and evaporate to dryness.

8.4.7 Dissolve the residue in 10 ml of 80% (v/v) ethanol (5.4). Place a drop of the clear solution on a microscope cover slip and let it spread over the slip. Wait until crystallization starts at the edges of the cover slip, then invert the slip and lay it on a microscope slide.

8.4.8 During further crystallization, examine the crystals under the microscope (6.8) at about 200 X linear magnification.

9. INTERPRETATION OF RESULTS

9.1 If the melting point of the steryl acetate is found to be between 114,0 and 115,5°C, the laboratory sample shall not be considered to contain vegetable fat.

9.2 If the melting point of the steryl acetate is found to be 117,0 C or higher, the laboratory sample shall be considered to contain vegetable fat.

9.3 If, however, the melting point of the steryl acetate is found to be 115,5°C or higher, but lower than 117,0 C, repeat the redissolving, recrystallization and filtration twice more (see 8.3.11), dry the crystal cake and determine the melting point as described in 8.3.12 and 8.3.13.

9.3.1 If the melting point of the steryl acetate is then found to be 117,0°C or higher, the laboratory sample shall be considered to contain vegetable fat.

9.3.2 If, however, the melting point of the steryl acetate is found to have remained 115,5°C or higher, but lower than 117,0°C, then subject the sterol crystals to microscopical examination as described in 8.4.

9.3.2.1 If, on microscopical examination, the sterol crystals are found to have only the form of a parallelogram with an obtuse angle of 100°, which is characteristic of cholesterol (sec figure 2), the laboratory sample shall not be considered to contain vegetable fat.

9.3.2.2 If, on microscopical examination, some of the sterol crystals are found to have an elongated hexagonal form with an apical angle of 108°, which is characteristic of phytosterols, or if some of the crystals have a re-entrant angle (swallow-tail), which is characteristic of mixtures of cholesterol and phytosterols (see figure 2), the laboratory sample shall be considered to contain vegetable fat.

10. SENSITIVITY OF THE TEST

The sensitivity depends upon the nature of the vegetable fat which may have been added, i.e. upon the content and composition of the phytosterol mixture present in the vegetable fat.

11. TEST REPORT

The test report shall give the melting point of the steryl acetates and the number of re-crystallizations, a description of the microscopical appearance of the sterol crystals, if relevant, and the method used. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional (see note to 8.2.9), as well as any circumstances that may have influenced the result.

The report shall include all details required for the complete identification of the sample.

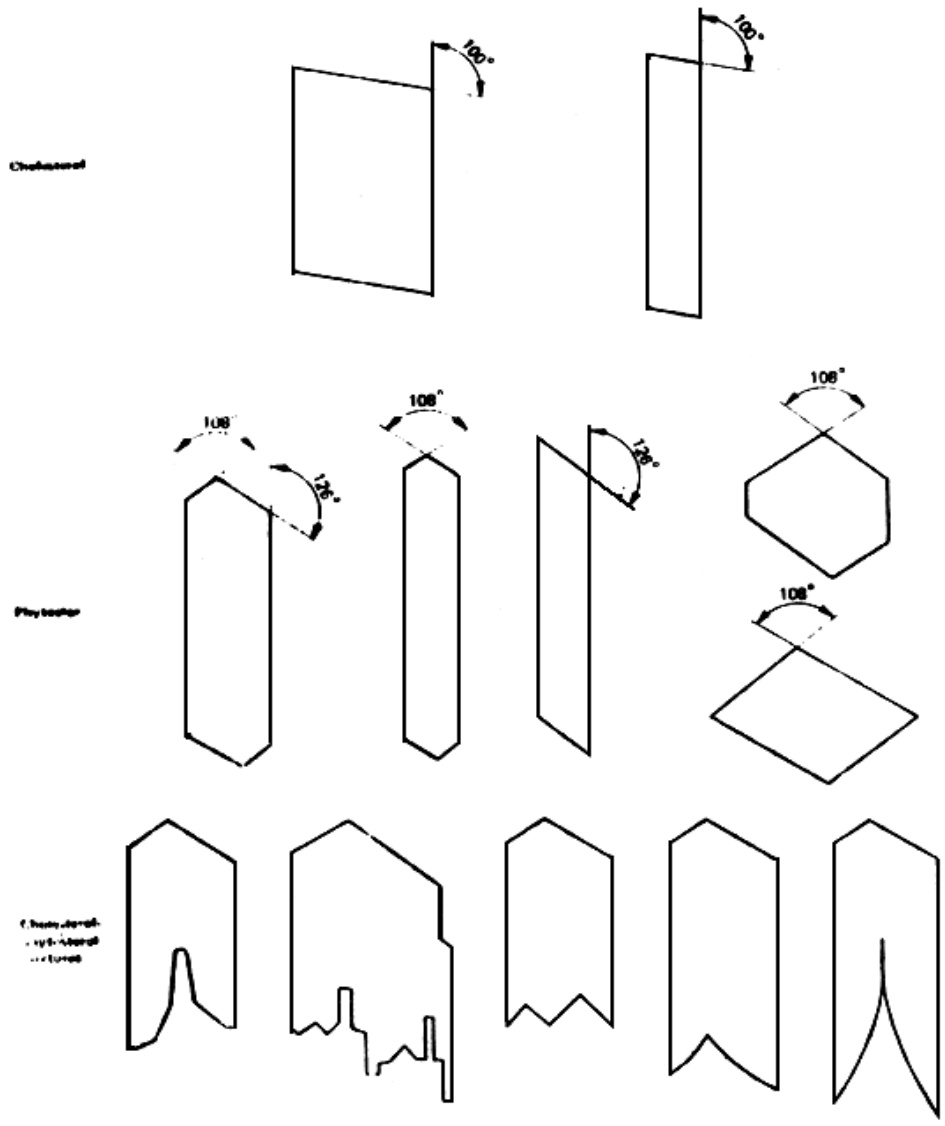

FIGURE 2 Crystal shapes of steryl

FAO/WHO STANDARD METHOD No. B-17

MILK FAT - DETECTION OF VEGETABLE FAT BY GAS-LIQUID CHROMATOGRAPHY OF STEROLS (REFERENCE METHOD)

1. SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method by gas-liquid chromatography (GLC) for the detection of the presence in milk fat of vegetable fats containing β -sitosterol. The limit of detection depends upon the β -sitosterol content of the added vegetable fat.

2. REFERENCES

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products" and B-16 Milk Fat -Detection of vegetable fat by the phytosteryl acetate test.

3. PRINCIPLE

Preparation of sterol digitonides as described in ISO 3595 and dissolution in a mixture of formamide and dimethylformamide. Extraction of the liberated sterols with pentane. Separation of the sterols by gas-liquid chromatography.

If, on the chromatogram, a peak with the retention time of β -sitosterol is obtained, the presence of vegetable fat in the fat sample under investigation is demonstrated. Peaks of other phytosterols may support this conclusion.

4. REAGENTS AND MATERIALS

All reagents shall be of analytical quality.

4.1 Formamide and dimethylformamide. mixture, in equal volumes.

4.2 n-Pentane.

4.3 Column packing: 2 to 4% loading of a methyl silicone gum rubber, stable up to at least 300°C, on a flux-calcined diatomaceous earth, acid washed and silanized, mesh size 80/100 (175 to 150 µm) or 100/120 (150 to 125 µm).

4.4 Sensitivity test solution; 1 mg of milk fat sterols in 1 ml of n-pentane, freshly prepared from milk fat (see 7.2).

4.5 Peak resolution test solution: 0,9 mg of rape seed oil sterols and 0,1 mg of milk fat sterols in 1 ml of n-pentane, both freshly prepared, from rape seed oil and milk fat respectively (see 7.2).

4.6 Reference test solution: 1 mg of soyabean oil sterols in 1 ml of n-pentane, freshly prepared from soyabean oil (see 7.2).

4.7 Carrier gas: nitrogen.

4.8 Hydrogen.

4.9 Oxygen or air.

5. APPARATUS

Usual laboratory equipment and

5.1 Gas chromatograph, fitted with hydrogen flame ionization detector, silver or glass injection system, or direct-on-column injection device, and recorder.

5.2 Gas chromatographic column, glass, U-shaped or coiled, length 100 to 200 cm, inside diameter 2 to 4 mm.

Notes Stainless steel should not be used as some types cause false results by deterioration of sterols.

5.3 Micro-syringe. capable of delivering a volume of up to 5 or 10 µl.

6. SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

7. PROCEDURE

7.1 Preparation of test sample

See FAO/WHO Standard B-16 "Milk Fat - Detection of vegetable fat by the phytosteryl acetate test".

7.2 Preparation of sterols

Dissolve about 10 mg of sterol digitonide, prepared as described in ISO 3595, in 0,5 ml of the formamide and dimethylformamide mixture (4.1) in a small test tube, if necessary with gentle heating. Add 2,5 ml of n-pentane (4.2) to the cooled solution, stopper the tube and shake. Let the layers separate and use the clear upper pentane layer, containing the liberated sterols, for gas chromatographic analysis. This solution contains about 1 g of sterol per millilitre.

7.3 Gas-liquid chromatographic conditions

Column temperature: 220 to 250°C.

Temperature of injection system, if it can be separately heated: 20 to 40°C above column temperature.

Nitrogen flow rate: 30 to 60 ml/min.

Disconnect the detector and operate new columns under these conditions for 16 to 24 h co that equilibrium is reached. Connect the detector, ignite the flame and regulate the hydrogen and oxygen or air flow rates so as to obtain appropriate flame height and de Sector sensitivity. Start the recorder at a suitable chart speed and adjust the zero setting and attenuator. If the base line is steady, the apparatus is ready for use.

7.4 Sensitivity test

Inject, 3 to 5 µl of the sensitivity test solution (4.4). Only one distinct peak of cholesterol will appear on the gas chromatogram. Adjust the attenuator so as to obtain approximately full-scale deflection on the recorder (see figure 1).

7.5 Peak resolution test

Inject 3 to 5 µl of the peak resolution test solution (4.5). Peaks of cholesterol, brassicasterol, campesterol and β -sitosterol will appear on the gas chromatogram (s e figure 2). Measure the retention distances (distance from sample injection to maximum peak height) of the peaks, d_{CH} for cholesterol, d_B for brassicasterol, d_C for campesterol, and d_s for β-sitosterol, and the peak base widths (retention dimension between inter sections of base line with tangents to the points of inflection on the front and rear sides

of the peak), W_{CH}, for cholesterol and WB for brassicasterol. The peak resolution, PR = 2 (d_B - d_{CH}) / (W_B + W_{CH}), shall be at least 1.

Notes To facilitate the measurement of the base widths, extend the longest straight portion of each side of the peak until it intersects the base line; the base width will be the distance between the points of intersection corresponding to each of the two sides.

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

7.6 Reference test

Inject 3 to 5 µl of the reference test solution (4.6). Peaks of campesterol, stigmasterol, and β-sitosterol will appear on the gas chromatogram (see figures 2 and 3). Measure the retention distances of the peaks, d_C for campesterol, d_{ST} for stigmasterol, and d_s for β-sitosterol.

Calculate the relative retention times, which are approximately as follows:

cholesterol	1,00 (about 15 min)
brassicasterol	1,13 to 1,15
campesterol	1,32 to 1,34
stigmasterol	1,44 to 1,46
β-sitosterol	1,66 to 1,68

7.7 Analysis

Switch the attenuator to an attenuation factor four times (usually two steps) lower and inject the same volume of the sterol solution (7.2) as used in 7.4. Record the gas chromatogram.

8. EXPRESSION OF RESULTS

If, on the gas chromatogram, a peak with the relative retention time of β-sitosterol and a height of at least 2% of the full scale is observed, the presence of β-sitosterol is indicated and the laboratory sample under investigation, from which the sterols have been isolated, is considered to contain vegetable fat.

The presence on the gas chromatogram of peaks of other phytosterols such as campesterol or stigmasterol may support the conclusion.

9. SENSITIVITY

The presence of β -sitosterol contents as low as 0,5% can be detected by the method described in this International Standard. The limit of detection of vegetable fat in milk fat cannot be given since this depends on the β-sitosterol content of the fat used for admixture, i.e. upon the nature of the fat or mixture of fats added to the milk fat.

10. TEST REPORT

The test report shall mention the method used and the results obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the results.

The report shall include all details required for the complete identification of the sample and shall be accompanied by the recorded gas chromatogram.

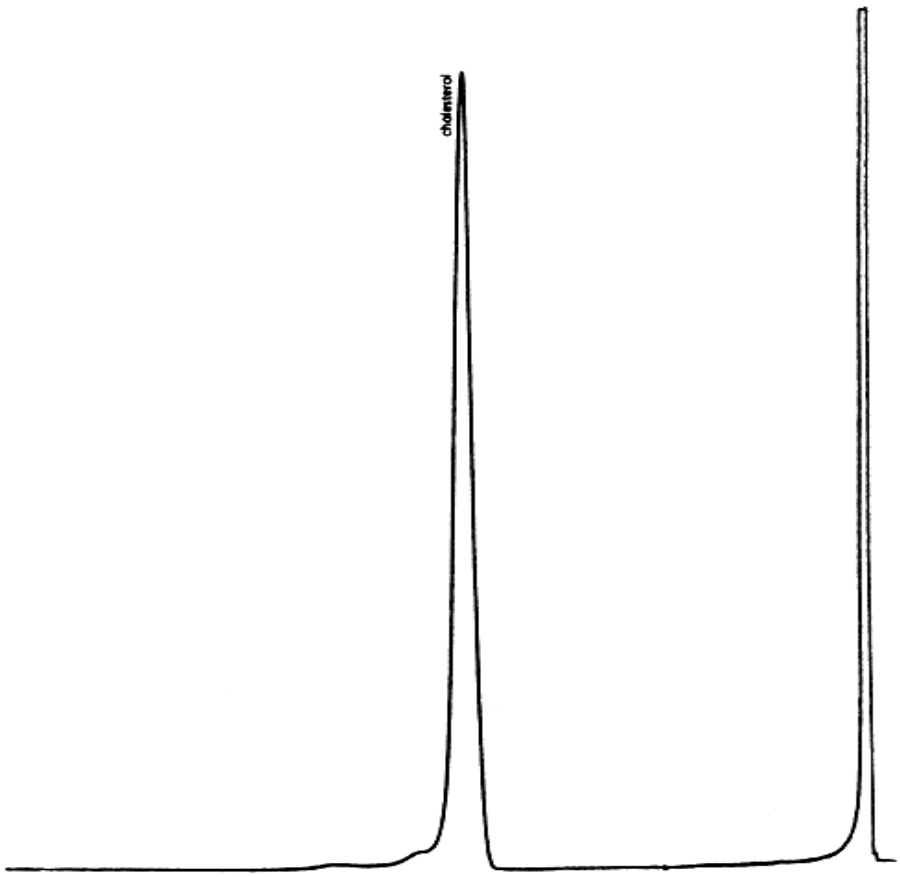

FIGURE 1 GLC of milk 1st sterols

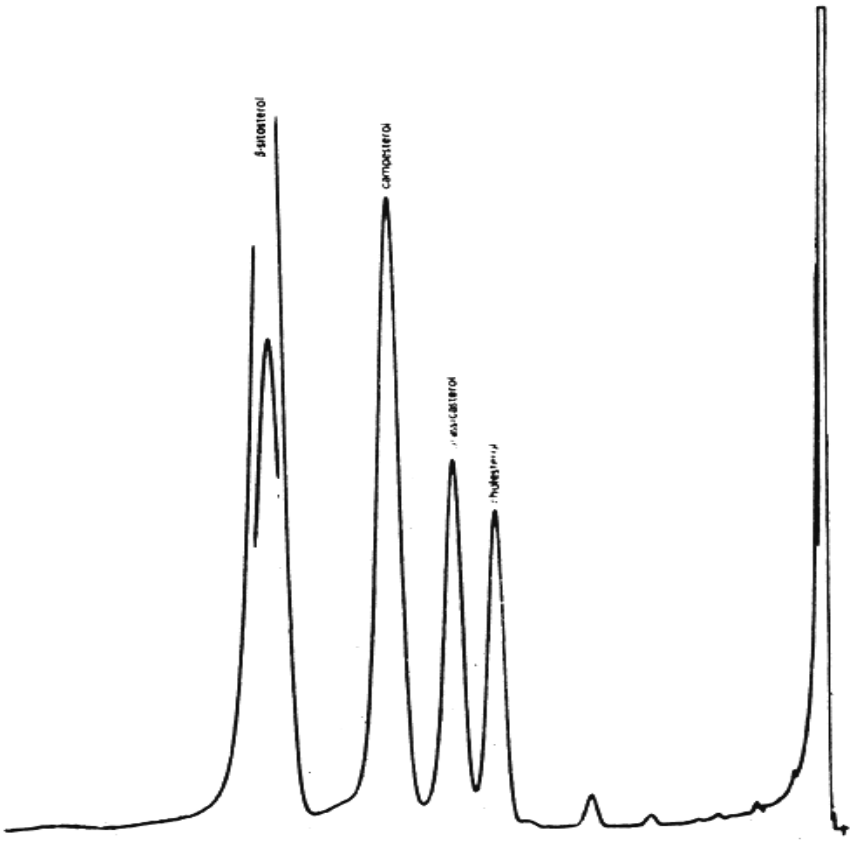

FIGURE 2 – GLC of rape seed oil sterols and cholesterol

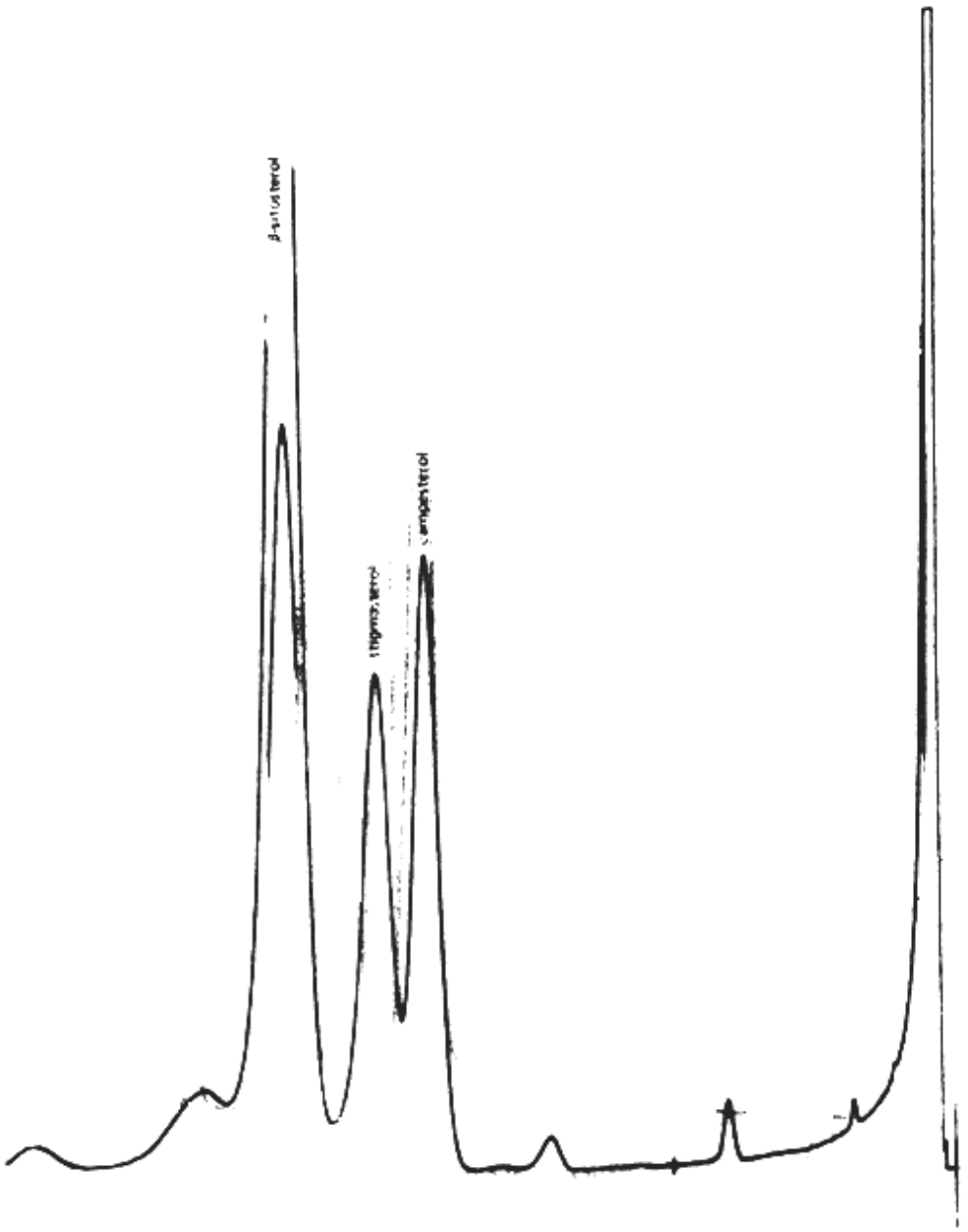

FIGURE-3 GLC of soysbeen oil sterols

FAO/WHO STANDARD METHOD No. B-18

CHEESE - DETERMINATION OF CHLORIDE CONTENT (REFERENCE METHOD)

1. SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the chloride content of cheese.

The method is applicable to all cheeses containing at least 0,5% of chloride.

2. REFERENCE

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

3. DEFINITION

Chloride content of cheese: The substances determined by the procedure specified. The chloride content may be expressed as a percentage by mass of Cl or sodium chloride or any other chloride used.

4. PRINCIPLE

Destruction of the organic matter of the cheese by means of potassium permanganate and nitric acid, and determination of the chloride content by argentimetric titration in nitric acid solution, in the presence of ammonium iron (lll) sulphate as indicator.

5. REAGENTS

All reagents used shall be of analytical reagent (quality.

5.1 Silver nitrate, approximately 0,1 N solution, standardized to the fourth decimal.

5.2 Potassium or ammonium thiocyanate, 0,1 N solution, standardized to the fourth decimal.

5.3 Ammonium iron (III) sulphate, saturated solution.

5.4 Nitric acid, P_20 1,40 to 1,42 g/ml, which corresponds to 66,9 to 71,6% (m/m) HNO_3.

5.5 Potassium permanganate, saturated solution.

5.6 Oxalic acid or glucose.

5.7 Water, not containing any impurity likely to affect the determination.

6. APPARATUS

6.1 Balance.

6.2 Conical flask, capacity 300 ml.

6.3 Pipette, calibrated to deliver 25 ml, conforming to ISO/R 648.

6.4 Graduated cylinders, capacities 15, 25 and 100 ml.

6.5 Burette, graduated in 0,1 ml, capacity 50 ml, conforming to ISO/R 385.

6.6 Suitable grinding device.

7. SAMPLING

See FAO/WHO Standard B-1 "Sampling Methods for Milk and Milk Products".

8. PROCEDURE

8.1 Preparation of the test sample [1/]

[1/] Special requirements for the preparation of the test sample of any type or variety of cheese might be laid down in national standards.

Before analysis, remove the rind or smear the mouldy surface layer of the cheese so as to give a test sample representative of the cheese such as it is usually consumed.

Grind the sample by means of an appropriate device (6.6); mix the ground mass quickly and grind if possible a second time and mix again thoroughly, Clean the grinding device after each sample. If the sample cannot be ground, mix it thoroughly by intensive kneading.

Transfer the test sample to an air-tight container until the analysis, which shall be carried out on the same day. If delay is inevitable, take all precautions to ensure proper conservation and to prevent condensation of moisture on the inside surface of the container.

Cheese in brine shall be sampled by taking fragments of at least 200 g each along with sufficient brine to cover the cheese in the sample container. Prior to analysis, place the sample on filter paper for 1 to 2 h.

8.2 Test portion

Weigh, to the nearest 0,001 g, about 2 g of the test sample into the conical flask (6.2).

8.3 Determination

8.3.1 Add, by means of the pipette (6.3), 25 ml of silver nitrate solution (5.1), then add, by means of a graduated cylinder (6.4), 25 ml of nitric acid (5.4) and mix thoroughly.

8.3.2 Heat to boiling, add approximately 10 ml of potassium permanganate solution (5.5) and keep the reaction mixture boiling gently.

When the reaction mixture decolorizes, add more potassium permanganate solution; generally another 5 to 10 ml are sufficient. The presence of excess permanganate (brown colour) indicates that destruction of the organic matter is complete. Remove the excess by the addition of a small amount of oxalic acid or glucose (5.6).

8.3.3 Add 100 ml of cold water (5.7) and 2 ml of ammonium iron (III) sulphate solution (5.3) and mix thoroughly.

8.3.4 Immediately titrate the excess silver nitrate with the thiocyanate solution (5.2) until the solution shows a red-brown colour which persists for about 30 s.

8.3.5 Carry out a blank test using 2 ml of water in place of 2 g of cheese.

8.3.6 Carry out two determinations on the same test sample.

9. EXPRESSION OF RESULTS

9.1 Method of calculation and formula

Calculate the chloride content, as a percentage by mass, by means of the formula

$$\frac{(V_1 - V_2) \times f \times T}{m}$$

where

V_1 is the volume, in millilitres, of thiocyanate solution used for the blank test;

V_2 is the volume, in millilitres, of thiocyanate solution used for the test portion;

T is the normality of the thiocyanate solution;

m is the mass, in grams, of the test portion;

f is the factor for expressing the result as a percentage of any chloride. The numerical values are, for example:
f = 3,55 for expression as % Cl
f = 5,85 for expression as % NaCl
f = 7,46 for expression as % KCl

Take as the result the arithmetic mean of the two determinations if the requirement concerning repeatability (9.2) is satisfied. Report the result to the second decimal place.

9.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,04 g of Cl^- (or the equivalent quantity of chloride used) per 100 g of the cheese.

10. TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details required for the complete identification of the sample.