

John Németh

REPORT OF THE

**FIFTH SESSION OF THE COMMITTEE
OF GOVERNMENT EXPERTS
ON THE CODE OF PRINCIPLES
CONCERNING MILK AND
MILK PRODUCTS**

Held in Rome, Italy
2-6 April 1962



FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

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Food and Agriculture Organization of the United Nations
June 1962 Rome Italy

LIST OF PARTICIPANTS

DELEGATES

AUSTRALIA

Mr. Joseph R. BROWN
Senior Dairy Produce Inspector
Department of Primary Industry
c/o Australian High Commissioner
Australia House
The Strand
London (United Kingdom)

AUSTRIA

Dipl. Ing. K.H. RAUSCHER
Bundesministerium für Land und Forstwirtschaft
Leiter der Abteilung Milchwirtschaft
Stubenring 1
Vienna 1 (Austria)

BELGIUM

Mr. P.R.V. JAMOTTE
(Expert)
Station Laitière d'Etat
Gembloux (Belgium)

Mr. J. SERVAIS
Director
Ministère de l'Agriculture
3 rue du Méridien
Brussels (Belgium)

DENMARK

Mr. P. KOCK HENRIKSEN
Director
Federation of Danish Dairy Associations
Mejerikontoret
Aarhus (Denmark)

Mr. K.P. ANDERSEN
Government Institute for Research in Dairy Industry
Hillerød (Denmark)

Mr. H. METZ
Director
Government Control of Dairy Products
Christians Brygge 22
Copenhagen K. (Denmark)

Mr. C. VALENTIN HANSEN
Agricultural Attaché
Danish Embassy
Viale del Policlinico 129A
Rome (Italy)

FRANCE

Mr. A. DESEZ
Inspecteur divisionnaire de la-Répression des
Fraudos
Ministère de l'Agriculture
42 bis rue de Beurgogne
Paris 7e. (France)

FEDERAL REPUBLIC OF
GERMANY

Dr. H.H. BEYSEN
Chief, Dairy Department
Ministry of Food, Agriculture and Forestry of
Schleswig-Holstein
Düsternbrookerweg 24
Kiel (Federal Republic of Germany)
Mr. Waldemar GODBERSEN
Bundesministerium für Ernährung und Landwirtschaft
Bonn (Federal Republic of Germany)

INDIA

Mr. KRISHAN MAHARAJ
Agricultural Attaché
Embassy of India
Via F. Denza 36
Rome (Italy)

ITALY

Prof. Scipione ANSELMI
Istituto Superiore di Sanità
Viale Regina Elena 299
Rome (Italy)
Mr. Rodolfo BARBATO
Confederazione Generale Agricola Italiana
101 Corso Vittorio
Rome (Italy)
Mr. Giovanni ELISEO
Chief, Foreign Commerce Service
Via Muzio Clemente 70
Rome (Italy)
Dr. Guido MARZANTO
Director of Division
Ministero Agricoltura e Foreste
Via XX Settembre
Rome (Italy)
Dr. Giovanni MENAPACE
Via Curtatone 3
Rome (Italy)
Mr. Antonio MASUTTI
Director
Assolatte
Via Muzio Clemente
70 Rome (Italy)
Dr. Giacomo PITTONI
Via di Santa Costanza 7
Rome (Italy)
Mr. Vincenzo SEPE
Ministero de Agricoltura e Foreste
Via XX Settembre
Rome (Italy)

Dr. Francesca ZAFARANA
FAO Committee
c/o Ministero Agricoltura
Via XX Settembre
Rome (Italy)

JAPAN
Mr. N. FUJII
Technical Official of Ministry of Agriculture
Livestock Bureau
Ministry of Agriculture and Forestry
Tokyo (Japan)

Mr. Rynichi IWASHITA
First Secretary, Japanese Embassy
Via Barnaba Oriani 46
Rome (Italy)

LUXEMBOURG
Mr. Norbert WILTGEN
Ingénieur Chimiste
Station de Chimie Agricole de l'Etat
Ettelbruck (Luxembourg)

NETHERLANDS
Mr. Th. C.J.M. RIJSSENBEK
Director of Animal Husbandry
Ministry of Agriculture
1 v.d. Beschstrasse 4
The Hague (Netherlands)

Dr. C. SCHIERE
Director, Inspection Institute for Milk and Milk
Products
L.V. Meerderveert
The Hague (Netherlands)

Dr. J.G. VAN GINKEL
Director, Government Dairy Station
Leiden (Netherlands)

Mr. H.P.H. RADIER
Secretary, Dairy Marketing Board
Hoenstraat 5
The Hague (Netherlands)

Mr. G. HIBMA
Secretary, Centrale Zuivelcommissie
Jan Van Nassaustraat 85
The Hague (Netherlands)

NEW ZEALAND
Mr. J.J. WALKER
Inspector of Dairy Products
St. Olaf House
Tooley Street
London S.E.1. (United Kingdom)

NICARAGUA
Mr. Eduardo ARGUELLO CERVANTES
Ambassador
Embajada de Nicaragua
Rome (Italy)

NORWAY	Prof. Rasmus MORK Vollebekk (Norway)
PAKISTAN	Mr. Nazir AHMED Agricultural Attaché Embassy of Pakistan Lungotevere dello Armi 22 Rome (Italy)
POLAND	Mr. Mieczyslaw GLODZ Vice-President Polish Dairy Cooperative Association Hoza 66/68 Warsaw (Poland) Dr. Imbs BEGUSLAW Department of Dairy Technology Agriculture School Olstyn (Poland)
SPAIN	Mr. G. ESCARDO PEINADOR Ing. Agronomo Spanish Embassy Via Lima 23 Rome (Italy) Mr. Santiago MATELLANA VENTURA Secretary, Spanish Committee of the International Dairy Federation Conde Valle Suchil 10 Madrid (Spain)
SWEDEN	Mr. W. LJUNG Director Svenska Mejeriernas Riksförening Postfack Stockholm (Sweden)
SWITZERLAND	Dr. E. ACKERMANN Monbijoustrasse 36 Bern (Switzerland) Mr. J. BURKHALTER Dipl. Ing, Agr. Wabersackorstrasse 37a Liefcefeld-Bern (Switzerland) Dr P. BERGEAUD A.F.I. CO., S.A. Tour de Peilz Vaud (Switzerland) Mr. Oscar LANGHARD Managing Director Swiss Cheese Union Monbijoustrasse 128 Bern (Switzerland)

UNITED KINGDOM

Mr. L.C.J. BRETT
114 Reigate Road
Ewell
Surrey (United Kingdom)

Mr. E. CAPSTICK
34 Palace Court
London W.2. (United Kingdom)

Mr. J.H.V. DAVIES
Principal, Food Standards Division
Ministry of Agriculture, Fisheries and Food
Great Westminster House
Horseferry Road
London S.W.1. (United Kingdom)

Mr. F.C. WHITE
Principal, Milk Products and Welfare Foods Branch
Ministry of Agriculture, Fisheries and Food
Great Westminster House
Horseferry Road
London S.W.1, (United Kingdom)

UNITED STATES OF AMERICA

Dr. W. HORWITZ
Adviser
Chief, Food Research Branch
Food and Drug Administration
Washington 25 D.C, (U.S.A.)

Mr. H.E. MEISTER
Delegate
Chief, Inspection and Grading Branch
Dairy Division
Agricultural Marketing Service
US Department of Agriculture
Washington 25 D.C. (U.S.A.)

Mr. D.R. STROBEL
Alternate. Delegate
Deputy Director
Dairy and Poultry Division
Foreign Agricultural Service
US Department of Agriculture
Washington 25 D.C. (U.S.A.)

OBSERVERS

ARGENTINE

Mr. V.C. BRUNINI
Embajada do la Ropublica Argentina
Rome (Italy)

Dra. R. HUERIN
Instituto de Racionalización de
Materiales J.R.A.M.
Via Chile 1902
Buenos Aires (Argentine)

ECUADOR

Mr. H. GUARDERAS
Embassy of Ecuador
Via Barnaba Tortolini 36 A
Rome (Italy)

SENEGAL

Mrs. BASSE
Sénégal Embassy
299 Via Nomentana
Rome (Italy)

DAIRY SOCIETY INTERNATIONAL

Mr. W.P. CROCKER
c/o The Nestlé Company Limited
St. George's House
Wood Street
London E.C.2. (United Kingdom)

EUROPEAN ASSOCIATION FOR ANIMAL PRODUCTION

Dr. K. KALLAY
Secretary General
Corso Trieste 67
Rome (Italy)

EUROPEAN COMMITTEE ON MILK-BUTTERFAT RECORDING

Dr. K. KALLAY
Secretary General
Corso Trieste 67
Rome (Italy)

INTERNATIONAL DAIRY FEDERATION

Prof. A.M. GUERAULT
President
10 rue Ortélius
Brussels 4 (Belgium)

INTERNATIONAL FEDERATION OF MARGARINE ASSOCIATIONS

Mr. M.E.J. HUMANS
Secretary General
Raamwog 44
The Hague (Netherlands)

PERMANENT COUNCIL OF THE STRESA CONVENTION

Dr. F. ZAFARANA
National FAO Committee
Ministero Agricoltura
Via XX Settembre
Rome (Italy)

INTERNATIONAL ORGANIZATION FOR STANDARDISATION (ISO) (TC/34 SO/5)

Dr. J.G. VAN GINKEL
Director
Government Dairy Station
London (Netherlands)

FAO PERSONNEL

Dr. K.V.L. KESTEVEN
Director
Animal Production and Health. Division

Dr. Hans PEDERSEN
Chief, Dairy Branch
Animal Production and Health Division

Mr. E. LANCELOT
Dairy Specialist
Dairy Branch
Animal- Production and Health Division

Mr. P.H. TOWNSHEND
Legal Research Officer
Rural Legislation Branch

OFFICERS OF THE COMMITTEE AND SUB-COMMITTEE

The Committee elected the following officers:

CHAIRMAN Mr. H.E. MEISTER (United States of America)

VICE-CHAIRMAN Dr. W. LJUMG (Sweden)

Sub-Committee A on Standards:

CHAIRMAN: Mr. P.C. WHITE (United Kingdom)

Sub-Committee B on Methods of Sampling and Analysis:

CHAIRMAN: Dr. P. BERGBAUD (Switzerland)

SUMMARY OF DISCUSSIONS AND PROPOSALS OF THE COMMITTEE

1. At its Fifth Session in April 1962, the Committee reviewed the replies received from the following 35 countries to the requests summarised in paragraph 21 of the Report of its Fourth Sessions

Australia	Netherlands
Austria	New Zealand
Belgium	Norway
British Guiana	Pakistan
Cambodia	Poland
Canada	Rhodesia and Nyasaland
Chile	Spain
Denmark	Syrian Arab Republic
Fiji	Sweden
Finland	Switzerland
France	Tanganyika
Federal Republic of Germany	Thailand
Ghana	United Arab Republic
Hong Kong	United Kingdom
Ireland	United States of America
Japan	Vietnam
Kenya	Zanzibar
Malagasy Republic	

2. The Committee noted that acceptances of the Code of Principles had now also been received from the following countries:

Argentine
British Guiana
Kenya
Nepal
Syrian Arab Republic

The number of acceptances of the Code had thus risen to 50. The Government of U.S.A. indicated that 34 States of the Union had accepted the Code and that there had been no negative replies.

In addition, the Government, of Burma had communicated its acceptance of the Code. Burma therefore passed from Acceptance Group IV to Acceptance Group III. Full details of all acceptances will be set out in the Third Edition of the Code of Principles, which will form a separate part of this Report.

3. At its Fourth Session (Report, paragraph 5) the Committee had requested the Secretariat to solicit such supplementary information from participating countries as might appear necessary for submission to it and to continue its efforts to achieve ever-wider acceptance of the Code. The Committee noted with satisfaction that the Secretariat had taken active steps to achieve this goal and that further new acceptances were expected in addition to the five already officially communicated and noted in paragraph 2 above. The Secretariat had also been requested to distribute to governments any observations, which might be received from them upon the application of the Code in practice. The Committee approved the action taken by the Secretariat in this respect and requested it to continue its work in the same manner as well as to report back to the Committee at its next Session. By such means, the Committee believed, the

Code of Principles would become an evermore-effective instrument at the service of the world dairy industry.

4. The Committee gave particular attention to the possibility of obtaining acceptance of the Code by the one government, which had indicated that it was unable at present to accept it. The Committee therefore requested the Secretariat to urge upon this Government the importance of accepting the Code and of associating itself with the other 50 Governments which had already done so. In this way acceptance of the Code would be unanimous throughout the world.

5. The Committee noted that 19 governments had accepted Standard Ho. 5 (Dried Milk). Consequently the Standard could be considered as adopted and would be published in the Third Edition of the Code of Principles together with any details of more stringent national requirements concerning this product. The Committee noted with particular satisfaction that several governments had announced during the Fifth Session that their national legislation had already been or would shortly be amended to conform with this Standard.

6. The Committee noted that 20 governments had accepted Standard No. 01 (Sampling Methods) and that a further 8 governments had agreed to give the Standard favourable consideration. Consequently the Standard could be considered as adopted; with the exception of the section on sampling of Cheese, and would be published in the Third Edition of the Code of Principles. In respect of the section on Sampling of Cheese, the Committee considered the draft submitted by the International Dairy Federation (IDF) (Standard FIL-IDF-2 % 1958), taking into account the comments received from the International Organisation for Standardization (ISO). A revised text was drawn up and was now submitted to Governments for acceptance. The text is given in Appendix A of this Report.

7. The Committee noted that the following Standards had been accepted by the number of governments indicated in brackets against each:

<u>Standard No. 02</u>	<u>Standard method for the determination of the fat content of milk powder according to Röse-Gottlieb</u>	(20)
<u>Standard No. 03</u>	<u>Standard method for the determination of the fat content of cheese according to Schmid-Bendzynski-Ratzlaff</u>	(20)
<u>Standard No. 04</u>	<u>Standard method for the determination of acidity in butterfat</u>	(19)
<u>Standard No. 05</u>	<u>Standard method for the determination of the refractive index of butterfat</u>	(20)

These Standards could thus be considered as adopted and would be published in the Third Edition of the Code of Principles.

8. Notwithstanding that the Committee noted that 19 governments had accepted Standard Ho. 06 (Standard method for the determination of the iodine value of butterfat according to Wijs), publication of this Standard would be deferred until the next Session, since the International Union for Pure and Applied Chemistry was now considering standard methods applicable to all fats and oils. It was expected that the information from this Organization would be available for the next Session of the Committee.

9. In order that all standard methods of analysis published under the Code conform to the ISO skeleton model for such standards, the Committee requested IDF in conjunction with ISO to submit a revised presentation of the Standards already adopted, before the Sixth Session of the Committee, so that the now version could be published in the Fourth Edition of the Code in 1963, as well as of the draft Standards submitted for preliminary consideration of the Committee (see paragraph 11 below). The Committee understood that in future all draft methods of analysis submitted by IDF for its consideration would conform to this presentation.

10. The Committee discussed in detail the draft Standards for Cheese set out in Appendix H of the Report of its Fourth Session, in the light of comments received from governments and from IDF. The Committee adopted the Standard Ho. 6 (General Standards for Cheese) set out in Appendix B, which is now submitted to Governments for acceptance, together with the following comments:-

- (a) The delegations of Denmark and Sweden were concerned about the possibility that consumers might be misled where low fat cheeses would be exempted from marking of the fat content in accordance with paragraph 1.4.2.
- (b) These general Standards foresee the establishment of individual standards for varieties of cheese entering international trade. The Committee fully realized that some time would elapse before such standards could be elaborated, but drew attention to the fact that the general Standards as now adopted were fully valid even though certain provisions would cease to 'apply when' international standards eventually came into force.
- (c) The Committee drew attention to the fact that paragraph 1.4.3 of the general Standards did not prejudice the minimum fat content of full fat cheeses for which an international standard might be elaborated in the future under the Code of Principles.
- (d) The Delegation of Italy proposed that paragraph 1.4.2 should contain a further exception covering cheeses such as "Parmigiano Reggiano", "Grana Padano", "Pocorino Romano", "Asiago", "Montasio", which were covered by Italian legislation laying down methods of production, standards of composition and presentation.
- (e) The Committee considered that the elaboration of individual international standards for the different varieties of cheese should be dealt with in the following manners any country producing a variety of cheese entering international trade could submit a draft international compositional standard to FAO. At the same time the government concerned should send a copy to all delegations which attended the last Session of the Committee (see paragraph 18 below). IDF would be kept informed by FAO and would be invited to send its comments upon the draft to FAO. The Committee would then consider the proposed standard together with the comments of governments and of IDP. In accordance with the Committee's normal procedures, all such standards adopted by the Committee would be submitted to governments for acceptance prior to their publication with the Code.

- (f) In order to accelerate work at its next session, the Committee further recommended that IDF should be provided with all the information submitted by governments concerning their national cheese standards when commenting on the Report of the Fourth Session. IDF should be requested to make proposals available to the Committee as soon as possible upon:-
- (1) requirements as to composition and other characteristics which should be included in individual international cheese standards;
 - (2) the criteria to be used by the Committee in considering requests by governments for such international standards.
- (g) Finally, governments should be requested in making their comments upon the general Standards for cheese set out in the attached Appendix B, to indicate the cheese varieties for which they have national standards that they would propose should be made the object of individual international standards.

11. The Committee made a preliminary survey of the following draft standards submitted by IDF:-

IDF-FIL-1	1955	Determination of the fat content of normal liquid milk according to Röse-Gottlieb.
IDF-FIL-4	1958	Determination of the dry matter content of cheese.
IDF-FIL 10	1960	Determination of the moisture content of butter.
IDF-FIL 12	1960	Determination of the salt content of butter.
IDF-FIL 13	1960	Determination of the fat content of evaporated and of sweetened condensed milk according to Röse-Gottlieb.

In the light of discussions held during the Session upon comments received from ISO, the Committee made a number of change in the IDF texts. The draft standards set out in Appendices C,D,E,F and G to this Report are now submitted to governments for detailed comments.

The Committee requested governments to give particular attention to the following points:

IDF Standard 4 - 1958 - Determination of the dry matter of cheese

- 1 - Is the use of a dessicator considered necessary?
- 2 - Is the use of sand considered necessary?
- 3 - A full description of the preparation of the sample is necessary.
- 4 - Use of air oven and/or vacuum oven.
- 5 - Details of drying techniques used or recommended.

IDF Standard 10 - 1960 - Determination of the moisture content of butter

- 1 - Is the use of a desiccators considered necessary?
- 2 - Is the use of pumice considered necessary?
- 3 - Preparation of the sample.

IDF Standard 12 - 1960 - Determination of the salt content of butter

- 1 - Use of nitric acid for destruction of nitrogenous matter.
- 2 - Use of an indicator other than chromate.
- 3 - Temperature of titration.

Should further comments be received from ISO they will be distributed to governments by the Secretariat.

12. The subject of skim milk to which vegetable fat had been added was again discussed (see Report of the Fourth Session, paragraph 4). The question at issue was whether or not brand names or marks which had been established for milk and milk products could be used on products in which the milk fat had been replaced by other fats. The Committee agreed that this subject should be placed on the agenda of the next Session and further urged all governments to comment in detail upon the matter in all its many aspects.

13. (a) The Committee received a further annual statement by the President of IDF giving details of the Federation's work on the preparation of basic standards for milk hygiene. The Committee again noted the progress being made and requested a further statement to be made, available to it at its next Session. The Committee underlined in particular the need to establish codes of practice covering hygiene requirements for dairy plants as well as the methods of inspection which should apply to each stage of the manufacturing process in order to satisfy these minimum requirements. Such codes of practice would be particularly useful to developing countries.
- (b) The Committee was informed of the activities of the Joint FAO/WHO Committee of Experts on Milk Hygiene, and on publications now being prepared on this subject, namely a monograph on milk hygiene, on guide lines for the erection of milk plants as well as the FAO handbook already published on "Principles of Milk Legislation". The Committee noted the importance in this connection of the seminars organized by IDF such as that held in Berne in 1960 on keeping quality of milk, the results of which were published in the Annual Bulletin of the Federation. The Committee was also informed that the Government of the United States of America, principally through the United States Department of Agriculture and the United States Public Health Service, had published* bulletins on Hygienic Milk Production, Milk Plant Construction and related subjects. The Committee noted that such information could be extremely useful to interested governments. The Committee also took note with satisfaction of the possibilities of further collaboration with WHO which would follow from the new Joint FAO/WHO Program on Food Standards, recently approved by the two Organizations.

* The Secretariat understands that these publications can be obtained from the USDA and the USPH, at Washington 25 D.C., U.S.A.

14. The Committee affirmed the view it expressed at its Fourth Session (Report, paragraph 9) that consideration of product grading under the Code of Principles program was premature at the present stage. Such work should only be undertaken once international standards of composition for the principal milk products had already been elaborated.

15. At its Fourth Session (Report paragraph 6) the Committee considered coordination between IDF, ISO and American Organization of Agricultural Chemists (AOAC) within the framework of the Code of Principles program. The Committee heard statements in this respect by the President of IDF and the Secretary of ISO TC/34 SC/5. The Committee noted with approval that effective steps had already been taken between the two organizations respecting their mutual collaboration and that this cooperation was to be developed further during the coming year with a view to permitting the two organizations to provide joint proposals for draft standards on all subjects of common interest. In the case of AOAC, the Committee was informed that steps were now being taken to enable that body to collaborate in the preparation of draft standards for consideration under the Code of Principles program. It was expected that these arrangements would be completed before the Committee's next Session,

16. The Committee considered a proposal by the Secretariat to improve the numbering systemic of standards published under the Code of Principles and to ensure identity of text and numbering between these standards and the corresponding standards published by IDF. The Committee agreed that its standards should be published in two series, Series A for Standards of Composition and Series B for Methods of Sampling and Analysis, the letter A or B preceding the number of each standard. It also agreed that the year in which each standard was adopted should accompany the number. In this way the Standards numbered 01, 02, etc... until now would henceforth be published as B.1, B.2, etc..., and these numbered 1, 2, 3, etc... as A.1, A.2, A.3, etc... The Committee recommended that the IDF print a note on each of its standards submitted for consideration under the Code of Principles program indicating clearly that such submission had been made and that a revised version should be published when the standard had been finalized by the Committee This revised version should either bear only the same number as that adopted by the Committee or else print this number alongside the IDF number, so that confusion would be avoided.

17. The Committee was particularly appreciative of the substantial contributions made to the Code of Principles program by IDF and ISO and of the steps taken by both organizations to integrate their common work and so greatly facilitate the task of the Committee.

18. The Committee again noted with regret that the response made to its request for early replies to the questions set out in its Reports had not in a number of cases met with the success it had hoped for. The Committee reiterated that it was essential for governments to make early replies to all requests made in its Reports if rapid progress was to be made. On the other hand, several governments were extremely prompt in making their replies available and the Committee hoped that these would serve as examples to all. The Committee again pointed out that valuable time could be saved if governments sent copies of their comments to each delegation attending the previous Session at the same time as they dispatched them to FAO.

19. The Committee requested the Director-General to give earnest consideration to increasing the speed of processing and dispatching documents. If all documents could be got out rapidly and placed in the hands of governments shortly afterwards, this would greatly facilitate the task of governments in making the early replies to the requests made by the Committee mentioned in paragraph 18 above.

20. In the same way followed for the Report of its Fourth Session the Committee decided to present this Report in two parts:

Part I contains:

- a) Summary of discussions and list of participants.
- b) The texts of standards adopted by the Committee and submitted to Governments for acceptance.
- c) The texts of standards still under discussion and submitted to governments for further comments.

Part II contains%

- a) Text of the Code and Explanatory Note,
- b) Status of acceptances of the Code.
- c) Text of standards finally adopted by the Committee.
- d) Status of acceptances of these standards.

Part II of the present Report forms the Third Edition of the Code of Principles showing the position as at 1 May 1962,

21. SUMMARY OF ACTION RECOMMENDED

The Committee, therefore, requests the Director-General when submitting the present Report to all member governments to invite each governments

- a) to give earnest and sympathetic consideration to the application of the following standards
 - (i) section on Sampling of Cheese to be inserted in Standard No. A.I. (see paragraph 6 and Appendix A - Sampling of Cheese).
 - (ii) General Standards for Cheese (see paragraph 10 and Appendix. B.).
- b) to comment in detail upon the following draft standards:

IDF-FIL-1	1955	Determination of the fat content of normal liquid milk according to Rose-Gottlieb. (<u>sec Appendix C</u>)
IDF-FIL-4	1958	Determination of the dry matter content of cheese. (see Appendix D)
IDF-FIL-10	1960	Determination of the moisture content of butter. (see Appendix E)
IDF-FIL-12	1960	Determination of the salt content of butter. (see Appendix F)
IDF-FIL-13	1960	Determination of the fat content of evaporated and of sweetened condensed milk according to Rose-Gottlieb, (see Appendix G)
- c) To comment in detail on the question concerning the use of brand names and marks (see paragraph 12).
- d) to provide the Secretariat with any details of difficulties in the application of the Code in practice throughout the world, and if specifically so requested by individual letter, with supplementary information needed to complete details of the state of application of the Code (see paragraphs 3 and 4)".

APPENDIX A

STANDARD ADOPTED BY THE COMMITTEE AND SUBMITTED TO GOVERNMENTS FOR ACCEPTANCE

STANDARD No. B.1 (1962) -(Part)

SAMPLING OF CHEESE

1. Objective of sampling

The aim of sampling should be to select from a portion of the consignment a mean sample representative of the different cheese comprising the whole consignment, and to obtain from each cheese to be examined a sample representative of the entire cheese.

2. Weight of sample

The weight of the sample shall be not less than 50 grammes.

3. Sampling equipment

3.1 Hard steel or stainless steel cheese triers of a shape and size suited to the cheese to be sampled.

3.2 Stainless steel knife with pointed blade.

3.3 Tinfoil or aluminium foil.

3.4 Sample containers permitting air tight closure.

4. Sampling technique

One of the following three techniques shall be employed, depending upon the shape, weight, type and maturity of the cheese:

a) sampling by cutting a sector;

b) sampling by means of a trier;

c) taking a complete cheese as a sample.

When a choice must be made between a) and b), method a) is preferable but method b) is acceptable, especially with hard cheese of large size.

4.1 Sampling by cutting out a sector

Using a knife with a pointed blade, make two cuts radiating from the center of the cheese. The size of the sector thus obtained shall be such that after removal of inedible surface layer, the remaining edible portion shall weigh not less than 50 grammes.

4.2 Sampling by means of a Trier

According to the shape, weight and type of cheese, the following techniques are employed:

4.2.1 The trier may be inserted obliquely towards the centre of the cheese once or several times into one of the flat surfaces at a point not less than 10 to 20 cm. from the edge. From the plug or plugs thus obtained cut off not less than 2 cm. of the extremity containing the rind and use this piece to close the hole made in

the cheese. The remainder Of the plug or plugs shall constitute the sample.

The plug holes shall be closed with great care, especially with large cheese, and if possible they should he sealed over with an approved compound,

- 4.2.2 The trier may he inserted perpendicularly into one face and passed through the centre of the cheese to reach the opposite face.
- 4.2.3 The trier may he inserted horizontally into the vertical face of the cheese, midway "between the two plane faces, towards the centre of the cheese.
- 4.2.4 In the case of cheese transported in barrels, boxes, or other hulk containers, or cheese which is formed into large compact "blocks, sampling may he performed by passing the trier obliquely through the contents of the container from the top to the base.

4.3 Sampling by taking an entire cheese

This method shall normally be reserved for small cheese and for wrapped portions of cheese packaged in small containers. A sufficient number of packages shall be taken to have a minimum weight of 50 g.

In the sole case of soft cheese, sold by the piece,, for which a minimum weight of dry matter is legally specified, the cheese must be weighed at the moment of sampling and the weight stated on the label.

4.4 Sampling of cheese in brine

Cheese in brine shall be sampled by taking fragments of at least 100 g. each and sufficient brine to cover the choose in the sampling container.

5. Treatment and storage of samples

5.1 Immediately after sampling the samples (plugs, sectors, entire small cheese) shall be placed in a sample container of suitable size and shape.

The sample may be cut into pieces for insertion into the container but it shall not be compressed or ground up.

5.2 No preservative substance shall be introduced into the receptacle containing the sample.

5.3 The receptacles containing the sample shall be sent immediately to the laboratory, where the analysis shall be commenced as soon as possible. If transit or analysis are delayed, samples must be maintained under, such conditions to avoid fat or moisture separation. Soft cheeses must always be maintained at temperature between 0c-and 80 c.

5.4 Whatever the method of sampling employed, use of a trier or cutting a sector, care should be taken to remove only the inedible surface layer of the cheese, such as mould and horny portions. Only in the case of soft cheese sold by the piece, for which a minimum weight of dry matter is legally specified, the outer rind or crust shall not be removed when determining the weight of dry matter in the piece of cheese.

APPENDIX B

STANDARD ADOPTED BY THE COMMITTEE AND SUBMITTED TO GOVERNMENTS FOR ACCEPTANCE

STANDARD No. A, 6 (1962)

GENERAL STANDARD FOR CHEESE

1. Cheese

1.1 Definition

"Cheese" is the fresh or matured product obtained by draining after coagulation of milk, cream, skimmed or partly skimmed milk, buttermilk or a combination of some or all of these products.

1.2 Permitted additions

The following substances may be added, provided that such substances are not intended to take the place of any milk constituents

- a) harmless substances which are necessary for the manufacturing process;
- b) natural flavouring substances not derived from milk such as spices, in such quantity that they can be considered only as flavouring substances, provided that the cheese remains the major constituent and that the addition is declared in the designation of the product (e.g. cheese with celery, etc.), unless the presence of spices is a traditional characteristic of the cheese.

1.3 Designations

The terms used to designate the variety of cheese shall only be applied to these products which conform to the definition of cheese given in paragraph 1.1 and which possess the characteristics normally associated with that variety.

1.4 Marking and Labelling

1.4.1 The original cheese, or where not possible, the original pack or prepared consumer pack shall be marked with:-

- (a) the name or other clear indication of the producing country in the case of cheese designated by the name of a variety not originating in the producing country;
- (b) the name of the variety;
- (c) unless paragraph 1.4.2. applies, the minimum fat content in the dry matter whenever below 45%.

1.4.2 It shall not be obligatory to mark the minimum fat content whenever the variety complies with an international Standard of composition fixing minimum fat and maximum moisture content, adopted under the Code of Principles; or until such an international standard has been adopted, whenever:

as respects exclusively the homo market, the variety complies with national legislation defining its composition.

- 1.4.3 The designation "full fat", or equivalent expression, may be used for a cheese having minimum 45% fat in the dry matter, provided such a designation is already traditionally used in respect of that type of cheese.
- 1.4.4 The fat content of cheese shall be expressed as a percentage of the dry matter. Marking of fat content, producing country and cheese designation shall be made in distinct and legible words and figures.
- 1.4.5 All exported cheese, or its pack as well as commercial documents referring thereto shall in every case bear the name of the producing country, and an indication of the manufacturer or exporter in plain or in code.

2. Whey Cheese

2.1 Definition

Whey cheese is the product obtained by concentration or coagulation of whey, with or without addition of milk and milk fat.

2.2 Export Standards:

- 2.2.1 The standard for composition of "whey cheese" is the percentage fat content in the dry matter.
- 2.2.2 The minimum percentage fat in the dry matter in full cream whey cheese shall be 33%.
- 2.2.3 The minimum percentage fat in the dry matter in full fat whey cheese shall be 10%.

2.3 Marking and Labelling:

- 2.3.1 Whey, cheese or its pack shall bear the designation "whey cheese", the name of the producing country and the minimum fat content in the dry matter.
- 2.3.2 The marking of fat percentage in the dry matter and the designation "whey cheese" shall be made in distinct and legible figures and words.

APPENDIX C

STANDARD GIVEN PRELIMINARY CONSIDERATION SUBJECT TO DETAILED COMMENTS TO BE RECEIVED FROM GOVERNMENTS.

STANDARD METHOD FOR THE DETERMINATION OF THE FAT CONTENT OF NORMAL LIQUID MILK ACCORDING TO ROSE-GOTTLIEB

I. Definition of the Fat Content

The fat content means the total content of fat and fatty substances, expressed in percent by weight, that is obtained when determining the fat content of normal liquid milk by the Rose-Gottlieb method,

II. Analysis

1) Apparatus and utensils

- 1.1 Analytical balance, sensitivity 0.1 mg.
- 1.2 A centrifuge of appropriate type in which to place the extraction tubes or flasks, and in which a speed of 500-600 r.p.m. can be maintained,
- 1.3 Drying oven maintaining constant temperature or vacuum drying oven,
- 1.4 Erlenmeyer or flat-bottomed flasks holding 150-250 ml, with provision for permanent or semi-permanent marking of the reference number of the sample,
- 1.5 Substance to facilitate boiling, free of fat, which does not disintegrate.
- 1.6 Suitable extraction tubes or flasks, with airtight cork or neoprene stoppers.

2) Reagents

- 2.1 Ammonia solution 25-30% clear, colourless.
- 2.2 Ethyl alcohol, 96 volume % (± 1 vol. %),
- 2.3 Ethyl ether, boiling point 34° - 35°C, free of peroxide.
- 2.4 Petroleum ether, boiling point 30° - 60° C.

Instead of pure alcohol, ethyl alcohol denatured with either methyl alcohol or benzene may also be used.

The reagent's used shall leave no residue after evaporation.

In order to control the reagents, a blank test corresponding exactly to the mode of operation, is to be carried out using 10 ml. of distilled water in place of milk. A small quantity of pure fat is introduced into the receiving flask before it is dried and weighed. The blank estimation must be taken into account in the final calculation of the analysis, but the value should not exceed an almost negligible quantity.

3) Preparation of the sample:

Prior to analysis, bring the sample to $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$, and mix carefully. When the fat is not evenly dispersed, heat the sample slowly to about 40°C ., mix gently and cool to $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$. before the analysis.

4) Mode of operation

- 4.1 Weigh about 10 g. of milk to an accuracy of 1 mg. into the extraction apparatus.
- 4.2 Add 2 ml, of ammonia solution and mix carefully.
- 4.3 Add 10 ml, of ethyl alcohol and mix the contents.
- 4.4 Add 25 ml, of ethyl ether, close the extraction apparatus, then mix the contents by shaking vigorously and turning upside down repeatedly for one minute.
- 4.5 Add 25 ml. of petroleum ether, close the extraction apparatus, mix the contents and shako for approximately one minute carefully in order to avoid the formation of emulsions. (when a centrifuge is available this precaution is not necessary).
- 4.6 Let the extraction apparatus stand or centrifuge (for not less than one minute at 500-600 r.p.m.) until such time as the ether-petroleum other layer is practically clear and has entirely separated from the aqueous layer.
- 4.7 Transfer the ether - petroleum other layer as completely as possible by decanting or by moans of a pressure siphon (taking care, however, that no part of the aqueous layer is carried along) into an Erlenmeyer or flat-bottomed flask containing a substance which facilitates boiling. Then rinse the stopper of the extraction apparatus and the pressure siphon with a few milliliters of ethyl other.
- 4.8 Repeat the extraction a second and third time, using each time 15 - 25 ml, of ethyl other and petroleum other, following the procedure as indicated in 4.5 and 4.6, and transfer on each occasion into the same flask the other - petroleum ether layer as indicated in 4.7 and 4.8. In the case of the Mojonnier flask add alcohol for the second extraction and water for the third extraction to bring the aqueous solution to the correct level.
- 4.9 Carefully evaporate the solvents from the flask.
- 4.10 Dry the fat either in a vacuum drying oven for one hour at $70 - 75^{\circ}\text{C}$. (pressure less than 50 mm. Hg) or in a drying oven under normal pressure at $100-105^{\circ}\text{C}$. The drying process can be accelerated if the vapours remaining in the flask after evaporation of the solvents are blown off with a gentle current of air and if the flask is dried in a horizontal position.
- 4.11 Lot the flask cool and weigh it as soon as it has reached the room temperature, using as counter weight a reference flask treated in an identical manner, or in the case of a single pan analytical

balance, correct weight by the change in weight of the reference flask.

- 4.12 Continue the drying process with hourly weighings to constant weight or to a slight increase of weight. In the latter case, take the last value found before an increased weight as a "basis for the calculation.

Eliminate the fat through three successive washings with petroleum ether using each time 5 ml. The purpose of this operation is to eliminate any error due to minor entrainment of non-fatty matters during extraction. Weigh the flask after elimination of the solvents according to 4.9 and 4.10. The weight of fat is the difference between the two weighings.

5) Accuracy of the method

The results of duplicate determination should not differ by more than 0.04 g. fat for 100 g. of the product.

APPENDIX D

STANDARD GIVEN PRELIMINARY CONSIDERATION SUBJECT TO DETAILED COMMENTS TO BE RECEIVED FROM GOVERNMENTS

STANDARD METHOD FOR THE DETERMINATION OF DRY MATTER CONTENT OF CHEESE

I. Definition of Dry Matter

The dry matter of cheese is the mass, expressed in per cent by weight, which remains after the drying process described hereafter.

II. Analysis

1. Apparatus; utensils and auxiliary agents

- 1.1 Analytical balance, sensitivity 0.1 mg.
- 1.2 Desiccator provided with efficient drying agent (silica gel with hygrometric indicator or calcium chloride).
- 1.3 Drying oven capable of maintaining a constant temperature up to 110° C.
- 1.4 Nickel or aluminium dishes, height about 2 cm, diameter 6 to 8 cm.
- 1.5 Coarse grained quartz sand or sea sand, purified with hydrochloric acid, washed ignited.
- 1.6 Flat ended glass stirrers.

2. Preparation of the sample

(Full description requested from Governments).

3. Code of Operation

- 3.1 Place about 20 g. of sand and a glass stirrer in a nickel or aluminium dish.
- 3.2 Dry the dish containing the 'sand and the stirrer' in the drying oven at 105° C. to constant weight.
- 3.3 Allow the dish to cool in the desiccator, and weigh.
- 3.4 Quickly place about 3 g. of the prepared cheese sample in the dish, and weigh again.
- 3.5 Gently mix the cheese with the sand by means of the stirrer*.
- 3.6 Dry the dish in the oven at 105° C, for 4 hours,
- 3.7 Allow to cool in the desiccator and weigh.
- 3.8 Repeat the drying process with weighings at half-hour intervals to constant weight.

* With cheeses which melt to a horn-like mass at a temperature of 105°C. it is recommended that the dish containing the crushed cheese mass shall first be held in a desiccator for 16 hours at laboratory temperature and under normal air pressure, The contents of the dish shall be thoroughly mixed with the stirrer from time to time to prevent the formation of a rind.

4. Accuracy of the method

The results of duplicate determination should not differ by more than 0,2 g. dry matter in 100 g. of the product.

APPENDIX E

STANDARD GIVEN PRELIMINARY CONSIDERATION SUBJECT TO DETAILED COMMENTS TO BE RECEIVED FROM GOVERNMENTS

STANDARD METHOD FOR THE DETERMINATION OF THE MOISTURE CONTENT OF BUTTER

I. Definition of the Moisture Content

The moisture content means the percentage loss of weight on drying the butter at 102° C. ($\pm 2^\circ$ C.).

II. Analysis

1. Apparatus and utensils

- 1.1 Analytical balance, sensitivity 1 mg.
- 1.2 Drying oven maintaining constant temperature between 100°-104° C
- 1.3 Desiccator charged with silica gel.
- 1.4 Porcelain or non-corrosive metal dishes with a height of about 2 cm. and a diameter of 6 to 8 cm.
- 1.5 Pure, granular, sieved pumice with a diameter of 2.8 to 3.4 mm.

2. Preparation of the sample

The sample is to be maintained at 18 to 20° C. Mixing is not necessary unless the sample gives evidence for it. If so, the mixing shall be done at a temperature not exceeding 25° C. and according to an acknowledged method.

3. Mode of operation

- 3.1 12 to 15 g. pumice are placed in the dish.
- 3.2 Dry the dish with the pumice at a temperature of $102 \pm 2^\circ$ C. to constant weight.
- 3.3 Allow the dish to cool in the desiccator and weigh.
- 3.4 Accurately weigh into the dish about 5 g. (or 10 g.) of the butter sample.
- 3.5 Dry the dish in the oven at $102 \pm 2^\circ$ C. for 2 hours
- 3.6 Cool in the desiccator and weigh.
- 3.7 Repeat the drying process, drying each time for half an hour, to constant weight (within 3 to 6 mg.). In case of an increase in weight, the lower weight is used for the calculation.

$$100 \times \frac{\text{Difference in weight of the butter before and after drying}}{\text{Weight of the butter before drying}}$$

4. Accuracy of the method

The results of duplicate determination should not differ by more than 0.1 g. water for 100 g. of the product.

APPENDIX F

STANDARD GIVEN PRELIMINARY CONSIDERATION SUBJECT TO DETAILED COMMENTS TO BE RECEIVED FROM GOVERNMENTS

STANDARD METHOD FOR THE DETERMINATION OF THE SALT CONTENT OF BUTTER

I. Definition of Salt Content

The salt content means the content of sodium chloride expressed in percent by weight.

II. Analysis

1. Principle

The butter is melted in hot water and the chlorides in the mixture are titrated with a solution of silver nitrate, using potassium chromate as indicator (Mohr's method).

2. Apparatus

2.1 Analytical balance, sensitivity 1 mg.

2.2 Erlenmeyer flask, holding 200 ml.

2.3 Burette calibrated to 0.1 ml.

3. Reagents

3.1 Solution of silver nitrate of 0.1 N.

3.2 Solution of potassium chromate, 5% 3.3 Calcium carbonate free from chloride.

4. Preparation of the sample

Mix the sample thoroughly according to an acknowledged method at a temperature not exceeding 25° C.

5. Procedure

5.1 Weigh into the Erlenmeyer flask, to the nearest 10 mg., about 5 g. of the sample.

5.2 Carefully add 100 ml. of boiling distilled water. Allow standing with occasional swirling for 5-10 minutes.

5.3 After cooling to 50-55° 0. (titrating temperature), add 2 ml. of the potassium chromate solution. Mix by swirling.

5.4 If the butter is of the acid type (pH < 6.5), add a pinch of calcium carbonate. Mix by swirling.

5.5 Titrate at 50-55° C. with the 0.1 l silver nitrate solution while swirling continuously, until the brownish colour persists for half a minute.

5.6 Carry out a blank test,

- 5.7 Calculate the salt (sodium chloride) content by means of the formulas

$$\text{Salt \%} = \frac{5,85 N (V_1 - V_0)}{P}$$

where :

N = normality of silver nitrate solution.

V_1 = number of ml. of the silver nitrate solution used in the titration of P grams of the sample.

V_0 = number of ml. (to 0.05 ml.) of the silver nitrate solution of 0.1 N used in the blank test.

P = weight (in grams) of the butter taken for analysis.

6. Accuracy of the method

The results of duplicate determination should not differ by more than 0.02 g. sodium chloride for 100 g. of the product.

APPENDIX G

STANDARD GIVEN PRELIMINARY CONSIDERATION SUBJECT TO DETAILED COMMENTS TO BE RECEIVED FROM GOVERNMENTS

STANDARD METHOD FOR THE DETERMINATION OF THE FAT CONTENT OF EVAPORATED AND OF SWEETENED CONDENSED MILK ACCORDING TO ROSE- GOTTLIEB

I. Definition of the Fat Content

The fat content means the total content of fat and fatty substances expressed in per cent by weight, obtained by the Röse-Gottlieb method.

II. Analysis

1. Apparatus and utensils

- 1.1 Analytical balance, sensitivity 0.1 mg.
- 1.2 A centrifuge of appropriate type in which to place the extraction tubes or flasks, and in which a speed of 500-600 r.p.m. can be maintained,
- 1.3 Drying oven maintaining constant temperature or vacuum drying oven.
- 1.4 Erlenmeyer or flat-bottomed flasks holding 150-250 ml, with provision for permanent or semi-permanent marking of the reference number of the sample.
- 1.5 Substance to facilitate boiling, free of fat, which does not disintegrate.
- 1.6 Suitable extraction tubes or flasks, with airtight cork or neoprene stoppers.

2. Reagents

- 2.1 Ammonia solution 25-30% clear, colourless.
- 2.2 Ethyl alcohol, 96 volume % (± 1 vol. %).
- 2.3 Ethyl ether, boiling point 34° - 35° c., free of peroxide.
- 2.4 Petroleum ether, boiling point 30° - 60 C. Instead of pure alcohol, ethyl alcohol denatured with either methyl alcohol or benzene may also be used.

The reagents used shall leave no residue after evaporation.

In order to control the reagents, a blank test corresponding exactly to the mode of operation, is to be carried out using 10 ml, of distilled water in place of milk. A small quantity of pure fat is introduced into the receiving flask before it is dried and weighed. The blank estimation must be taken into account in the final calculation of the analysis, but the value should not exceed an almost negligible quantity.

3. Preparation of the sample

Mixing of the sample is necessary if a more or less extensive partial separation of some components, e.g. proteins, fat, calcium salts or lactose, has occurred.

For Evaporated milks

Open the container at the edge of the lid, pour the milk slowly into another container and mix the components by repeated transfers. Any milk or fat adhering to the lid is to be reincorporated into the sample. Heat the covered sample to a temperature of 40° C. and thoroughly mix by stirring. Allow to cool.

For Sweetened condensed milks

Open the container at the edge of the lid. Any milk or fat adhering to the lid is to be reincorporated into the sample. Heat to a temperature of 40°C. and thoroughly mix by stirring from top to bottom with a spoon. Allow to cool.

4. Mode of operation

- 4.1 Weigh about 5 g. of the evaporated milk (or 2 to 3 g. of the sweetened condensed milk), to an accuracy of 1 mg., into the extraction apparatus.
- 4.2 Add water up to a volume of about 10.5 ml. while gently shaking and heating in a water bath (40 to 45° C.) until the milk is completely dispersed.
- 4.3 Add 1 ml. of ammonia solution and mix carefully.
- 4.4 Add 10 ml. of ethyl alcohol and mix the contents.
- 4.5 Add 25 ml. of ethyl ether, close the extraction apparatus, then mix the contents by shaking vigorously and turning upside down repeatedly for one minute.
- 4.6 Add 25 ml. of petroleum ether, close the extraction apparatus, mix the contents and shake for approximately one minute carefully in order to avoid the formation of emulsions. (When a centrifuge is available this precaution is not necessary).
- 4.7 Let the extraction apparatus stand or centrifuge (for not less than one minute at 500 - 600 r.p.m.) until such time as the ether-petroleum ether layer is practically clear and has entirely separated from the aqueous layer.
- 4.8 Transfer the ether-petroleum ether layer as completely as possible by decanting or by means of a pressure siphon (taking care, however, that no part of the aqueous layer is carried along) into an Erlenmeyer or flat-bottomed flask containing a substance which facilitates boiling. Then rinse the stopper of the extraction apparatus and the pressure siphon with a few milliliters of ethyl ether.

- 4.9 Repeat the extraction a second and third time, using each time 15 - 25 ml. of ethyl ether and petroleum ether, following the procedure as indicated in 4.6 and 4.7. and transfer on each occasion into the same flask the ether-petroleum ether layer as indicated in 4.8 and 4.9. In the case of the Mojonnier flask add alcohol for the second extraction and water for the third extraction to bring the aqueous solution to the correct level.
- 4.10 Carefully evaporate the solvents from the flask.
- 4.11 Dry the fat either in a vacuum drying oven for one hour at 70° - 75° C. (pressure less than 50 mm. Hg) or in a drying oven under normal pressure at 100° - 105° C. The drying process can be accelerated if the vapours remaining in the flask after evaporation of the solvents are blown off with a gentle current of air and if the flask is dried in a horizontal position.
- 4.12 Let the flask cool and weigh it as soon as it has reached the room temperature, using as counter weight a reference flask treated in an identical manner, or in the case of a single pan analytical balance, correct weight by the change in weight of the reference flask.
- 4.13 Continue the drying process with hourly weighings to constant weight or to a slight increase of weight. In the latter case, take the last value found before an increased weight as a basis for the calculation.

Eliminate the fat through three successive washings with petroleum ether using each time 5 ml. The purpose of this operation is to eliminate any error due to minor entrainment of non fatty matters during extraction. Weigh the flask after elimination of the solvents according to 4,10 and 4.11. The weight of fat is the difference between the two weighings.

5. Accuracy of the method

The result's of duplicate determination should not differ by more than 0.05 g. fat in the product.

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

Report of the Meeting of Government Experts on the Use of Designations, Definitions and Standards for Milk and Milk Products, Rome, Italy, 8-12 September 1958. In English, French and Spanish (Meeting Report : No. 1958 15).

Report of the Second Meeting of Government Experts on the Use of Designations, Definitions and Standards for Milk and Milk Products, Rome, Italy, 13-17 April 1959. In English, French and Spanish (Meeting Report No. 1959/AN-2).

Report of the Third Meeting of Government Experts on the Use of Designations, Definitions and Standards for Milk and Milk Products, Rome, Italy, 22-26 February 1 960. In English, French and Spanish (Meeting Report No. AN 1960 2).

Code of Principles concerning Milk and Milk Products and Associated Standards, First Edition, 1 March 1960.

Report of the Fourth Session of the Committee of Government Experts on the Code of Principles concerning Milk and Milk Products, Rome, Italy, 6-10 March 1961. In English, French and Spanish (Meeting Report No. AN 1961/3).

Code of Principles concerning Milk and Milk Products and Associated Standards 2nd Edition, 1 April 1961.