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ALINORM 72/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME  
CODEX ALIMENTARIUS COMMISSION  
Ninth Session, Rome, 6-17 November 1972  
REPORT OF THE SEVENTH SESSION OF THE  
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING  
Budapest, 12-18 September 1972

PART I

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Seventh session from 12 to 18 September 1972 in Budapest at the courtesy of the Government of Hungary. The session was presided over by Mr. A. Miklovicz, chairman of the Hungarian Codex Committee. In welcoming the participants at the session, Dr. K. Karcsai, General Secretary of the Hungarian National FAO Committee stressed the importance of reaching agreement on appropriate methods of analysis and sampling as part of international standardization of food. The session was attended by delegates from 25 countries and 11 international organizations. The list of participants, including officers from FAO, is contained in Appendix I to this report.

ADOPTION OF THE AGENDA

2. The Committee adopted the provisional agenda without rearrangement of the order of the items but agreed to discuss the problem of sampling under item 4 of the Agenda.

APPOINTMENT OF RAPORTEURS

3. Dr. W. Horwitz of the delegation of the USA and Ir. J. Gosselé of the delegation of Belgium agreed to act as rapporteurs.

PART II

MATTERS ARISING FROM THE REPORT OF THE 8th SESSION OF THE CODEX ALIMENTARIUS COMMISSION AND THE 18th SESSION OF THE EXECUTIVE COMMITTEE

4. The Committee noted the decisions of the Commission concerning the determination of contaminants in margarine and the determination of tocopherols in olive oils contained in paras 180-182 of the Commission's Report ALINORM 71/31. The Delegate of Australia drew the Committee's attention to the conclusions of the 18th Session of the Executive Committee concerning the meaning of acceptance of Codex referee methods of analysis, i.e., that in accepting such methods a country undertook to use them in cases of dispute involving food moving in international trade (see ALINORM 72/3, paras 27-28). In the opinion of the Delegate of Australia the application of Codex referee methods to such disputes, while applying a different method for the settlement of disputes at a national level, could lead to discrimination between commodities which had been imported and those produced in the country concerned, and this matter should be considered by the Commission (see also para 86).

PART III

SAMPLING

Sampling in General

5. The Committee also noted the conclusions of the Commission concerning sampling and, more specifically, the sampling plans which had already been adopted (see ALINORM 71/31 paras 87-90). During the discussion on the problem of sampling a number of basic issues

were raised. The Committee agreed that the most expeditious way of dealing with these problems was to set up an ad hoc working group to meet during the session. The following countries were designated as members of the working group: Canada (Dr. D. Smith), Denmark (Mr. Bergström-Nielsen), Federal Republic of Germany (Dr. P. Vogel), United Kingdom (Mr. D.L. Orme), U.S.A. (Mr. R.P. Farrow), the Netherlands (Dr. A. Kruysse) and Hungary (Mr. T. Zoltán). Dr. L.G. Ladomery of the FAO Secretariat assisted the meeting of the working group. The Committee was in general agreement with the ideas raised by the ad hoc working group, the Report of which is given in Appendix II to this Report. The conclusions of the Committee based on the recommendations of the ad hoc working group on sampling are given in the following paragraphs.

6. The Committee discussed the proposal of the ad hoc working group that sampling requirements for Codex Standards should be developed as guidelines rather than as a mandatory part of standards to cover dispute situations. It was recognized that the General Principles for the Establishment of Methods of Analysis (Codex Procedural Manual, 2nd Ed. p. 59), if applied to the question of sampling, would mean that Codex methods of sampling were basically intended for the settlement of international disputes involving trade in food. Such an extension may not be justified, particularly since not all of the criteria for methods of analysis are applicable. Thus the Committee agreed that it was necessary to extend the above principles to include criteria on the basis of which to select appropriate sampling provisions, and requested the delegations of Canada, Hungary, the Netherlands, the United Kingdom and U.S.A. to draft such principles for the next session of the Committee. The United Kingdom undertook to prepare the first draft the Hungarian Secretariat will send to delegations for comment.

7. The Committee, however, noted that by developing sampling provisions only for the rare international dispute situation, it would unduly narrow its scope of work, since the purpose of the Codex Alimentarius was to facilitate trade and protect the interest of the consumers. It was, therefore, desirable to take steps to prevent disputes from occurring and to provide a common approach to the enforcement of Codex standards in respect of sampling. The Committee agreed to develop guidelines on the approach to sampling as possibly a first step towards the elaboration within the Codex of mandatory sampling provisions in Codex standards. This initial step was considered necessary in view of the difficulty in drawing up sampling provisions to cover the many different dispute situations, especially in the face of the differing administrative set-ups and basic differences in legal approach to sampling which exist in various countries. Apart from such differences, the Committee agreed that the interpretation of the results of sampling and analysis (e.g., decision based on the average as against the requirement that all units of production should conform with the standard) represented an area of difficulty, especially in relation to minimum platform standards.

#### Vocabulary of Sampling Terms and Definitions

8. The Committee had before it the ISO document: TC 34/WG 1 (Secret. 13) 26 Bis: Draft Vocabulary of Sampling Terms and Definitions. The Committee was informed that this Vocabulary was partly based on the ISO Recommendations No. R645 and R1786: Statistical Vocabulary and Symbols. The Committee recognized that there was a need, in the Codex work, for uniformity in the terminology of sampling methods and sampling plans. It was of the opinion that the very important work done by ISO in this field should be communicated to all members of the Codex Alimentarius Commission. It considered that ISO/TC 34 and TC 69 should be invited to fully cooperate with Codex in this area. The Committee further considered that the development of standard terminology should be left to liaison between the Secretariats of ISO and FAO, taking into consideration the comments of government members of the Codex Alimentarius Commission.

#### Standard Layout and Guide to the Drafting of a Standard Method of Sampling

9. The Committee had before it the ISO document (TC/34 WG 1 (Secret. 20) 40: Standard Layout and Guide to the Drafting of a Standard Method of Sampling from a Lot, and government comments on it, appearing in the paper CX/MAS 72/10. The Committee did not discuss this document in detail but considered it as a basic reference for Codex purposes. The Committee agreed that those parts of the Standard Layout and Guide which could be adapted to Codex work together with the Codex Technical Procedures for taking Samples (see para 10) should be used to prepare a new paper on sampling. The Committee agreed that the Secretariat of the Codex Committee on Methods of Analysis and Sampling should undertake this work.

#### Technical Procedures for Taking Samples

10. The Committee agreed that this document, which had reached Step 6 and appeared as Appendix VI to ALINORM 69/23, would be incorporated at a further stage into the

Codex Standard Layout and Guide, and other Codex documents on Sampling. The Committee was also informed by the United Kingdom delegation that the British Standards Institute had developed procedures for taking samples and would send them to members of the Committee and the Secretariat. It was understood that the Codex Standard Layout and Guide would cover the field of Technical procedures for taking samples and that there was no urgent need to develop these procedures separately.

#### Sampling of Cocoa Beans

11. The Committee temporarily endorsed the ISO Draft Recommendation DR 2292 but agreed that this method would be reconsidered after the General Principles for the Elaboration of Codex Sampling Methods had been established (see paras 6-7).

### PART IV METHODS OF ANALYSIS

#### GENERAL RESERVATION BY CANADA

12. The delegation of Canada made the following statement concerning the acceptance and application of Codex referee methods of analysis:

"In Canada preference must be given for legal reasons in the enforcement of mandatory food regulations to those methods which have been most thoroughly tested. Thus in a number of cases Canada will have to indicate that it considers such collaboratively tested methods as "suitable alternatives" to a Codex method for which evidence of such collaborative testing is not available and, until the Codex method can be proved equivalent, Canada will have to give preference to the existing tested method".

#### GENERAL METHODS OF ANALYSIS FOR PRESERVATIVES IN FOODS

13. The Committee had before it government comments (paper CX/MAS 72/5) on the methods for determination of preservatives in foods (CX/MAS 70/C/3) which had been prepared by the delegation of the Netherlands for consideration by the Sixth Session of this Committee. The Committee was informed that the delegation of the U.S.A. in its written comments (Appendix I to CX/MAS 72/5) had proposed a revised method for the determination of organic preservatives in food, which combined the Woidich and Nordic Analytical Committee methods. Several delegations were in favour of the U.S.A. proposal, while other delegations indicated that this method was still subject to improvement with regard to the use of specific reagents and to the choice of appropriate polyamide substrate. The Committee agreed that the delegations of the Netherlands and the U.S.A. should amend the present method, taking into account these suggestions, organize a collaborative study, and report to this Committee at its Eighth Session.

#### GENERAL METHODS OF ANALYSIS FOR ANTIOXIDANTS IN FOODS

14. The Committee had before it the document of General Methods for Antioxidants in Foods prepared by the delegation of the Netherlands for the Sixth Session (CX/MAS 70/C/3). The delegation of the Netherlands indicated that no new document had been prepared on this subject. The Committee was also informed that the European Economic Community (EEC) had developed a general method for the determination of antioxidants in fats and oils. This method was based on a separation of fat or oil by specific solvents, separation of antioxidants by acetonitrile and petroleum ether, and identification by thin-layer chromatography. It was pointed out that such a method should be placed before the Committee, so that it can be considered in the light of other methods in use at present. The Committee agreed to a proposal made by the observer from the EEC to make this method available to the Secretariat so that it could be distributed to governments for comments not later than 30 May 1973.

#### METHODS FOR THE DETECTION AND IDENTIFICATION OF COLOURS IN FOOD

15. The Committee had before it the Method of Identification of Food Colours using Thin-Layer Chromatography, prepared by the delegation of the United Kingdom (CX/MAS 70/C/4) which had been already discussed at the Sixth Session of the Committee. The Committee took note of the comments received from the governments as regards the national legal status of food colours (paper CX/MAS 72/6) and agreed that they would serve in updating the U.K. document.

16. The Committee noted that another procedure published in the Journal of the AOAC, had been proposed by the delegation of the U.S.A. It was also informed that two methods, one for water-soluble synthetic colours, the other for fat-soluble colours, had been developed and collaboratively studied by the EEC, which would make them available to the Secretariat in due course. The Committee recognized that, in many cases, Codex

standards provided for a maximum level for colours and that, consequently, a Codex method required a quantitative technique. The Committee was informed that collaborative studies on the recovery of colours were being performed by the AOAC. The U.S.A. would try to incorporate the U.K. method (not yet published) in the studies. The Committee was also informed that the Codex Committee on Food Additives was preparing a list of permitted food colours, as well as lists of other additives and that these lists would be sent to governments in the near future.

17. The Committee agreed that it should concentrate its activities on the detection and determination of permitted food colours, as it would not be possible to identify all existing colours by only one analytical method. The Committee also agreed to the proposal made by the delegations of the U.K. and the U.S.A. to continue their efforts in collaborative tests. According to the proposals made by other delegations (Japan, Austria, the Netherlands) it was agreed that their methods should be sent to the U.K. delegation. The Committee decided not to take any action before the results of such collaborative studies were available.

#### METHODS FOR THE DETERMINATION OF METALLIC CONTAMINANTS

18. The Committee had before it a summary of government comments (CX/MAS 72/7) on General Methods for the Determination of Metallic Contaminants in Food (CX/MAS 70/C/2) prepared by the Canadian delegation and already discussed at the Sixth Session of this Committee. The Committee was informed that methods for determination of lead, cadmium and mercury, including methyl mercury, had also been discussed by the Joint FAO/WHO Expert Committee on Food Additives at its 16th Session, Geneva, April 1972. The Committee was in general agreement that methods based on atomic absorption spectrophotometry (AAS) should be referred to the Codex Commodity Committees for trial on their commodities. The Committee, however, noted that in the case of very low levels of metallic contaminants, of the order of less than 1 ppm, AAS methods in many cases might not be applicable, without special separations and background corrections.

19. The Committee noted that several collaborative studies were under way as regards the AAS method and agreed to a proposal made by the Canadian delegation to update their paper for the eighth session of this Committee, when the results of such collaborative studies were known. The Committee also recognized that these methods were to be used in relation to the provisions laid down in Codex standards and that, in many cases, the detection of levels of contaminants lower than 1 ppm would not be needed (see also paras 39, 63 and 76 (ii)).

#### METHODS OF ANALYSIS FOR SOLVENT RESIDUES IN FOOD

20. The Committee had before it various proposals made by governments for the determination of hydrocarbons, chlorinated hydrocarbons and other solvents in food (CX/MAS 72/3) as well as extracts of the FAO Nutrition Meetings Report (Series No. 48B: Specifications for the Identity and Purity of some extraction solvents and certain other substances (CX/MAS 72/3 Addendum 1)), which contain methods for the determination of solvent residues in food.

21. The Committee noted that two different classes of solvents should be considered separately:

1. Extraction solvents, which should not be present in food at levels more than trace amounts,
2. Carrier solvents, which are intended to remain in the food to which they have been added. As far as the carrier solvents are concerned, the Committee was informed that lists of those which have been cleared toxicologically, as well as those which were still subject to evaluation, would be published prior to the eighth session of the Committee.

22. The Committee agreed that, before discussing the analytical methods themselves, general information was required on the levels at which solvents were present in food. Therefore it agreed to request those Commodity Committees which dealt with foods for which extraction solvents were used, e.g. the Codex Committee on Fats and Oils and the Codex Committee on Cocoa Products and Chocolate, to supply this necessary information. The Committee also re-expressed its opinion, already given at its fifth session (ALINORM 70/23, para 10) that the question of levels of residues of extraction solvents should, if possible, also be considered by the Codex Committee on Food Additives and by the Joint FAO/WHO Expert Committee on Food Additives.

## METHODS OF ANALYSIS IN STANDARDS FOR FOODS FOR INFANTS AND CHILDREN

23. The Committee had before it proposals made by the Codex Committee on Foods for Special Dietary Uses for methods of analysis applicable to the Codex Standard for Infant Formula, as well as to other standards for foods for infants and children, together with government comments on these proposals (CX/MAS 72/11) and the U.S. Working Papers (1970) distributed to the heads of the delegations after the sixth session of this Committee (ALINORM 71/23 paras 65)

24. The U.K. delegation pointed out that, in many cases, the specific method of analysis was an integral part of the limits specified in the standards. The Committee also noted the general remarks made by the delegation of the U.K. that the methods to be used for proximate analysis of food products were intended to establish the nutritional value of the products. National food composition tables had been established on the basis of specific methods and conversion factors, both of which may vary from country to country. Therefore, if one single method was internationally agreed for referee purposes, only those food composition tables which were based on these methods would still remain valid. Since the Codex Committee on Foods for Special Dietary Uses had selected food composition tables based on certain methods, these methods would have to be preferred. The Committee was also informed that the FAO Food Composition Tables for International Use, as well as other Food Composition Tables prepared under the auspices of FAO on a regional basis, were generally based on conversion factors of the Atwater system as they appeared in the Handbook No. 8 of the U.S. Department of Agriculture: Composition of Foods. The methods used to develop the tables contained in this Handbook were the AOAC methods.

### Moisture

25. The Committee agreed with a general remark made by the delegation of Poland that, when moisture is determined by desiccation, the determination would be better referred to as "loss on drying". The delegation of the Netherlands was of the opinion that no single method could cover all food products. The delegation of the Federal Republic of Germany was in favour of a desiccation at lower temperature in order to avoid the Maillard reaction in products containing sugars and proteins. The delegation of Czechoslovakia and other delegations were in favour of using the method elaborated by the International Dairy Federation (IDF) for dairy products.

26. The Committee, after discussion, decided to temporarily endorse the AOAC method XI, 7.003 (drying in vacuum-oven at 95-100°C). It further agreed that this endorsement would be reconsidered in the light of the experience gained by countries in the use of the method. A reservation was made by the delegation of Czechoslovakia, the Netherlands and the Federal Republic of Germany. The reservation of the Federal Republic of Germany concerns the Infant Formula based on milk.

### Ash

27. There was a discussion on whether the temperature to be used should be 550° or 600°. The Committee agreed to endorse the method AOAC XI, 7.010 (2 hours at 600°C) which was based on collaborative studies and, on the proposal made by the U.S. delegation, further agreed to reconsider the method in the light of any further studies or collaborative data submitted to the Codex Committee on Food for Special Dietary Uses 6 months prior to its 1973 session.

### Crude Fat

28. Two AOAC methods had been proposed for endorsement, one applicable to cereal-containing foods, (XI, 7.050 - Acid hydrolysis and ether extraction), the other applicable to other products (XI, 7.047-7.052 - Direct ether extraction). Several delegations doubted whether the AOAC methods would be applicable in all cases and were in favour of a single method using acid hydrolysis before extraction and proposed the use of the Weibull-Stoll method for all products. The Committee recognized that there was a need for collaborative testing of the Weibull-Stoll method against the AOAC method in different kinds of products. As regards the standard for Infant Formula, the Committee recognized that some clarification was needed of the meaning of the term "fat", whether it applied to triglycerides only, or included other extractable substances such as phosphatides. The Committee agreed to reconsider this point when this information was available from the Codex Committee on Foods for Special Dietary Uses.

### Crude Fibre

29. The Committee was informed that the proposed AOAC (XI, 7.053-7.057 - Acid and alkali treatment) and ISO/TC 34/WG 3 (Secret. 8)11, 1970 methods were very similar and agreed that a final decision would be made when a jointly agreed text was available.

The Committee agreed to request ISO and AOAC to develop such a text.

#### Crude Protein

30. The Committee considered the proposed method (AOAC, XI, 2.049-2.051 - Digestion using  $H_2SO_4$ , and  $Hg\ O$  as a catalyst) for determination of nitrogen content. The Committee noted with interest that the use of mercury was considered by several delegations undesirable as such a practice was a possible source of pollution. It requested ISO and AOAC to get together and agreed to await the adoption of a final text jointly by AOAC and ISO before reconsidering this problem (see also para 31).

#### Conversion Factors for Available Calories (Available Kilojoules) and Nitrogen

31. The Committee agreed (in principle) to adopt the specific coefficients already in use in the joint FAO/WHO work, as they appear in the most recent Reports of the FAO/WHO Expert Committees, but considered that there was a need to call the Codex Committee on Foods for Special Dietary Uses' attention to the necessity to specify the method used for proximate analysis, which in turn is requested for calculation of calories. It further agreed that the question of conversion factors from nitrogen to protein had also to be referred to that Committee.

#### Available Carbohydrates

32. The Committee took note that, as regards food in general, available carbohydrates were calculated by subtraction of crude fibre from carbohydrates obtained by difference. The Committee agreed that this question was not within its competence and referred it to the Codex Committee on Foods for Special Dietary Uses as, in some cases, a direct determination of carbohydrates might be preferable.

#### Calcium

33. The Committee endorsed the method AOAC, XI, 14.014 (oxalate permanganate method). The ISDI, however drew the Committee's attention to a difficulty which might arise in the presence of large quantities of phosphorus. Other delegations stated that they had found no interference from this source.

#### Phosphorus

34. The Committee endorsed the method AOAC, XI, 22.044-22.046, (gravimetric quinoline - molybdate method) recognizing, however that the IDF method (norm 42: 1947) could be considered as a possible alternative, at a later stage, should collaborative tests demonstrate its equivalence.

#### Iron

35. The Committee endorsed the method AOAC, XI, 14.011-14.013 (O-phenanthroline - spectrophotometric method).

#### Iodine

36. The Committee endorsed the method AOAC XI, 33.056-33.058 (oxidation with Br to  $KIO_3$ , and  $Na_2S_2O_3$  titration).

#### Fluorine

37. The Committee did not consider any method as there was no provision for fluorine content in the standards for foods for infants and children. (1)

#### Sodium-Potassium

38. The Committee agreed that the methods adopted for low-sodium foods would also be endorsed for other foods for special dietary uses (see paras 60-61).

#### Copper, Manganese, Zinc and Magnesium

39. The Committee endorsed the AAS method AOAC, XI, 2.097-2.102 on the understanding that the general method for the determination of metallic contaminants as updated by the delegation of Canada (see paras 18-19) may include these metals.

#### Vitamin A and Carotenes

40. The Committee endorsed the method AOAC, XI, 39.001-39.017 (Saponification, extraction, chromatographic separation and colourimetric determination for Vitamin A, and spectrophotometric determination for carotene) and took note of a remark made by the delegation of the Netherlands that, in cereals used as infant foods, there was sometimes a co-precipitation of vitamin A with starch after the addition of ethanol.

(1) Note by the Secretariat - The Codex Committee on Foods for Special Dietary Uses should propose a method for determination of chloride content in Infant Formula.

Thiamine

41. The Committee endorsed the method AOAC, XI, 39.024-39.030 (Thiochrome fluorescence method).

Riboflavin

42. The Committee endorsed the method AOAC, XI, 39.039-39.047 (fluorometric method). The delegation of the U.K. stated that it would prefer a microbiological method.

Niacin and Nicotinamide

43. The delegation of the U.K. pointed out that, as regards methods of analysis for vitamins of the B complex, it would prefer microbiological methods as the figures given in the standards were generally based on results obtained by using such methods of analysis. The delegation of the U.S.A. declared that the figures given were generally based on results obtained by the methods proposed by the Codex Committee on Foods for Special Dietary Uses. The Committee endorsed the methods AOAC, XI, 39.044-39.046 (colorimetric method, for most products) and 39.101-39.109 (microbiological method, for milk-based products) for the determination of niacin and nicotinamide.

Vitamin C (reduced and total)

44. The Committee endorsed the method AOAC, XI, 39.051-39.055 (indophenol method) for determination of the reduced vitamin C in those products (e.g. fruit juices and potatoes) for which it had been originally developed and the method AOAC, XI, 39.055-39.062 (microfluorometric method) for determination of total vitamin C in all products, including those containing interfering materials (e.g. reductones). The Committee agreed that the method for reduced vitamin C was not applicable to products containing reducing substances (other than ascorbic acid) such as Infant Formula or heated starchy products.

Vitamin D

45. The Committee was informed by the delegation of the U.K. that a new chemical method for the determination of vitamin D had been developed in their country. The Committee also noted that another chemical method, quicker than the biological one, had been published by the Netherlands in the JAOAC, May, 1972. The Committee endorsed the biological method (AOAC, XI, 39.149-39.162) on the understanding that a chemical method might be substituted in the future when results of collaborative studies on the U.K. and the Netherlands methods were available.

Vitamin E

46. The Committee considered whether the SAC method, already endorsed for two Codex standards on fats and oils, or a thin layer chromatographic method recommended by the U.S. delegation was the most suitable. The AOAC method (U.S. Working Paper No. 28, 1970) was temporarily endorsed but a number of delegations considered that both methods involved considerable losses of tocopherols and that newer techniques based on column chromatography and gas liquid chromatography appeared to overcome this problem and might supersede this method before final endorsement.

Vitamin B<sub>6</sub>

47. The Committee endorsed the method AOAC, XI, 39.142-39.147 (Separation on ion exchange column and microbiological assay).

Folic Acid

48. The method AOAC, XI, 39.093-39.097 (growth stimulation of Streptococcus faecalis) was endorsed with the following amendment: "Ascorbate-phosphate buffer may be used in lieu of the phosphate buffer for extraction".

Pantothenic Acid

49. The Committee endorsed the method AOAC, XI, 39.110-39.118 (growth stimulation of Lactobacillus plantarum - ATCC 8014) as applicable to enriched foods, and the procedure described in the U.S. Working Paper No. 31 applicable to un-enriched foods (U.S. Department of Agriculture Handbook No. 97).

Vitamin B<sub>12</sub>

50. The Committee endorsed the method AOAC, XI, 39.085-39.092 (growth response of Lactobacillus leichmannii - ATCC 7830).

Biotin

51. The Committee endorsed the method described in the U.S. Working Paper No. 33

(growth response of Lactobacillus plantarum - ATCC 8014, Ref: Skeggs, Analytical Microbiology, F. Kavanaugh Ed., Academ. Press, 1963, p.461).

#### Linoleic Acid

52. The Committee did not make any decision on a method stated to be for "linoleic acid" by the Codex Committee on Foods for Special Dietary Uses, which was a method prepared by Canada for cis-methylene interrupted polyunsaturated fatty acids (CX/MAS 72/04) as, in its opinion, the standard for Infant Formula was not sufficiently precise as regards exactly what was the criterion and it requested the Codex Committee on Foods for Special Dietary Uses to specify which compound (or compounds) was intended to be covered by the standard. If only linoleic acid was intended, the Committee was informed that a GLC method for the determination of fatty acids was being developed by the Codex Committee on Fats and Oils which would meet in 1974. The delegation of the U.K. proposed that this question should await the outcome of the work of that Committee.

#### Vitamin K

53. The Committee requested the Codex Committee on Foods for Special Dietary Uses to propose a method.

#### Choline

54. (See para 65 of this Report).

#### Quality of the Protein (in Standard for Infant Formula at Step 8)

55. The Committee was informed by the delegation of the U.S.A. that collaborative studies comparing a number of methods were under way. It also noted that the FAO/WHO Protein Advisory Group (PAG) would probably examine these methods at its next session December 1972). The Committee, therefore, deferred endorsement at this stage.

#### Water Capacity of the Container (in Standard for Infant Formula at Step 8)

56. The Committee endorsed the methods for metal containers and glass containers that were already endorsed for canned fruits and vegetables at its sixth session (ALINORM 71/23 para 44 and ALINORM 71/20, e.g. Appendix II, section 7.6).

#### Identification of the Ingredients (in the three Standards for Foods for Infants and Children)

57. The Committee considered whether there was a need for a referee method, as a wide range of optional ingredients was permitted in these products. Therefore the Committee referred this matter back to the Codex Committee on Foods for Special Dietary Uses for reexamination.

#### Residues of Hormonal and Antibiotic Substances (in the three Standards for Foods for Infants and Children)

58. The delegation of the Netherlands informed the Committee that a collaborative study had been performed on the determination of oestrogenic residues in meat and meat products. The observer of the EEC undertook to make available to the Committee those EEC methods which have been collaboratively studied when they were ready. The delegation of the U.K. was of the opinion that the Joint FAO/WHO Expert Committee on Food Additives should examine that problem and the order of the residues to be determined. The Committee did not make any decision and requested the EEC and other organizations if such is the case, to provide information to the Committee when available.

#### METHODS OF ANALYSIS IN THE STANDARD FOR FOODS WITH LOW-SODIUM CONTENT

59. The Committee reconsidered the methods of analysis which had been referred back to the Codex Committee on Foods for Special Dietary Uses as well as new proposals made by that Committee at its sixth session (ALINORM 71/23, paras 25-28 and ALINORM 72/26 paras 119-120). The Committee had before it CX/FSDU 71/7 detailing proposals for methods as well as CX/MAS 72/12 including comments on these methods.

##### (a) ALL FOODS WITH LOW-SODIUM CONTENT

#### Determination of Sodium Content

60. The Committee had before it CX/FSDU 71/7, CX/FSDU 71/17 and CX/MAS 72/12 containing the proposed methods of determination of sodium in low-sodium foods as well as government comments on this subject. The Committee temporarily endorsed the U.S. flame photometry method as described in CX/FSDU 71/17, using dry ashing at 525-550°, but was of the opinion that a rewording of this method was needed, taking into account the determination of small quantities of sodium in presence of large quantities of potassium and/or calcium. The Committee considered that, in these cases, the flame

photometer should be calibrated with solutions containing potassium and/or calcium at concentrations equivalent to that of the samples. It was understood that this method would also be applicable to salt substitutes and might serve other purposes as being of general application to normal foods.

#### Determination of Potassium Content

61. The Committee did not endorse a method and considered that the method proposed for sodium determination would also be valid for potassium determination. The delegation of the U.S.A. agreed that the reworded text of CX/FSDU 71/17 would include a part for potassium determination.

#### Note concerning the Determination of Sodium and Potassium in Salt Substitutes

62. The Committee agreed to the following note which should appear in the section "Methods of analysis and sampling" of the standard, in connection with the flame photometry method:

"Note: Sodium and potassium in a salt substitute shall be determined by comparison with a standard preparation the composition of which is similar to that of the salt substitute".

#### (b) SALT SUBSTITUTES

##### Determination of Calcium and Magnesium

63. The Committee endorsed the method AOAC, XI, 2.097-2.102 (Atomic Absorption) for both calcium and magnesium.

##### Determination of ammonium

64. The Committee considered that it was not likely that salt substitutes would contain significant amounts of protein which might interfere with the determination of ammonia and endorsed the method AOAC, XI, 2.057 (distillation and titration).

##### Determination of Choline

65. The Committee was informed by the delegation of the U.S.A., that the proposed methods were not satisfactory for referee purposes. It noted that a method using the micro-organism *Neurospora crassa* had been developed in the EEC. The U.S.A. delegation pointed out that high results are obtained on yeast with this organism. The Committee agreed not to take any decision before the EEC method is available. The observer of the EEC agreed to send this method to the Secretariat in due course.

##### Determination of phosphorus

66. The Committee temporarily endorsed the method AOAC, XI, 8.025-8.028 (magnesium ammonium phosphate) and agreed to a proposal made by the delegation of the U.S.A. to make available for the eighth session of the Committee a more modern method which would take into consideration the IDF method.

##### Determination of iodine

67. The Committee endorsed the method AOAC, XI, 33.056-33.058 (see para 36).

##### Determination of silica

68. The Committee endorsed the method AOAC, XI, 35.049 ( $H_3SiO_3$ , precipitation and ignition to  $SiO_2$ ) and agreed that results should be expressed as  $SiO_2$ .

##### Qualitative tests for the identification of the anions

69. The Committee was of the opinion that no referee method was needed for the identification of the anions and referred this matter back to the Codex Committee on Foods for Special Dietary Uses.

#### RECONSIDERATION OF A TEST FOR FERMENTABILITY OF FRUIT JUICES

70. The Committee was informed that the Group of Experts had recently amended some standards for fruit juices to permit small quantities of sulphur dioxide present for reasons of carry over. The Committee was of the opinion that the only aim of this test was to assess whether or not a fruit juice was capable of fermentation. The Committee endorsed the proposed method in this sense only, i.e. under the condition that the title was changed into "Test for fermentability" and that the Scope section be amended. The Committee agreed to request the International Federation of Fruit Juice Producers to amend the method IFFJP No. 18 accordingly.

METHODS OF ANALYSIS IN STANDARDS FOR PROCESSED FRUITS AND VEGETABLES AND QUICK FROZEN FOODS

Determination of Drained Weight (Canned Mandarin Oranges and Canned Pears at Step 8)

71. The Committee endorsed the Method I of CAC/RM 36-1970.

Reconsideration of Determination of Mineral Impurities in Canned Strawberries and Quick-Frozen Strawberries

72. The Committee had before it a new proposal (swirling-decanting) for determination of mineral impurities (sand) in canned strawberries, which appears in the Report of the ninth session of the Codex Committee on Processed Fruit and Vegetables (ALINORM 72/20 A, Appendix XIV). The Committee was of the opinion that a single method should apply to both canned strawberries and quick frozen strawberries. The Committee was not yet in a position to discuss the proposed new method because of the unavailability of the document and decided to report its discussion to its eighth session.

Reconsideration of Determination of Calcium in Canned Strawberries and other Products

73. In view of obtaining uniformity of methods of analysis, the Committee endorsed the method AOAC, XI, 32.014-32.016 (complexometry), which also appears as CAC/RM 38-1970 and had been already endorsed for other products. This method was also endorsed for the determination of calcium in Jams (Fruit Preserves) and Jellies and in Citrus marmelade (Standards at Step 5).

Syrup Measurements (Canned Mandarin Oranges and Canned Pears at Step 8, Jams and Jellies and Citrus Marmelade at Step 5)

74. The refractometric method AOAC, XI, 31.011, 47.012 and 47.015 was endorsed for canned mandarin oranges and canned pears, and also 22.019 applicable to Jams and Jellies and Citrus marmelade only.

PROCESSED MEAT PRODUCTS

Determination of Total Fat Content

75. The Committee had before it the ISO method R1443 proposed by the Commodity Committee. The Committee provisionally endorsed this method for all standards at Step 8 but drew the Committee on Processed Meat Products' attention to the fact that a final endorsement would need collaborative studies and that, for referee purposes, it was necessary that only one solvent be selected.

COCOA PRODUCTS AND CHOCOLATE

Reconsideration of the methods endorsed at the sixth session

76. The Committee confirmed its previous endorsement of methods described in the report of its sixth session (ALINORM 71/23 para 62(a) to (m)) with the following amendments:

- (i) The endorsement of method IUPAC II.D.5.2 for unsaponifiable matter in cocoa butter (para 62-f) will be reexamined after the collaborative study presently under way (using light petroleum as well as ethyl ether) is achieved.
- (ii) The determination of heavy metals (Iron 62/g, Copper 62/h and Lead 62/j) will be included in the General Methods for Determination of Metallic Contaminants prepared by the Canadian delegation (see para 18-19).
- (iii) Cocoa powder and cocoa mass will be added to the list of products mentioned in the determination of total fat (62-m).

The delegation of Czechoslovakia pointed out that they would prefer the Wissmann apparatus, which gives a continuous extraction, to the Soxhlet apparatus.

Cut test (Cocoa Beans)

77. The Committee was informed that the Cut Test was a preliminary test for the examination of defective beans. The delegation of Australia drew the Committee's attention to a statistical study they had made on the number of beans to be taken. In its opinion this number of 300 provides an unnecessarily high risk factor. The delegation of Australia was requested to send its paper to the Secretariat. The method was temporarily endorsed, with reservations made by the delegations of Australia and the U.K.

Moisture content (loss on drying) in Cocoa Beans

78. The Committee was informed that the routine method proposed for endorsement (ISO DR 2291) was the only one presently developed by the ISO for the determination of moisture content of cocoa beans. The Committee endorsed this method.

Determination of pH

79. The Committee considered that the determination of pH was unnecessary for referee purposes and did not endorse the proposed method (AOAC/OICC as published in the AOAC, XI, 13.008).

Sampling of cocoa beans

80. (See para. 11).

Moisture content (loss on drying) in Chocolates

81. The Committee endorsed the method AOAC, XI, 13.001-13.002 (previously published as AOAC, X, 12.001-12.002) and the method OICC 3E (1952) (using sand) to be used alternatively for high fat as well as lower fat products.

Fat-free cocoa solids (or dry, fat-free cocoa solids) in sweet (plain) chocolate containing cocoa, sugar and fat only

82. The observer of the OICC informed the Committee that although the problem of cocoa dry, fat-free cocoa solids had not been solved properly, the method AOAC, XI, 13.033 was the only method available. A working party of the OICC had undertaken work, on the determination of purines (theobromine), which would be ready by 1973. The Committee endorsed the AOAC method with the reservations of Australia and Austria.

PART V

MISCELLANEOUS

FUTURE ACTIVITIES AND SCOPE OF WORK OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

83. The Committee was informed that the Commission, at its 8th Session, had requested the Committee to review the replies of Governments concerning its future activities and scope of work, but that no replies had been received in response to the circular distributed by the Secretariat. The delegation of Sweden was of the opinion that it would be desirable to review the work done by other international organizations engaged in the field of methods of analysis and sampling.

84. The delegation of the USA, supported by the delegation of the UK, was of the opinion that it was desirable to draw up guidelines setting out details of the procedure to be followed in submitting methods of analysis and sampling for consideration by this Committee. A definite orderly procedure should be developed for submission of methods to this Committee (e.g. through Commodity Committees, by assignment, etc.). Such a submission should include background materials, synopsis of methods considered and their references, and a summary of collaborative studies. The Secretariat was requested to prepare, with the assistance of the Hungarian secretariat, such a protocol based upon the USA proposal and UK suggestions.

85. The delegation of Poland informed the Committee that it had prepared a paper which was nearing completion setting out, in order of priority, the various sections dealing with general methods contained in Appendix II of the report of the 6th Session of the Committee (ALINORM 71/23) as requested at that session. The Committee agreed that the paper prepared by Poland should be placed on the agenda of its next session.

86. The delegation of Denmark proposed that, in the light of the experience gained at the last 7 sessions of the Committee it would be desirable to review the terms of reference of the Committee. The Committee requested the delegation of Denmark, with the assistance of the Secretariat, to prepare a paper for the next session. Some delegations were of the opinion that some confusion still existed concerning the nature of Codex methods of analysis. The Committee confirmed the view of the Executive Committee (Report of the 18th Session, ALINORM 72/3, para. 28) that Codex methods of analysis were intended to be adopted by Governments for use in dispute situations involving food in international trade, where no agreement could be reached concerning the results of analysis (see also para. 4).

## OTHER BUSINESS

### Determination of pesticide residues in food products

87. The delegation of the USA drew the Committee's attention to the difficulties experienced by the Codex Committee on Pesticide Residues in recommending methods for the determination of pesticide residues and pointed out that, therefore, it might be desirable to consider whether or not this Committee should assume responsibility for this work. The delegate of the Netherlands, speaking as Chairman of the Codex Committee on Pesticide Residues, pointed out that the Codex Alimentarius Commission had decided to charge the Codex Committee on Pesticide Residues with the task of recommending methods of analysis. That Committee had felt it difficult to establish referee methods and was, moreover, of the opinion that there appeared no need for such methods. It therefore decided to recommend, for the time being, so-called regulatory methods. Additional difficulties were due to the attempt to keep pace with new substances and applications as a result of constantly changing use of pest control agents. A number of delegations supported the view of the delegation of the USA and indicated further that it would be desirable for all methods of analysis to be considered by the Codex Committee on Methods of Analysis especially as the future work of this Committee appeared to make this possible. The Chairman of the Codex Committee on Pesticide Residues pointed out that a number of organizations, notably FAO in cooperation with IUPAC, were already engaged in work on pesticide residue analysis, which was a specialized field requiring close scrutiny of the nature of and tolerances for pesticide residues on which methodology depended. The delegation of the USA pointed out the availability of AOAC collaboratively studied multi-residue methods of analysis for pesticide residues, which had not been considered by the Committee on Pesticide Residues, and a number of delegations indicated that they would discuss this matter at the forthcoming meeting of the Codex Committee on Pesticide Residues. The Committee did not come to any decision on this matter but agreed that it had an overall responsibility for all matters relating to the analysis and sampling of food.

### Methods of analysis for edible fungi and fungus products

88. The Secretariat drew the Committee's attention to the decision of the 1971 Session of the Coordinating Committee for Europe to adopt all methods of analysis for the standards for edible fungi and fungus products, which had been endorsed by this Committee (see ALINORM 72/19, para. 20 and ALINORM 71/23, Appendix III). The Committee took note of this decision and also of the need to develop methods of analysis for mineral impurities (HCl insoluble) and salt content (NaCl) in fungi in oil, water content in dried fungi, freeze-dried fungi and dried fungus Shii-ta-ke and lactic and/or citric acid in sterilized fungi. Governments were invited to send information on the above to the Secretariat, as well as their comments on preparation of test sample in the case of determination of salt content in edible fungi and fungus products.

### Methods of analysis for natural mineral waters

89. The Committee was informed by the delegation of Switzerland that a meeting of experts had been held in Bern in April 1972 to discuss methods of analysis for mineral waters in the light of the new definition of that commodity adopted by the Coordinating Committee for Europe (ALINORM 72/19A). As first priority, the meeting of experts had proposed methods for the determination of total dissolved solids and free carbon dioxide, which were mandatory provisions in the standard on natural mineral waters. It had been also decided to include "short analysis" methods for the determination of a number of cations and anions (e.g.  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Mg}^{++}$ ,  $\text{Ca}^{++}$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{HCO}_3^-$ ,  $\text{SO}_4^{=}$  and silicic acid and for  $\text{Li}^+$  where the level exceeded 1 mg/litre). Some delegations were of the opinion that Codex referee methods were required only for those provisions of the standard which were mandatory and specifically stated in the standard. The delegation of Switzerland pointed out that the recognition of natural mineral waters by the responsible authorities can only be based on the results of analysis using scientifically recognized methodology which included chemical, physical, microbiological and other methods: for this reason it would be desirable to develop standard methods. The Committee did not come to any conclusion and decided to consider this matter at a future session.

## FUTURE WORK AND PRIORITIES

90. The Committee agreed that top priority should be given to the question of sampling (see paras. 5-11) and that, as a second priority, future activities should include the development of methods of general application. The endorsement of methods proposed by Commodity Committees was regarded as a continuing function. However, the Committee was of the opinion that it was essential to solve the question of sampling so as to make

Codex methods of analysis more meaningful.

DATE AND PLACE OF NEXT SESSION

91. The Committee noted that its sessions were on an 18 months interval basis and that the Commission would discuss a timetable of Codex sessions at its 9th Session in 1972. The Committee was strongly of the opinion that it was essential to hold a session within a year from its present session to discuss principally questions of sampling. It was also agreed that it was desirable for sessions of the Committee to be phased so as to be consecutive with sessions of ISO/TC 34, which met on a yearly basis. The 8th session of the Codex Committee on Methods of Analysis and Sampling would be held in Budapest.

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ALINORM 72/23  
APPENDIX I

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APPENDIX II

REPORT OF THE AD HOC WORKING GROUP ON SAMPLING

1. It has been stated that Codex methods are only intended for application in cases of dispute in international trade <sup>1/</sup>. It appears, however, that Codex Standards, when accepted, will in fact involve the incorporation of the methods of analysis and sampling into the regulatory procedures of the country concerned so that imports and domestic production will be treated on the same basis. Because of the differing points of inspection, equivalence of treatment may be more difficult to achieve in the case of sampling. Inspection of domestic production should be equivalent with that of import, which in many countries are examined at point of entry. This means that sampling should take

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<sup>1/</sup> Report of the 18th Session of the Executive Committee (ALINORM 72/3 para 28).

place when the domestic production first enters trade channels. It is only where large, identifiable lots exist that equivalent sampling plans can be applied.

2. Analytical methods can be compared objectively, whereas in sampling requirements there is the arbitrary selection of a risk of making an incorrect judgement. It is difficult to reach international agreement on an arbitrary matter of this nature and it would appear to be preferable to develop sampling requirements in the form of guidelines rather than as a mandatory part of the Codex Standards for referee purposes, in that such requirements would usually have to be adopted as official regulatory methods, i.e. form part of the law of the country after acceptance. Implicit in any sampling requirement is the acceptance of some product not meeting a tolerance, limit or other criterion. This inherent feature of any sampling requirement poses the legal difficulty in some countries of using evidence based on an interpretation of results containing an admitted margin of error. This is due to the fact that only a small portion of the merchandise has been examined and that the decision is based on the recognition that a portion of the consignment may not conform in some respect. This legal difficulty is especially critical for Codex standards where quality criteria are set at levels such that non-conforming items will be sub-standard. It is felt that, in principle, each and every item must conform. In practice, however, this requirement is circumvented by the application of administrative decisions as to the sampling and the degree of re-sampling before legal action is taken. Such administrative discretion is difficult to codify in mandatory standards. Furthermore in some countries such administrative discretion is not subject to central control.

3. The question therefore arises as to whether there should be referee sampling requirements for cases of an international dispute, which are likely to be extremely rare. It needs to be recognized that such cases will have to be dealt with on an individual basis taking into account the relevant circumstances. There would seem to be little possibility of developing any detailed requirements which could embrace the wide range of circumstances which might need to be taken into account. The most practical course would seem to be for agreement to be reached between the parties as to the taking of a representative sample and how this should be divided into portions and prepared for analysis.

4. It would appear, then, that sampling requirements in Codex standards could best be developed as guidelines or codes of practice to encourage uniformity in administrative procedures with the possibility that they may eventually be incorporated more formally. As recommended at an earlier session <sup>1/</sup>, the development of detailed sampling plans and selection of techniques of sampling should be the responsibility of Codex Commodity Committees. However this Committee should develop guidelines on sampling for the use of the other Codex Committees to ensure uniformity of terminology and approach in collaboration with ISO as is at present under way. This Committee should also have the task of examining sampling requirements to see if these guidelines have been met. The sampling plans so far included in the standards should also be reviewed.

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<sup>1/</sup> Report of the 5th Session of the Codex Committee on Methods of Analysis and Sampling (Appendix VII).