codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

JOINT OFFICE: Via delle Terme di Caracalla 00100 ROME: Tel. 5797 Cables Foodagri

ALINORM 76/23

CODEX ALIMENTARIUS COMMISSION
Eleventh Session, Rome, 29 March - 9 April 1976

REPORT OF THE NINTH SESSION OF THE
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Budapest, 27-31 October, 1975

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Ninth Session from 27-31 October 1975 in Budapest by the courtesy of the Government of Hungary. The Session was opened by Dr. K. Karcsai, General Secretary of the Hungarian National FAO Committee who welcomed the participants. Dr. Sütő, President of the Hungarian Codex Committee stressed the importance of arriving at agreed methods of analysis and sampling in relation to the international standardization of food. He introduced the Chairman of the Session, Professor R. Lasztity, Vice Rector of the University of Technology, Budapest. The Session was attended by delegates from 24 countries and observers from seven International Organizations. The list of participants, including officers from FAO, is contained in Appendix I to this Report.

ADOPTION OF THE AGENDA

2. Before discussing the adoption of the agenda several delegations pointed out that there had not been time to study all the prepared documents since some were available only on arrival at the meeting and others had been late in arriving in their countries. The Committee agreed to take such matters into consideration. The delegation of the Republic of Argentina made a general reservation on all matters being discussed at this meeting.

3. It was pointed out that item 5(b), Sampling for the Determination of Net Contents, was an important and specialized subject and this would best be dealt with by the setting up of a Working Group which would discuss this item only and report its conclusions to the Committee later in the Session. The Committee agreed to establish an ad hoc Working Group chaired by Dr. Anderson of the delegation of Canada with representatives of the delegations of Denmark, the Federal Republic of Germany, Hungary, the Netherlands, Norway, Switzerland and the United States, and of the EEC.

4. The Committee also agreed to appoint a small group consisting of representatives of Austria, the Federal Republic of Germany, the Netherlands and Poland, and chaired by Dr. Horwitz of the delegation of the United States, to consider item 6(a), Endorsement of Methods of Analysis and Sampling proposed by Codex Commodity Committees. This group would also take into account any relevant matters arising from item 4.

5. In discussion of item 5 of the agenda in plenary session the Committee agreed to take item 5(d) in advance of the other points under the item Sampling. This apart, the Committee agreed to the adoption of the agenda as presented.

APPOINTMENT OF RAPPORTEURS

6. Mr. R. Sawyer of the delegation of the United Kingdom and Madame Castang of the delegation of France agreed to act as rapporteurs.

MATTERS ARISING FROM THE COMMISSION AND CODEX COMMITTEES

7. The Committee had before it document CX/MAS 75/2 containing information of interest to the Committee. It was agreed that questions covered in the paper which related to later items on the agenda should be referred to in the appropriate items of the agenda.

Tenth Session of the Codex Alimentarius Commission

8. It was noted that the Commission had concluded that the revision of adopted methods included in Codex Commodity Standards or replacement with other methods might or might not constitute an amendment within the Codex meaning of the term. It would be a matter of judgement to be made in each individual case. Since matters of principle were involved the Committee was of the opinion that changes other than editorial ones should be submitted to governments for comment.

9. As regards the general principles for the establishment of Codex methods of analysis and as regards the terms of reference of the Committee (the Procedural Manual of the Commission, 4th Edition) the Commission had noted that the stated Principles and Terms of Reference were appropriate and adequate and required no change.

FAO/WHO Environment Activities

10. The Committee was informed of recent developments arising from the implementation of recommendations Nos. 78 and 82 of the UN Conference on the Human Environment. Financial assistance had been received from UNEP and in this way the FAO/WHO Food Contamination Monitoring Programme was complementing the work of the Codex Alimentarius Commission. UNEP had approved the setting up of a programme of consultation with experts to look into the question of appropriate methodology for determination of contaminants in food. The Committee requested that the results of these consultations be made available to the Committee so that any methods of analysis or sampling elaborated by the experts could be taken into consideration in the future work of the Committee. The view was expressed that where possible uniform methodology should be applied in surveys on foodstuffs. Note was also taken of the resolutions adopted by the International Conference on Ceramic Foodware Safety (CL 1975/25).

Codex Committee on Foods for Special Dietary Uses

11. The Committee noted that the above Committee had expressed the view that there was no need to elaborate Codex methods (a) for optional ingredients, unless these are known and specified in the standards and (b) for the identification of anions permitted in salt substitutes.

Codex Committee on Sugars

12. The Committee noted that document CAC/RM 42-1969 on Sampling Plans for Prepackaged Foods contained certain errors which would be corrected in a future edition of the Sampling Plans. The delegation of the USA pointed out that the results of collaborative studies carried out in testing of ICUMSA methods were no longer available. As a result a small interlaboratory study may be set up in the USA. Other member governments interested in collaborating in these studies would be welcomed.

Codex Committee on Fish and Fishery Products

13. The Committee noted that certain methods such as thawing and draining procedures were not truly analytical methods requiring endorsement by the Committee on Analysis. It was agreed that the treatment of such procedures was a matter of judgement to be applied in individual cases.

Codex Committee on Processed Meat Products

14. The Committee was informed that methods for the determination of nitrite and ascorbic acid in Canned Corned Beef should be put into the Standard by the Danish Secretariat, but that such methods had not been included in the standard at Step 8. The Secretariat was requested to clarify the situation with the Danish Secretariat of the Processed Meat Products Committee. The delegation of the USA drew attention to the fact that nitrite and nitrate methods were under evaluation by the IDF/ISO/AOAC Working Group.

Codex Committee on Fats and Oils

15. The Committee was informed that a list of "extraction" solvents had been established for use with edible oils and that methods of analysis were necessary for the determination of residues. In addition the list of "processing" solvents still required to be finalized. It was noted that IUPAC and AOAC were active in this field

and that methods would be submitted for endorsement in due course.

16. As regards the determination of water in margarine the Committee noted that a method using sand had been submitted to Governments for comments and that the Codex Committee on Fats and Oils would consider the method at its next session in London in November 1975.

Codex Committee on Edible Ices

17. It was noted that the above Committee had requested the Joint IDF/ISO/AOAC Working Group on Analysis to elaborate appropriate methods of analysis and sampling for edible ices and that such methods would, after consideration by the Edible Ices Committee, be submitted to this Committee for endorsement.

Joint EEC/Codex Group of Experts on Fruit Juices

18. The Committee noted that a Working Group on Methods of Analysis for Fruit Juices would meet following the present session to finalize methods of analysis, as requested in ALINORM 70/14 (para 24).

Joint Codex/IOOC Meeting on Table Olives

19. The Committee was informed that the 10th Session of the Commission had accepted certain editorial changes to the determination of salt content of brine, acidity and pH, and had deleted reference to the alternative method for salt content. The Commission had referred the choice back for reconsideration at the Joint Meeting on Table Olives. The delegation of the United Kingdom was of the opinion that the Volhard method rather than the method involving potassium chromate would be preferable as an alternative method.

DOCUMENTS RELATING TO SAMPLING

20. The Committee had before it for information documents CX/MAS 73/13-14 Rev., CX/MAS 15/2, 15/4 and an ISO/TC 34/WG 1 document No. 42 dealing with a sampling method for meat and meat products. Document CX/MAS 75/3 dealing with the question of net content was considered by the ad hoc Working Group set up during the session (see para 3) and the result of these deliberations was reported later (see paras 111 and 112).

21. As regards document CX/MAS 73/13-14 Rev. prepared by the Hungarian Secretariat the Committee expressed its appreciation to the Secretariat and agreed that the document contained valuable reference material for use by the ad hoc Working Group on Sampling as well as by the Committee itself in future work.

GENERAL PRINCIPLES FOR THE SELECTION OF CODEX PROCEDURES FOR SAMPLING

22. The Committee had before it the document CX/MAS 75/4 which contained the draft criteria prepared by the UK for selection of appropriate sampling provisions. It noted that the UK document started from the premise that the general principles were to be used to generate sampling procedures for use in the dispute situation. The Committee was informed that acceptance sampling plans already adopted by the Commission (CAC/RM 42-1969) were part of some commodity standards and in these cases were not restricted to a dispute situation only. Other sampling procedures such as that being developed by the Codex Committee on Pesticide Residues (see para 27) were practical procedures intended for use in situations where destructive testing was necessary. A historical summary of the work of the Committee with regard to Sampling was made by the delegation of Australia and is appended to this Report (Appendix II).

23. The delegation of the Netherlands was of the opinion that Codex methods of sampling should be drawn up for legal food control purposes to check compliance with the provisions of Codex standards. It was pointed out that Governments would be obliged to adopt the standards,and any sampling procedure contained therein, into legislation following acceptance of Codex Standards. In the opinion of the delegation of the Netherlands disputes of the type envisaged rarely occurred and in such cases it was not possible to draw a further identical sample for referee analysis. There was difficulty with the legal principle that each and every item should conform with particular requirements of a standard concerned with quality and health. This problem could be solved in the way proposed by the Codex Committee on Pesticide Residues (CX/MAS 75/2, Appendix II). However, statistical sampling plans (such as the acceptance type plans) could be drawn up for commercial purposes if it was thought that such plans would

facilitate trade. Other delegations were of the opinion that standard sampling procedures were required as the lack of harmonization in this field led to difficulties in international trade in food and that such procedures should be referee methods.

24. The Committee agreed that the Principles drawn up by the UK represented a suitable and concise first draft which should be further elaborated. It was also agreed that General Principles were indeed needed from which detailed sampling procedures could be worked out by the appropriate commodity committees. The Principles would define the purposes of sampling methods, and would also include further guidance for the development of appropriate methods. The delegation of the Netherlands stated that it had a document in preparation which would form the basis of a detailed discussion paper. A Working Group was formed consisting of the following countries: Australia, Canada, Hungary (liaison with ISO), Netherlands, Norway, Poland, Switzerland, UK (rapporteur)and the USA. These countries would collaborate with the UK by correspondence and would draw up revised Principles for consideration by the next Session of the Committee.

COLLABORATION WITH ISO

25. Dr. Kanizsay, speaking on behalf of ISO, described their work on sampling and also informed the Committee of a recent meeting of representatives of ISO, AOAC and Codex in Budapest at which cooperation between these bodies was discussed. He stressed the need for coordination and cooperation, particularly in the field of sampling, where much work was still to be done at an international level. It was also important to define the specific fields in which, on the basis of the particular competence of the above three bodies, a fruitful cooperation could be achieved. The Committee noted with satisfaction that the central Secretariat of ISO, AOAC and Codex had taken steps to ensure practical cooperation which would further the aims of the Commission.

SAMPLING PROCEDURES PROPOSED BY COMMODITY COMMITTEES

26. The Committee considered the inspection procedure for the determination of fat free protein content (PFF) proposed by the Codex Committee on Processed Meat Products for Cooked Cured Ham and Cooked Cured Pork Shoulder (see CX/MAS 75/2 or ALINORM 76/16). It was pointed out that there was an inconsistency between the numerical definition of zone L (i.e. 16.5% PFF or below) and the instructions contained in paragraph d) of the procedure which stated that "when the first sample drawn is below 16.5 the lot is not acceptable". The Committee endorsed the inspection procedure in principle but suggested that the following amendments were necessary to remove the inconsistency stated above:

Zone L:	$\leq$ 16.5% PFF	
Zone A:	$\geq$ 16.5% to	$<$ 17.3% PFF
Zone B:	$\geq$ 17.3% to	$<$ 18.0% PFF
Zone C:	$\geq$ 18.0% PFF	

27. The Committee noted that the Codex Committee on Pesticide Residues had developed a method of sampling for the determination of Pesticide Residues in food (see Appendix V, ALINORM 76/24 or Appendix II, CX/MAS 75/2) and had requested government comments thereon at Step 3 of the Codex Procedure. The Committee noted that in accordance with the Guidelines given in the Procedural Manual of the Commission (4th Ed.) the above sampling method would be submitted to it by the originating Committee for endorsement at an appropriate step (between Steps 3 to 5) of the Procedure. The Committee stressed the need for harmonization of sampling methods on the basis of agreed principles, which have yet to be finalized. It also reiterated that the terms of reference under which the Committee was required to function by the Codex Alimentarius Commission would ensure such a harmonization, only if the various committees followed the procedural rules in respect of submissions to the Committee.

ANALYTICAL METHODS

General Referee Method for determination of Chlorides in Foods

28. The Committee had before it a summary of Government comments (CX/MAS 75/6) on two general methods for the determination of chlorides in foods which had been circulated with CL 75/7 as Appendices I and II.

29. At its Eighth Session, the Committee had discussed these general methods (a)according to Charpentier-Volhard, proposed by the delegation of France, and (b) a potentiometric end point titration proposed by the delegation of USA (ALINORM 74/23, para 38).

30. The general referee method proposed by the delegation of the USA, which had previously been submitted to the Codex Committee on Foods for Special Dietary Uses (Appendix I, CX/MAS 73/7), had been collaboratively studied for canned meat and vegetables and had been endorsed for the determination of chloride (expressed as sodium chloride) in processed tomato concentrate (ALINORM 72/20, Appendix IV). The method now proposed in CL 75/7, Appendix II, had been collaboratively studied by 12 laboratories and had been validated for the determination of total chloride in a wide range of foods.

31. The Committee noted that governments which supported the potentiometric method did so because of its speed, simplicity and precision and that others considered that the Volhard method could be included as an alternative method because it used more generally available and simpler apparatus. The delegation of the Netherlands pointed out that in comparison tests they had encountered certain difficulties with both methods but that the potentiometric method had been clearly demonstrated to be the more generally applicable. The delegation also noted that in their hands the potentiometric method gave improved results when a back titration procedure was used.

32. The Committee agreed to endorse the potentiometric method for determination of chloride for use with Infant Foods, Processed Vegetable Products and Table Olives and to advance it as a general method under the title "General Referee Method for Determination of Chlorides in Foods (calculated as Sodium Chloride)" to Step 5 of the Codex Procedure (see Appendix IV of this Report).

33. It further agreed that in the official Codex Alimentarius text describing the method the characteristics of the equipment necessary should be specified but that trade names should as far as possible not appear except as an example on the basis of which the equivalence of equipment can be judged.

34. It was understood that any interested governments and international organizations could make a further collaborative study of the Volhard method if they wished and that any results submitted would be considered at a future Session of the Committee.

GENERAL METHODS FOR PRESERVATIVES

35. The Committee had before it a working paper, CX/MAS 75/7, containing the results of a joint AOAC/CCMAS collaborative study of a General Method for the Detection of Organic Preservatives in Food by Thin Layer Chromatography (TLC) which had been undertaken by seven European and two American laboratories. The delegation of the USA, introducing the paper, pointed out that the study showed that at present the method is not satisfactory for all the preservatives tested, and that further studies were necessary. A method will be required for the detection of formic, propionic and trichloroacetic acid. The Committee noted that it was important to be able to isolate and detect both permitted and non-permitted preservatives in foodstuffs and eventually to provide suitable quantitative methods for the permitted preservatives for application to commodities or groups of commodities.

36. Whilst it was appreciated that TLC was the preferred method for the detection of preservatives, it was pointed out that GC might prove to be useful in some cases although some delegations expressed reservations on the general applicability of the method at this time and were of the opinion that results should be confirmed by TLC. Other methods of detection, e.g. by infra-red spectrometry and combined GC mass spectrometry, were mentioned by some delegates.

37. The Secretariat pointed out that expensive methods such as detection by mass spectrometry were not acceptable in developing countries at this time and should not therefore be considered for inclusion in a general standard.

38. The Committee noted that the delegation of USA was willing to continue its research into general methods for the detection of preservatives and that the delegations of Australia, Belgium, Federal Republic of Germany, France, Netherlands, Norway, Spain, Switzerland and the UK expressed their willingness to take part in further collaborative studies.

39. The Committee agreed that future work should be coordinated by the delegation of the USA and, in order to help in the planning, that information on current methods in use by member governments together with information on the various preservatives used in member countries should be sent to Dr. Horwitz.

GENERAL METHODS FOR METALLIC CONTAMINANTS

40. The Committee had before it document CX/MAS 75/8 prepared by the delegation of Canada. This document updated the previous report (CX/MAS/70/C/2). In introducing the paper the delegation of Canada pointed out that only those methods which had been subjected to collaborative tests had been included in document CX/MAS 75/8. The delegation of Poland referred to a General Rule elaborated at the 2nd Session of the Committee (ALINORM 66/23, para 18) that interested international organizations whose methods were being considered by the Committee should make available to the Committee the publications containing the methods and the results of collaborative studies and that they should be forwarded in good time for detailed consideration (see para 2).

41. The Committee noted that an ISO/IDF/AOAC group would consider methods for the determination of metallic contaminants in milk and milk products in the future, and that an FAO/WHO Expert Committee would consider suitable methods for Hg, Cd and Pb early in 1976. As regards the methods in CX/MAS 75/8 under consideration, it was agreed that these would be elaborated as general international referee methods. As some of the analytical results obtained on application of these general methods to a wide variety of foods were subject to relatively large coefficients of variation, the delegation of the Netherlands expressed the opinion that individual methods might sometimes be preferable even though the great majority of Codex Standards have no contaminant section. It was pointed out that large coefficients of variation were to be expected in inter-laboratory studies.

42. The Committee agreed that the delegation of Canada be asked to continue to receive and collect information and comments on general methods for trace metals which have been the subject of collaborative studies.

Mercury

43. In considering the AOAC (1975) flameless AAS method, the Committee's attention was brought to a potential danger to operators when using a closed Teflon lined sealed crucible. The representative of AOAC expressed his appreciation for this notification and undertook to bring this matter to the attention of AOAC collaborating laboratories. He also informed the Committee that the amount of sample for digestion (1 g) referred to wet mass rather than dry mass which was not so stated in the AOAC method.

44. The delegation of the Netherlands reported that the AOAC method was not specific for Hg and also that there was evidence of interference when some types of foodstuffs were analyzed. Such interference was seen with milk products but could possibly also be experienced with other foods tested. The use of palladium chloride on glass wool, which specifically absorbed Hg, made it possible to determine whether the absorbance was due to Hg alone or Hg plus interfering substances. He also informed the Committee that by using the open tube system for reading the absorbance the quantity of sample used could be reduced to 200 mg and that this method had been tested collaboratively (Bureau International Technique du Chlorure Anal. Chim Acta 72 37 1974).

45. The Committee noted that to date no maximum limits had been set for Hg in foods by the Codex Alimentarius Commission. After some discussion as to whether a Codex referee method was required, it was agreed that it would probably be useful to endorse a method which governments could use in monitoring work. Results obtained from food control activities would then be of a comparable value and the usefulness of the data obtained would be enhanced. The Committee adopted the conclusions given in document CX/MAS 75/8 concerning the determination of Hg and requested governments to send their comments at Step 3 of the Procedure on the method (AOAC(1975)XII, 25.103 - 25.107). It was also agreed that the problems raised in connection with interfering substances and the possible hazard from the use of closed digestion systems would be considered at the next session following receipt of information from governments. In the interests of public safety governments and organizations are urged to transmit in writing information on safety hazards of any method to the sponsor immediately.

Lead

46. In considering the AOAC (1975) AAS method the Committee noted that, with the exception of evaporated milk which was studied at levels down to 0.1 ppm, and fruit juice at 0.5 ppm, the method proposed had been collaboratively tested at levels from 2 to 30 ppm: maximum limits proposed by Codex for this contaminant were generally below 2 ppm and the Committee considered that collaborative tests were needed to establish the status of the method at levels below 2 ppm. It was also noted that a number of methods, e.g. earlier AOAC colorimetric methods and ICUMSA methods, had already been endorsed for fats and oils and sugars respectively, and that the Committee on Fruit

Juices was considering methods elaborated by IFFJC (International Federation of Fruit Juices Committee.

47. The Committee agreed to consider the method of the AOAC (1975)XII, 25.060-25.064 as a general method and requested governments to send their comments at Step 3 of the Procedure. It was agreed that the method would be reconsidered at the next session in the light of comments and in the light of further collaborative tests (see para 46 above). As regards the AOAC methods for evaporated milk and fish (AOAC (1975) XII, 25.065-25.086),the Committee agreed that these methods should be considered at the next session as possible methods for these commodities and that they should be placed at Step 3 of the Procedure.

Arsenic

48. The delegation of the Netherlands was of the opinion that the B.S. method, which was a slight modification of the AOAC (1965) method, gave better results. The Committee, noting that the method of the AOAC (1965) X. 24.008, 24.011, 24.016 and 017 had already been endorsed for a number of foods, adopted it as a general method and requested governments to send their comments at Step 3 of the Procedure. It was also agreed that the modification to the AOAC method would be considered by the delegation of Canada. The Committee also considered that a study of the data from collaborative tests on this and other procedures recently carried out in the UK should be made by the delegation of Canada.

Cadmium

49. The delegation of the UK considered that the coefficient of variation of the AOAC (1975) AAS method was too large to make this method suitable as an international referee method. It pointed out that separation by use of chelating agents followed by direct aspiration of the organic solvent phase gave better results. In reply the representative of AOAC pointed out that such variations following inter-laboratory tests were not unusual and should be expected at the low concentrations studied. Intra-laboratory variation, of course, was usually of a lower magnitude. The Committee noted the reservations expressed by the UK and adopted the method of the AOAC (1975) XII. 25.026-25.030. It further requested Governments to comment at Step 3 of the Procedure. It was noted that no maximum limits had as yet been included for cadmium in Codex Standards.

Copper

50. The Committee adopted the AOAC (1975) AAS method AOAC (1975) XII. 25.041-25.045 as a general method and requested governments to comment at Step 3 of the Procedure. It was considered that the methods previously endorsed for individual foods could eventually be considered as suitable alternative methods.

Zinc

51. The Committee adopted the AOAC (1975) AAS method AOAC (1975) XII. 25.143-25.147 as a general method and requested governments to comment at step 3 of the Procedure. It was considered that the methods previously endorsed for individual foods could eventually be considered as suitable alternative methods.

Tin

52. The Committee noted that no satisfactory methods had as yet been elaborated which could be considered as fulfilling the requirements of Codex and that, furthermore, work was in progress on AAS methods for the determination of tin in food. The Committee decided to await the outcome of such work before taking further action on the two methods (AOAC (1970) XI. 25.008, 25.075-25.076 and Analytical Methods Committee, Analyst, 92, 320 (1967)) as possible general methods.

INTERNATIONAL ORGANIZATIONS WORKING IN THE FIELD OF ANALYSIS

53. The Committee had before it a paper (CX/MAS 75/9) prepared by the delegation of the Federal Republic of Germany. The contents were divided into four parts:

1. List of names, addresses and abbreviations of organizations
2. Which organization is working in what field
3. Collection of methods
4. Individual methods in preparation or already available orginating from different institutions.

54. The Committee expressed its appreciation to the delegation of the Federal Republic of Germany for the preparation of the document which would be revised in the light of amendments, corrections and suggestions submitted by delegates. The question was raised as to whether the lists should be restricted to international organizations. It was agreed that national lists would be of value and it would be left to the FRG to cull the material supplied.

55. The Committee noted that it was the intention of the delegation to review the contents when necessary and to complete section 4 in detail. Because of the work involved the document would be published in English only.

METHODS FOR THE DETERMINATION OF CRUDE FAT

56. The Committee had before it the report of the Collaborative Study on Fat Determination in Infant Foods (CX/MAS 75/10) which had been organized by the delegation of the Netherlands following a request to this Committee from the Codex Committee on Foods for Special Dietary Uses (ALINORM 74/23, paras 26 and 27).

57. It was reported that three methods had been studied on eight samples of baby food with different fat contents. The results showed that:

(1) the Schmid-Bondzynski-Ratzlaff method could not be recommended.
(2) the Weibull-Stoldt method was applicable to all the foods tested and could be recommended as a general method.
(3) the Röse-Gottlieb method could be recommended as an alternative method for infant foods containing sugar and/or dextrine maltose, but not for those containing starch, meat or vegetable products. Further work was necessary before the Röse-Gottlieb method could be used for materials containing starch.

58. The delegation of the Netherlands drew attention to the statistical analysis contained in the report which illustrated that duplicate determinations were of limited value in exercises of this kind. The delegation of the United States suggested that the report be submitted to the AOAC for future publication.

59. The Committee thanked the delegation of the Netherlands for the organization of the study and were of the opinion that this was an excellent example of an international collaborative study to establish the reliability of a proposed Codex Method.

60. The Committee decided to endorse the Weibull-Stoldt method and, as an alternative, the Röse-Gottlieb method with the limitations already stated.

COLLABORATIVE STUDIES GENERAL POLICY

61. With regard to the organization of collaborative studies in general the Committee agreed that the government responsible should be clearly stated in the future reports of the meetings.

ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PROPOSED BY CODEX COMMODITY COMMITTEES

62. The Committee had before it the report of the ad hoc Working Group (presented by the Chairman of the Group, Dr. W. Horwitz), documents CX/MAS 75/5, CX/MAS 75/5, Add.1 and Conference Room Document No. 6.

63. The delegate of Australia wished it to be recorded that he was the sole representative of Australia and did not consider that there was sufficient time to assimilate the contents of the reports of the two Working Groups.

64. The Committee noted that the ad hoc Working Group wished to call to the attention of the Secretariat the implication of the decision made at the second session of the Codex Committee on Methods of Analysis and Sampling (ALINORM 66/23, para 18) that international organizations sponsoring methods of analysis for endorsement must make available to governments through the Codex Secretariat a copy of each method for which endorsement was requested.

CODEX COMMITTEE ON COCOA PRODUCTS AND CHOCOLATE

Draft Standard for Cocoa Butters

65. Melting Behaviour (Fincke)

(i) Slip point — According to IOCC
(ii) Clear melting point — 8 b 1961

The Committee noted that these were methods specific for cocoa butter recommended by the international organization specializing in this product, and agreed to their endorsement.

66. Unsaponifiable Matter (petroleum ether) (IUPAC II.D.7.3 revised by AOAC/OICC)- This method is supported by a collaborative IOCC/AOAC study published in JAOAC. The Committee agreed that copies of the study and the text of the method should be circulated, following which the method could be endorsed.

Draft Standard for Chocolate

67. Total Ash - The Committee noted that an AOAC/IOCC collaborative study had been completed and published (JAOAC 55, 1027 (1972)) and endorsed the method published in AOAC (1975) XII. 13.003.

o

CODEX COMMITTEE ON PROCESSED MEAT PRODUCTS

Draft Standard for Canned Corned Beef

68. Protein (ISO/R 937) - The Committee noted that this method utilized copper catalyst and boric acid absorbent about which there was some controversy. It decided to postpone discussion pending the development of a general method for Kjeldahl nitrogen in foods requested by this Committee from ISO-AOAC.

Draft Standard for Luncheon Meat

69. Fat (ISO/R 1443) - The Committee endorsed this method; the details corresponded with those of the method studied collaboratively for foods for infants and children which, especially for meats and vegetable products, gave excellent results (see CX/MAS 75/10 and paras 56-60 of this Report).

CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

Draft General Standard for Jams (Fruit Preserves) and Jellies

70. Determination of Mineral Impurities (CAC/RM 49-1972, p.9)- the Committee endorsed the method.

Draft Standard for Citrus Marmalade

71. Sampling (CAC/RM 42-1969) - The Committee agreed that this method be held pending a decision on the general principles of sampling.

72. Test Procedures - soluble solids (AOAC (1975) XII. 22.024 and 31.011) - The Committee noted that the test procedures applied a standard collaboratively studied method (JAOAC 15, 384 (1932)) to an applicable commodity and endorsed the method.

73. Water capacity of Containers (CAC/RM 46-1972) - The Committee endorsed the method.

Draft Standard for Canned Mature Processed Peas

74. Dry Solids Content (AOAC (1975) XII. 32.004, expressed as % dry solids)- The Committee endorsed the 70° vacuum oven version of this method 32.004 (3) which has been collaboratively studied (JAOAC 57.1193 - 1197(1974)).

JOINT ECE/CODEX ALIMENTARIUS GROUP OF EXPERTS ON STANDARDIZATION OF QUICK FROZEN FOODS

Draft Standard for Quick Frozen Peaches

75. Sampling (CAC/RM 42-1969)

The Committee decided that this method should be held pending a decision on the general principles of sampling.

76. Thawing Procedure (CAC/RM 32-1970) - The Committee endorsed the method.

Test Procedures:

77. Net Weight (Section 8.3 CAC/RS 41-1970, CAC/RM 34-1970) - The Committee endorsed the method.

78. Drained Fruit partial (surface) thawing and draining with washing off of solid sugar when necessary. The Committee endorsed the method.

Analysis:

79. Total Soluble Solids (CAC/RM 36-1970) - The Committee endorsed the method.

Draft Standard for Quick Frozen Bilberries

80. Sampling (CAC/RM 42-1969) - The Committee decided that this method should be held pending a decision on the general principles of sampling.

81. Thawing Procedure (CAC/RM 32-1970) - The Committee endorsed this method.

Test Procedures:

82. Net Weight (Section 8.3 CAC/RS 41-1970, CAC/RM 34-1970) - The Committee endorsed the method.

83. Drained Fruit (see para 78 above) - The Committee endorsed the method.

Analysis:

84. Total Soluble Solids (CAC/RM 36-1970) - The Committee endorsed the method.

85. Mineral Impurities (AGRI/WP.1/GC.3/CRP No. 5, Appendix I) "Mineral Impurities in Quick Frozen Fruits and Vegetables" - The Committee noted that the document cited was not available. The Committee agreed to postpone its decision until such time as the method could be compared with the standard Codex method CAC/RM 49-1972, p.9.

CODEX COMMITTEE ON FOODS FOR SPECIAL DIETARY USES

Foods for Infants and Children

86. Quality of Protein (Protein Efficiency Ratio) - The Committee endorsed the AOAC method (1975) XII. 43.183-43.187. This is a well standarized test for a protein, not for a diet, so exceptions for all possible special dietary conditions are impracticable. The Committee noted that there was a possibility of lactose interference in the estimation of PER and agreed that governments should be requested to supply evidence to the AOAC.

87. Carbohydrate Determined by Difference - The Committee agreed that the term "available carbohydrates" be changed to "carbohydrate determined by difference" to differentiate this method of determination from the method where the carbohydrates may be determined directly or biologically.

88. The Committee noted the discussion of methods for crude fibre and that substitutions of enzymatic digestion or other methods for the classical acid/alkali digestion or other methods for the classical acid/alkali digestion require further demonstration of equivalence and applicability. The AOAC/ISO joint text is still pending.

89. Calories by calculation (ALINORM 76/26 A, para 60) - The Committee noted that the Committee on Foods for Special Dietary Uses deemed the term "available calories" inappropriate, and suggested that the terms "calories by calculation" could be substituted.

90. Linoleic Acid - The Committee noted that no method is yet available or endorsed. The question as to the correct method for elaboration included the problem of extraction of linoleic acid from foods without alteration. The Working Group was informed that IUPAC has performed an inter-laboratory collaborative study on fats using a combination of the lipoxidase and GLC methods with satisfactory results. The Committee agreed that IUPAC should be requested to supply a report of this study.

91. Vitamin K_1 - The Committee was informed that the AOAC will appoint an Associate Referee to study the recently proposed GLC method for Vitamin K_1. The delegations of Switzerland and the UK indicated their willingness to participate in the inter-laboratory study. Any laboratory wishing to participate should inform the AOAC, Box 540 Benjamin Franklin Station, Washington D.C 20044, USA.

92. The Committee noted that the chick bioassay method appears to be impracticable as a reference method and did not therefore endorse it.

Appendix VI - Report of the Ad Hoc Working Group on Methods of Analysis in Standards for Foods for Infants and Children

93. Fat - endorsed (see para 69 above).

94. Crude Fibre - (see para 88 above).

95. Carbohydrates - (see para 87 above).

96. Crude Protein - It was pointed out that there was an inconsistency in the Report of the 7th Session of the Codex Committee on Foods for Special Dietary Uses with respect to the conversion factors to be used for protein in mixtures which contained variable amounts of the three major sources of protein (ALINORM 74/26, para 9).

97. The Committee agreed that the sentence beginning in line 10 of para 9 should be better understood as follows: "It is further recommended that where a food is comprised of a known major amount (e.g. 80% dry weight) of either wheat cereal, soya or milk derived protein ingredients, the factor used for that protein shall be the appropriate factor as shown above. Where the remaining protein is of an unknown mixture (of these proteins) the factor 6.25 shall be applied to the remaining amount of protein."

98. Linoleic Acid - (see para 90 above)

99. Protein Efficiency Ratio (PER) -(see para 86 above)-The Committee agreed to endorse the method AOAC (1975) XII. 43.183-43.187.

100. Vitamin A - The Committee noted that the present state of knowledge did not allow for the recommendation of a single method for the widely different commodities, margarine and mixed feeds. Therefore the two methods should remain as endorsed. If foods for infants and children do not include margarine or similar products, there is only one applicable method.

101. Ascorbic Acid - The Committee noted that because of special interferences encountered with specific foods, it was difficult to have a single method applicable to all foods. A single universal method was not yet available and the two methods should therefore remain as endorsed.

102. Pantothenic Acid - The Committee noted that there was a method (AOAC) for enriched foods and another (USDA) for unenriched foods which require enzymatic liberation. There is therefore no overlap if infant foods are enriched with pantothenic acid.

103. Carotenes - No comment was considered necessary.

104. General Considerations - No comment was considered necessary.

OTHER MATTERS

105. Loss on Drying - The Committee noted that this determination was necessary for the calculation of calories. It agreed to request specific comments from governments regarding analytical problems that they may have encountered with products containing whey, meats, and vegetable and fruit products. Such comments, preferably with data comparing proposals for their solution with the temporarily endorsed method, should be sent to Dr. P.L. Schuller, National Institute of Public Health, P.O. Box 1, Bilthoven, Netherlands.

106. Ash - The Committee agreed to request comments from governments particularly regarding predrying, when necessary, and ashing overnight at not more than 550°. These should also be sent to Dr. Schuller.

107. Iodine - The Committee noted that an AOAC Associate Referee report indicating the inapplicability of the endorsed method for iodine will be submitted to the Secretariat for circulation. A recommended method is not yet available. Since the method was applicable to iodized salt only, the Committee agreed that the endorsement should be revoked.

108. Sodium and Potassium - The Committee agreed to ask for comments from governments which should be sent to Dr. Schuller. The temporary endorsement should remain.

109. Vitamin E - The Committee noted that the Working Group was informed that IUPAC was currently reviewing methods for Vitamin E applicable to oils. It agreed that the current method should be endorsed and its status reviewed when a report was received from IUPAC.

110. Vitamin D - The delegation of the United Kingdom reported that five laboratories had agreed to participate in a study using their own methods. The methods used at present are lengthy and involved. The Committee noted that the work is continuing and further results will be reported when available.

REPORT OF THE AD HOC WORKING GROUP ON ACCEPTANCE SAMPLING FOR THE DETERMINATION OF NET CONTENTS

111. The Committee had before it the Report of the above mentioned Working Group. Mr. G.E. Anderson, Chairman of the Group, introduced the Report and gave an outline of the issues involved by giving a statistical expression to the meaning of net contents in relation to lots of prepackaged foods. He informed the Committee that the issue of internationally acceptable sampling plans had been reduced basically to two positions. These differed somewhat in the acceptance probability of lots averaging at the declared net content. The Working Group had reached agreement on an approach which might be acceptable internationally. However, complete details of such an acceptance sampling plan had not been worked out.

112. In view of lack of time the Committee did not consider the Report of the Working Group in detail and agreed that it would consider it at its next Session. It also agreed that the Report of the Group should be appended to the Report of the Committee (Appendix III) for the information of governments. As regards the recommendations of the Group to the Committee, it was agreed that it was desirable to study the proposed approach for a "Moderate Acceptance Plan" and requested the Working Group, under the chairmanship of Mr. G.E. Anderson, to develop details of such a plan for the next session of the Committee. Delegations were invited to send their comments on the above approach to the Working Group (i.e. to Mr. Anderson). It was also agreed that the "Moderate Acceptance Plan", as developed by the Working Group, should be sent to governments for comment after consideration by the Committee at its next session.

OTHER BUSINESS

113. The delegate of the USA wished to remind the representative of ISO that the joint AOAC/ISO method for the determination of crude fibre and Kjeldahl Nitrogen was still to be finalized.

114. The delegate of Australia drew the Committee's attention to the fact that no work had been undertaken on the method for the determination of mineral oil in raisins as agreed at the Eighth Session (ALINORM 74/23). In addition he sought further information on the outcome of the IUPAC Symposium on Collaborative Studies. The Committee was informed that no action had been taken in the Food Section of IUPAC but that ISO/TC 69 had prepared a text on collaborative tests.

FUTURE WORK

115. The Committee noted that there was sufficient outstanding work in progress and a number of methods still required endorsement or reconsideration. It was therefore agreed that there was no need to discuss additional further work. The Secretariat undertook to examine previous reports in order to ensure that all such matters would be placed before the next session of the Committee.

DATE AND PLACE OF THE NEXT SESSION

116. The Committee was informed that the next session would be held in Budapest during the first half of 1977.

LIST OF PARTICIPANTS*
LISTE DES PARTICIPANTS
LISTA DE PARTICIPANTES

Chairman

Prof. R. Lasztity
Technical University
Belgrád rkp. 3
H-1111 Budapest

Secretary

Dr. D. Törley
Assistant Professor
Technical University
Belgrád rkp. 3
H-1111 Budapest

———

ARGENTINA
ARGENTINE

Mr. A. Moschini
Secretary, Embajada a Budapest
Rippl Rónai utca 1.
H-1068 Budapest

AUSTRALIA
AUSTRALIE

Dr. F.E. Peters
Australian Government Analyst
P.O. Box 449
Woden A.C.T. 2606

AUSTRIA
AUTRICHE

Dr. H. Woidich
Director
Lebensmittelversuchsanstalt
Biaasstr. 29
A-1190 Wien

BELGIUM
BELGIQUE
BELGICA

Mrs. S. Srebrnik
Ministère de la Santé Publique
Laboratoire des Denrées Alimentaires I.H.E.
14, rue Juliette Wytsman
1050 Bruxelles

BULGARIA
BULGARIE

Dr. M.G. Marinov
DSO. Zarnani Hrani
Bul. Vitoscha 15
Sofia

CANADA

Dr. G.E. Anderson
Director
Legal Metrology and Laboratory Services
Department of Consumer and Corporate Affairs
Ottawa, Ontario

Dr. J.P. Barrette
Dept. of Agriculture Canada
Laboratory Service Section
Ottawa, Ontario K1A OC5

CUBA

Prof. N.L. Navarro Figueira
Food Hygiene Department
Inhem, Infanta y Crucero
La Habana

CZECHOSLOVAKIA
TCHECOLOSVAQUIE
CHECOSLOVAQUIA

Mr. J. Barvir
Quality Inspection of Food
18000 Pobrezni 10
Prague-8 - Karlin

---

* The Heads of Delegations are listed first.
Les chefs de délégations figurent en tête.
Figuran en primer lugar los jefes de las delegaciones.

CZECHOSLOVAKIA (cont.)

Mr. M. Fellegiova
Research Institute of Hygiene
Bratislava

Dr. L. Kroutil
Hygienist
Koospol Foreign Trade Corp.
Oljanska 1
Prague-3

Mr. J. Laciny
Quality Inspection of Food
Pobrezni 10 18000
Prague-8

Mr. D. Prochazka
Quality Inspection of Food
Podjavorinskej 4
Bratislava

DENMARK
DANEMARK
DINAMARCA

Mr. P.F. Jensen
Director
Inspection Service for Fish Products
Ministry of Fisheries
Dronningens Tvaergade 21
Copenhagen K

FINLAND
FINLANDE
FINLANDIA

Mr. E. Pajunen
Research Officer
Technical Research Centre of Finland
Biotechnical Laboratory
Box 192 SF-00121
Helsinki 12

Mr. P.L. Penttila
Inspector of Foods
National Board of Trade and Consumer Interests
Haapanienmenkatu 4 A SF-00530
Helsinki 53

Mr. E. Petaja
Board of Customs
Erottajankatu 2
SF-Helsinki 10

Dr. M. Raevuori
First Assistant
Department of Food Hygiene
College of Veterinary Medicine
Hameentil 57 SF-00550
Helsinki

FRANCE
FRANCIA

Dr. J. Castang
Laboratoire Interrégional de la Répression des Fraudes et du Contrôle de la Qualité
2, rue St. Pierre
34000 Montpellier

Mrs. C. Soules
Commission Générale d'Unification des Méthodes d'Analyse
Service Répression des Fraudes et Contrôle de la Qualité
42bis, rue de Bourgogne
75000 Paris

GERMANY, FED. REP.
ALLEMAGNE, REP. FED.
ALEMANIA, REP. FED.

Prof. W. Krönert
Director
Bundesgesundheitsamt
1 Berlin 33 Postbox

Dr. H. Meyer
Nestlé-Haus
Frankfurt-Niederrad

Dr. W. Trapp
Baudirektor, Bayer
Landesamt für Mass und Gewicht
Franz Schrank Str. 9
G-8 München 19

Dr. P. Vogel
Südplan 44
D-4194 Bedburg-Hau

HUNGARY
HONGRIE
HUNGRIA

Dr. N. Albert
Secretary of the Hungarian National Codex Committee
H-1091 Budapest

Mr. K. Kismarton
Head of Section
Ministry of Agriculture and Food
Kossuth L. tér 5
H-1680 Budapest

Prof. K. Lindner
Academy of Commerce and Gastronomy
Alkotmány u. 9-11
H-1054 Budapest

Dr. L. Körmendy
Deputy Director
Hungarian Meat Research Institute
Gubacsi ut 6/b
H-1094 Budapest

HUNGARY (cont.)

Prof. F. Ürsi
Technical University
Institute of Biochemistry and Food Technology
Belgrád rkp. 3
H-1111 Budapest

Prof. J. Takács
Director of Veterinary Meat Control Service
Soroksári u. 58
H-1095 Budapest

Mr. L. Vékony
Ministry of Agriculture and Food Industries
Kossuth L. tér 11
H-1680 Budapest

Mr. E. Zukál
Dozent, University for Agriculture
Institute of Microbiology
H-4200 MOSONMAGYARÓVAR

IRELAND
IRLANDE
IRLANDA

Dr. T.M. O'Toole
Food Scientist
Department of Agriculture and Fisheries
Kildare Street
Dublin 2

MOROCCO
MAROC
MARRUECOS

Mr. M. Boublouh
Inspecteur de la répression des fraudes
Oujda

Mr. A. Janah
Ministère de l'Agriculture
Service de la répression des fraudes
Rabat

NETHERLANDS
PAYS-BAS
PAISES BAJOS

Dr. J. Eisses
Ministry of Agriculture and Fisheries
Rijkszuivelstation
Vreewijkstraat 12 b
Leiden

Dr. P.W. Hendrikse
Anal. Chemist,
Unilever Research Laboratory
Postbox 114
3170 Vlaardingen

Dr. A. Kruysse
Ministry of Public Health and Environmental Protection
Dokter Reijersstraat 12
Leidschendam

NETHERLANDS (cont.)

Mr. M.J. Osse
Ministry of Agriculture and Fisheries
Department of Agricultural Industries and International Trade
73 Bezuidenhoutseweg
The Hague

Dr. P.L. Schuller
National Institute of Public Health
Postbox 1
Bilthoven

Dr. G.F. Wilmink
Cabinet Adviser
Ministry of Agriculture and Fisheries
73 Bezuidenhoutseweg
The Hague

NORWAY
NORVEGE
NORUEGA

Prof. O.R. Braekkan
Government Vitamin Institute
Directorate of Fisheries
P.O. Box 187
5001 Bergen

Mr. A.O. Hougen
Director Pack Institute NLH
Box 64
1432 Ås

Mr. S. Nossen
Government Analyst
Food Inspection
Ministry of Agriculture
Gladengveien 3B
Oslo 6

POLAND
POLOGNE
POLONIA

Dr. B. Brzozowska
State Institute of Hygiene
Chocimska 24 (Bad. ZYWN)
00-791 Warsaw

Dr. M. Dabrowska
Institute of the Fermentation Industry
Rakowiecka 36
Warsaw

Mr. W. Martinek
Ministry of Foreign Trade and Shipping
Quality Inspection Office
Stepinska 9
Warsaw

Mr. A. Zaboklicki
Ministry of Foreign Trade and Shipping
Quality Inspection Office
Czolgistow 8/12
81-378 Gdynia

SPAIN
ESPAGNE
ESPAÑA

Dr. R. García Faure
Jefe Laboratorios Agrarios Regionales
Ministerio de Agricultura
Madrid

SWEDEN
SUEDE
SUECIA

Prof. L. Reio
National Food Administration
Box 622
S-75126 Uppsala

SWITZERLAND
SUISSE
SUIZA

Prof. P. Koch
Swiss Federal Office of Weights
and Measures
Lindenweg 50
CH-3084 Wabern

Dr. G. Frey
Société d'assistance technique pour
produits
NESTLE
CH-1814 La Tour-de-Peilz

Dr. B. Kovaliv
Société d'assistance technique pour
produits
NESTLE
CH-1814 La Tour-de-Peilz

Mr. H.U. Pfister
Head of Codex Section
Federal Health Service
Haslerstrasse 16
3008 Bern

THAILAND
THAÏLANDE
TAILANDIA

Mrs. V.Charutamra
Senior Scientific Officer
Head of Food Analysis Section
Biological Science Division
Department of Science
Ministry of Industry
Rama VI
Bangkok 4

Mrs. S. Siwaraksa
Senior Scientific Officer
Food Analysis Division
Department of Medical Sciences
Yodse
Bangkok

UNITED KINGDOM
ROYAUME-UNI
REINO UNIDO

Mr. R. Sawyer
Superintendent
Food and Nutrition Laboratory
of Government Chemist
Stamford St.
London SE 1 9 NQ

Mr. C.D. Usher
Analytical Chemist
Unilever Research Lab.
Colworth House, Sharnbrook, Bedfordshire

Mr. R. Wood
Senior Scientific Officer
Food Science Division
Ministry of Agriculture, Fisheries and Food
Great Westminster House
London, SW 1 P 2 AE

UNITED STATES OF AMERICA
ETATS-UNIS D'AMERIQUE
ESTADOS UNIDOS DE AMERICA

Mr. Ch. H. Brokaw
Director of Quality Assurance Coca-Cola USA
P.O. Drawer 1734
Atlanta, GA 30345

Mr. E.R. Elkins
Manager Chemistry Division
National Canners Association
1133 20th Street N.W.
Washington D.C. 20036

Dr. W. Horwitz
Deputy Associate Director for Science
Bureau of Food HFF-101 Food and Drug
Administration
Washington D.C. 20204

Mr. P. Khan
Director
Food Protection ITT/Continental
P.O. Box 731 Rye
N.Y. 10580

Mr. J.S. Winbush
Acting Director
Division of Mathematics
HFF-110 Food and Drug Administration
200-C Street, S.W.
Washington D.C. 20204

Mr. B. Levy
Director
Statistical Staff USDA/APHIS/MDI
12th St. Independence Ave. S.W.
Washington D.C. 20250

Mr. B. Larsen
Chief Staff Officer
STS, APHIS
US Department of Agriculture
Washington D.C. 20250

UNITED STATES OF AMERICA (cont.)

Mr. J.A. Yeransian
Senior Laboratory Manager
Analytical Laboratory
General Foods Central Research
20050 North Street
White Plains
New York 10625

INTERNATIONAL ORGANIZATIONS
ORGANISATIONS INTERNATIONALES
ORGANIZACIONES INTERNACIONALES

AOAC
(Association of Official Analytical Chemists)

Mr. B. Larsen
Chief Staff Officer, SIS, APHIS
U.S. Department of Agriculture
Washington D.C. 20250
U.S.A.

Dr. J.P. Barrette
Agriculture Canada
Laboratory Service Section
Ottawa
Ontario K1A OC 5
Canada

Dr. W. Horwitz
Executive Director
Box 540, Benjamin Franklin Station
Washington D.C. 20044, U.S.A.

CCE
(Commission des Communautés Européennes)

Mr. O. Demine
Administrateur
Direction Générale de l'Agriculture
Commission des Communautés Européennes
200, rue de la Loi
Bruxelles, Belgium

Mr. G. Vos
Administrateur Principal D.C.
Marché Intérieur CCE
200, rue de la Loi
Bruxelles, Belgium

ICC
(International Association for Cereal Chemistry)

Dr. A. Mosonyi
GTKI Budapest
Kisrókus u. 15/b
Budapest II
Hungary

ISDI
(International Association of Dietetic Food Industry)

H. Meyer
Sekretariat Diatetische Lebensmittel Ind.
Kelkheimer Str.
D-6380 Bad-Homburg
Germany, F.R.

ISO
(International Organization for Standardization)

J. Kanizsay
Deputy Technical Director
Hungarian Office for Standardization
Üllöi ut. 25
H-1091 Budapest, Hungary

ISO/TC 34 SECRETARIAT

Mr. P. Kiss
Senior Technical Officer
Hungarian Office for Standardization
Üllöi ut. 25
H-1091 Budapest, Hungary

ISO/TC 34 SECRETARIAT (cont.)

Mr. A. Sándor
Senior Technical Officer
Hungarian Officer for Standardization
Üllői ut. 25
H-1091 Budapest, Hungary

UNIDO
(United Nations Industrial Development Organization)

Dr. A. Miklovicz
Chief of Light Industry Section
P.O. Box 707
H-1011 Vienna
Austria

FAO/CODEX SECRETARIAT

Mr. J.M. Hutchinson
Food Standards Officer
Joint FAO/WHO Food Standards Programme
Via delle Terme di Caracalla
FAO
00100-Rome, Italy

Dr. L.G. Ladomery
Food Standards Officer
Joint FAO/WHO Food Standards Programme
Via delle Terme di Caracalla
FAO
00100-Rome, Italy

GENERAL BACKGROUND ON SAMPLING

(presented by the Delegation of Australia)

At its Fourth Session in 1969 the Committee on Methods of Analysis and Sampling expressed the need for a systematic and comprehensive approach to the subject of sampling.

This view was reaffirmed at the Fifth Session of the MAS Committee in 1970. In accepting the Sampling Plans for Prepackaged Foods into the Codex Standards, the Committee expressed the view that these Sampling Plans will need to be revised on a more general basis. Furthermore, the Committee suggested that perhaps a consultant could be employed to look at the whole question of sampling. Then a special meeting of the CCMAS, including statisticians and specialists in food technology, could be convened to discuss the technical procedures of sampling in general. The Committee recommended that the Sampling Plans at that stage should be considered as a guideline only and not as a mandatory part of the Codex Standards.

At its Seventh Session (1970) the Codex Alimentarius Commission requested the Executive Committee to examine the whole question of sampling plans. The CCMAS at its Sixth Session, strongly supported the above move and agreed to wait for the recommendation of the Executive Committee. Meanwhile, the CCMAS would postpone any decision on endorsement of methods of sampling.

The Commission at its Eighth Session in 1971 accepted the following recommendations from the Executive Committee:

1. International agreement on sampling plans was important but immediate attention was unnecessary;
2. the "Sampling Plans for Prepackaged Foods" should be published;
3. Commodity Committees should: (1) choose their own sampling plan from those available (including the above);
   (2) select their own AQL (acceptable Quality Level);
4. there was no need to employ a consultant;
5. a special meeting of the CCMAS need not be called until after the International Standards Organization (ISO) had finalized their working documents relating to sampling. Meanwhile Commodity Committees should use existing sampling plans or any other sampling plans which might be appropriate.

At the Seventh Session of the CCMAS in 1972 the Committee decided to extend the General Principles for the Establishment of Methods of Analysis so that sampling provisions could be developed as referee methods in order to cover international disputes. However, the Committee undertook to develop guidelines on disputes. The UK delegation was requested to prepare the first draft in consultation with several other countries. However, the Committee recognized that by developing sampling provisions only for the international dispute situation, it would unduly limit the scope of its work, since the purpose of the Codex Alimentarius is to facilitate trade and protect the interests of consumers. It was, therefore, desirable to take steps to prevent disputes from occuring 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 a first step towards 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 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 standards.

The draft of General Principles for the Establishment of Codex Methods of Sampling had been sent to countries for comment, but no comments were received by the 8th Session of the CCMAS. The Committee, therefore, agreed to defer the matter. Furthermore, the Committee wished to delay consideration of the General Principles until the findings were known. The Secretariat suggested that the concept of Codex methods of sampling would probably have to be reconsidered at some future date. The Committee requested the Secretariat to prepare a paper in collaboration with the UK delegation setting out all previous decisions and adopted texts relating to the definitions, general principles, acceptance etc. of Codex methods of analysis and sampling.

REPORT OF THE AD HOC WORKING GROUP ON ACCEPTANCE SAMPLING PLANS FOR THE DETERMINATION OF NET CONTENTS OF PREPACKAGED COMMODITIES

INTRODUCTION

1. After opening the 9th Session of the Codex Committee on Methods of Analysis and Sampling, held in Budapest, 27-31 October 1975, the Chairman suggested that the ad hoc Working Group which was charged with the responsibility for studying acceptance sampling plans for the determination of net contents during the 8th Session should be reconvened. The membership of the reconvened Working Group consisted of the following representatives:

CANADA	Mr. G.E. Anderson (Chairman)
DENMARK	Mr.P.F. Jensen
EEC	Mr. G. Vos
GERMANY, Fed.Rep. of	Mr. W. Trapp
HUNGARY	Mr. P. Kiss Mr. F. Örsi Mr. E. Zukál
NORWAY	Mr. A.O. Hougen
SWITZERLAND	Mr. H.U. Pfister Mr. P. Koch Mr. P. Kovaliv
USA	Mr. C.H. Brokaw Mr. B. Levy Mr. J.S. Winbush

PREAMBLE

2. The Codex paper (CX/MAS 75/3-Add.1) dated October 1975 which has been distributed at this session to the Codex representatives summarizes the replies received during 1974 from 10 countries in which they commented upon the 11 "points of agreement" outlined in the report of the 8th Session (see ALINORM 74/23).

3. In general it would appear that the responding countries were prepared, with certain reservations, to accept the "points of agreement". Consequently a further questionnaire was distributed to member countries in which more detailed answers were requested.

4. The Chairman of the ad hoc Working Group announced that he had received a total of 14 replies to the latest questionnaire which was circulated to the member countries of the Codex Alimentarius in July 1975.

5. These replies indicated that there exists a very considerable diversity in the enforcement of the laws of the various countries with respect to the control of the net content of packaged commodities.

6. Thus some countries had no formalized sampling plans at all while others had as many as four or five plans which were chosen in accordance with the requirements peculiar to certain classes of commodities or which were dependent upon whether or not the tests involved the destruction of the sample. Further, there appeared to be very limited agreement as to the parameters (e.g. percent defective) which would define an acceptable lot or as to the characteristics of the official sampling plans, such as AQL, producers' and consumers' risks, etc.

7. In spite of the diversity of viewpoints of the various countries with respect to existing sampling plans, one fact appeared to be clear - and that is that nearly all countries appeared to be willing to consider the adoption of an acceptance sampling plan which would meet the wishes of the majaority of countries. It should of course be noted that several countries expressed the hope that the sampling plan finally agreed upon would meet certain criteria.

8. In view of this apparent consensus as to the desirability of adopting a uniform approach to the problem of the sampling of prepackaged commodities, the Working Group continued to study various sampling plans and to explore the possibility of proposing a plan which might meet with general acceptance or which would at least meet with a minimum of objection.

PROCEEDINGS

9. The ad hoc Working Group reported at the Budapest meeting, September 3-7, 1973, that there appeared to be three types of plans for lot acceptance which had been proposed by various member countries:

(a) those which aim to give maximum protection to the consumer (the so-called "minimum" procedure)

(b) those which stress the need to prove beyond reasonable doubt that a lot is in violation before enforcement action is taken; and

(c) those plans which propose to adopt an intermediate position by equating the producers' and consumers' risks at 50% for a lot whose lot average was equal to the declared weight.

10. The replies received to the July 1975 questionnaire indicated the fact that the EEC member states have already adopted two directives related to the making up by volume of certain prepackaged liquids and to bottles used as measuring containers. The procedures in these directives use the average as a major criterion for acceptance. Similar procedures for the checking of net weight and other liquid volumes are under discussion among the member states. To the best of our knowledge, therefore, there are no countries remaining which will continue to insist on each package containing at a minimum the declared (label) net contents.

11. Thus, essentially there remain for consideration only two basic plans, (b) and (c) above, and these are the plans which have been the subject of continued study by the ad hoc Working Group during this 9th Session.

COMMENTS BY THE WORKING GROUP

12. The plans advocated by Canada, USA and Australia have been referred to as 50-50 plans or, Indifference Quality Level (IQL) plans. It is usually and correctly stated that such plans have equal consumers' and producers' risks when the lot average is at the declared value. This statement masks the fact that the practical consequences of those risks, in monetary or operational terms may not be equal. It should be pointed out that larger filling tolerances or increased acceptance probabilities theoretically result in lower operational costs which potentially could be passed on to consumers. On the other hand, tighter filling tolerances or decreased acceptance probabilities result in substantial economic or legal burdens falling on the producer.

13. In practice the IQL plan requires systematic over-filling. In addition the extent of over-fill may differ widely from producer to producer as a result of differences in equipment or procedures. A second practical consequence will be the detection and virtual elimination of lots in commerce which average below declared value.

14. The systematic over-filling required by the IQL plans tends to be quite appealing to consumers. However, they may not be consistent with legal and enforcement practices in many countries.

15. The plans advocated by the EEC and the Swiss can be termed High Acceptance Probability (HAP) plans. The name stems from the property that lots averaging at the declared value will have a high (95% or above) probability of being accepted. The plans provide for some latitude for modification in terms of the associated risk which may be deemed acceptable by a country, while giving a high degree of assurance that lots accepted in one country would be accepted in another.

16. Also, the EEC presently has adopted or probably will adopt such plans for virtually all commodities. Further, they are consistent with the traditional legal and enforcement procedures in most of the countries concerned.

17. A consequence of the high probability of acceptance of lots averaging at the declared value, is the high probability of acceptance of lots averaging slightly below the declared value. This means that a producer could target slightly below the declared value with only a slightly higher risk of rejection of the lot.

18. The Working Group recognizes the strengths and weaknesses of both of these approaches. In accordance with the objective of fostering international trade, the Group is seeking a single plan which can gain general acceptance. To this end the Group proposes to construct a plan which minimizes the negative effects of each and retains most of their merits. The plan being considered might be termed a "Moderate Acceptance Probability Plan".

PROPOSED "MODERATE ACCEPTANCE PLAN"

19. Under the conditions of the proposed plan, a lot averaging at the declared value would be accepted into commerce 83% of the time. This percentage was proposed for mathematical convenience over other values in this vicinity. The property of the IQL plans which eliminated under-filling, on the average, is expected to be retained virtually intact. The amount of variability between producers allowed by the IQL plan will be greatly diminished.

20. The plan will be more sensitive to modification of sampling particulars in various countries than the HAP plans. The variability of sampling practices allowable under this plan is expected to be within tolerable limits. The use of the proposed plan in itself may not provide sufficiently strong evidence of non-conformity to the declared net contents to satisfy "beyond all reasonable doubt" the legal requirements of certain countries. However, such countries may choose to reapply the procedure sequentially to questioned lots, thus increasing the level of confidence in any decision finally reached.

21. The construction of this plan is not complete. The operating characteristic of the suggested plan is specified at one point only; that is, at the declared value. Its shape depends on several factors, such as allowable tolerances, sample sizes, economics of sampling, variable vs. attribute schemes, etc., which have not yet been investigated adequately. Once those factors have been adequately studied it will be possible for each nation subscribing to the use of this plan to specialize its sampling procedures to its own needs while remaining in conformity with the essential elements of the plan.

SUPPLEMENTARY COMMENTS

22. A subject of interest to the Working Group but not directly related to the main thrust of its endeavours was raised by the delegation of Switzerland. They suggested that the effect on consumers be quantified in terms of an average over several purchases, rather than in terms of a single unit. This seems to have merit on the basis of the presentation and would be interpretable in terms meaningful to the consumer.

23. Such an approach would appear to eliminate the necessity of modifying procedures because of differently shaped distributions. This is an important mathematical simplification and certainly merits further study.

RECOMMENDATIONS

24. It is recognized that the major questions raised in adopting one plan or another are essentially those of a social and political nature. Once the social and political decisions are made, the technical implementation is felt to be within the scope and capability of the Working Group. The Working Group recommends, therefore, that the Committee agree, in principle, with the desirability of studying the newly proposed approach to a plan and instruct the Working Group to develop the details more fully. Once developed, it is further suggested that the plan should be forwarded to member countries for consideration according to established protocol.

GENERAL REFEREE METHOD FOR DETERMINATION OF CHLORIDES (CALCULATED AS SODIUM CHLORIDE) IN FOODS

1. Principle of method

Product is dispersed with H_2O and acidified; soluble chlorides are titrated potentiometrically with $AgNO_3$. Applicable to levels $\geq$ 0.03% NaCl. For convenience in calculations, weights or volumes and normality are specified so that 1 ml $AgNO_3$ = 0.1% NaCl. If balance permitting rapid weighing of specified weight is not available, convenient weight sample and normality $AgNO_3$ solution may be used.

2. Apparatus

2.1 Balance: Capacity, $\geq$ 200g, taring range, $\geq$ 100 g, readability, $\leq$ 0.01 g. Mettler No. P1200 (Mettler Instrument Corp., PO Box 100, Princeton, NJ 08540) or equivalent is convenient.

2.2 Electrodes: Ag billet combination electrode (Beckman No. 39187, or equivalent), or separate indicating Ag (Beckman 39604, Orion 94-17, Fisher 13-639-122, or equivalent), and glass reference (Beckman 40455, Orion 90-02, Fisher 9-313-216, or equivalent) electrodes. Before initial use and before each day's use, if necessary, clean Ag billet electrode tip with scouring powder or other suitable material and rinse thoroughly with H_2O. (Hot H_2O may be required with some kinds of samples.) Clean other electrodes as recommended by manufacturer. Reclean as frequently as necessary to prevent drifting of end point reading. With some samples, periodically rinse electrodes with H_2O and wipe with tissue to prevent accumulation of film. It is unnecessary to coat Ag billet electrodes with AgCl.

2.3 Magnetic stirrer: Operating through variable transformer to permit range of speed which, once set, is constant.

2.4 pH meter: Preferably direct reading, with scale divisions 10 mv or less; range at least ± 700 mv, e.g., digital type (Orion Model 701, or equivalent).

3. Reagents

3.1 Nitric acid, dilute: (1+49). Dilute 20 ml HNO_3 to 1 L with H_2O.

3.2 Silver nitrate standard solution: 0.0856N. Dissolve 14.541 g $AgNO_3$ in H_2O and dilute to 1 L in volume flask. Standardize as in 4, and adjust to exact normality specified so that with indicated sample weight 1 ml = 0.1% NaCl. Store in Pyrex container out of direct sunlight. The solution is stable in room light.

3.3 Sodium chloride standard solution: 0.0856N. Dissolve in H_2O 5.000 g NaCl (if assay is < 100.0% NaCl, divide 5.000 g by % NaCl/100 to obtain corrected weight), previously dried 2 hours at 110°, and dilute to 1 L in volumetric flask.

3.4 Water: Distilled or deionized, halogen-free by following test: Add 1 ml ca 0.1N $AgNO_3$ and 5 ml HNO_3 (1 + 4) to 100 ml of the H_2O. No more than slight turbidity is produced.

4. Standardization

Pipette 25 ml NaCl standard solution into 250 ml beaker, dilute to ca 50 ml with H_2O, and add 50 ml HNO_3 (1 + 49). Insert electrodes, start magnetic stirrer, and stir throughout titration at constant rate producing vigorous agitation without splashing. Titrate with $AgNO_3$ standard solution, adjusting increments with rate of voltage change so that accurate plot of mv against ml Ag NO_3 solution can be prepared. Add total of 50 ml $AgNO_3$ solution to obtain complete curve.

Determine inflection point by drawing two straight lines with 45° slope with respect to axes and tangent to titration curve at the two points of greatest curvature. Inflection point is at intersection of titration curve with line drawn parallel to and midway between other two lines. From volume $AgNO_3$ solution used, calculate normality and adjust to 0.0856N. Re-standardize occasionally. Use inflection point as end point in titrating samples. Re-check end point potential occasionally, and re-determine when

either individual electrode, combination electrode, or pH meter is replaced by preparing new titration curve.

For greatest accuracy, when series of determinations on same food is performed, determine and use end point from titration curve of that food rather than using end point obtained with NaCl standard solution.

5. Preparation of Sample

5.1 Clear liquids with low viscosity: (Fruit juices, clear soups, wines, etc.) Use directly.

5.2 Comminuted products: (Tomato juice, tomato catsup, strained vegetables, etc.) Thoroughly shake unopened container to incorporate any sediment. Transfer entire contents to large glass or porcelain dish and mix thoroughly, continuing stirring at least 1 minute. Transfer to glass-stoppered container, and shake or stir thoroughly each time before removing portions for analysis.

5.3 General method for heterogeneous (fish, meat, etc.), low moisture (cereal products, etc.) and hard-to-disperse, homogeneous (cheese, peanut butter, etc.) foods: Weigh 50.0 g sample into 1 L (quart) container of high-speed blender and add 450 g H_2O. Cover, start blender at low speed by use of variable transformer for initial dispersion, and blend thoroughly at high speed (1-2 minutes is usually adequate). Equivalent of 5 g sample is conveniently dispensed through 50 ml pipette with tip cut off. Thoroughly mix sample suspension immediately before pipetting aliquot for analysis so that solid material is uniformly suspended.

5.4 Other types of foods: Prepare sample by method 5.1, 5.2 or 5.3 or other suitable method.

To preserve samples or sample suspensions for future analysis, add 0.5 ml ca 37% HCHO solution/100 g sample or sample suspension, mix well, and store at room temperature. Correct for dilution by HCHO solution by multiplying % NaCl by 1.005.

6. Determination

6.1 For products containing less than 5 per cent salt: Place 5.00 g (or 5.00 ml if concentration is to be expressed on w/v basis) sample from 5.1 or 5.2 or 50.0 g from 5.3 into tared 250 ml beaker; add H_2O to ca 50 ml if 5.1 or 5.2 is used. (Use boiling H_2O with samples such as butter to melt fat.) Add 50 ml HNO_3 (1 + 49). Titrate as in 4, using 10 ml burette if salt content is $\leq$ 1%.

% NaCl = ml 0.0856N $AgNO_3$/10.

6.2 For products containing 5 or more per cent salt: Place 5.00 g (or 5.00 ml if concentration is to be expressed on w/v basis) sample from 5.1 or 5.2 into 100 ml volumetric flask and dilute to volume with H_2O. Mix, and transfer aliquot containing 50-250 mg NaCl to 250 ml beaker. If sample is prepared by 5.3, transfer weighed aliquot containing 50-250 mg NaCl to tared 250 ml beaker. Proceed as in 4, beginning " ... dilute to ca 50 ml with H_2O..."

% NaCl = F x ml 0.0856N $AgNO_3$/10, where F = dilution factor = 100/ml aliquot titrated if sample is prepared by 5.1 or 5.2 or 50/g aliquot titrated if prepared by 5.3.

6.3 General case: Accurately weight approximately sample weight stated. (If % NaCl $\geq$ 5%, weigh $<$ 5g sample rather than diluting to 100 ml, if more convenient.) Use ca 0.1N $AgNO_3$ solution, accurately standardized as in 4, without adjusting to specific normality, and titrate as in 4.

% NaCl = ml $AgNO_3$ x N $AgNO_3$
x 0.05844 x 100/g sample.

If sample is overtitrated, add NaCl standard solution, and complete titration. Correct for volume of standard solution added.