

**codex alimentarius commission**

FOOD AND AGRICULTURE  
ORGANIZATION  
OF THE UNITED NATIONS

WORLD HEALTH  
ORGANIZATION

JOINT OFFICE: Via delle Terme di Caracalla 00100 ROME: Tel. 57971 Telex: 610181  
FAO I. Cables Foodagri

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ALINORM 81/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Fourteenth Session, Geneva, 29 June-10 July 1981

REPORT OF THE TWELFTH SESSION OF THE  
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING  
Budapest, 11-15 May 1981

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Twelfth Session from 11 to 15 May 1981 in Budapest, by courtesy of the Government of Hungary. The Session was opened by Dr. K. Sütö, President of the Hungarian National Codex Committee and Vice-President of the Hungarian Office for Standardization, who welcomed the participants. Dr. Sütö stressed the importance of work on standardization of foods and, within this field, work on standard methods of analysis and sampling. He expressed the hope that the Committee would have a successful meeting and would reach a consensus and make appropriate recommendations, especially in the field of sampling.
2. The Committee was chaired by Professor Dr. R. Lásztity, University Professor, Technical University, Budapest.
3. The Session was attended by delegates from 21 countries and observers from 6 International Organizations. The list of participants, including officers from FAO, is attached as Appendix I to this Report.

APPOINTMENT OF RAPPORTEURS

4. The Committee agreed not to appoint rapporteurs for the preparation of the draft report. However, Ms. J. Castang and Ms. C. Soules of the delegation of France assisted in the preparation of the French version of the report. The Committee expressed its appreciation for this assistance.

ADOPTION OF AGENDA

5. The Committee adopted the Provisional Agenda without rearrangement of the order of items but noted that item 4.5 would not be discussed as the working document on the Sampling Plan for the Determination of Net Weight in relation to lots was not available.

MATTERS OF INTEREST ARISING FROM CODEX REPORTS

6. The Committee received a verbal report from the Secretariat on matters of interest arising from Codex reports. Although no working paper was available for this item on the Agenda, the Committee was provided with a Room Document No.2 in English only, in order to assist the discussions.

#### Codex Committee on Fats and Oils (CCFO)

7. The Committee noted that the Codex Committee on Fats and Oils was considering an IUPAC method for the determination of beta-sitosterol in total sterols of vegetable fats. The delegation of the United States of America stressed the need for the CCFO to supply appropriate information especially relating to collaborative studies by IUPAC to this Committee, so that the suitability of the proposed methods can be ascertained. IUPAC was urged to publish the results of its collaborative studies.

#### Codex Committee on Pesticide Residues (CCPR)

8. The Committee noted that, in the opinion of the CCPR, methods of residues analysis recommended by that Committee were of Type III or Type IV. In the opinion of the Working Group on Analysis of the CCPR, the Codex criteria for the selection of methods of analysis did not place sufficient emphasis on confirmatory tests. It was agreed that this matter be considered at a future session.

#### Codex Committee on Processed Fruits and Vegetables (CCPFV)

9. The Committee was informed that the above Commodity Committee would attempt to resolve an inconsistency in approach to the determination of moisture content in un-shelled pistachio nuts. This inconsistency resulted from the fact that three defining methods (Type I) were under consideration for the same parameter in the same food. The Committee agreed that this matter be considered at a future session.

#### Codex Committee on Foods for Special Dietary Uses (CCFSDU)

10. The Committee noted the view of the Working Group of the CCFSDU that "Defining methods" should first be elaborated before figures before the respective criteria are laid down in Codex standards. The delegation of the Federal Republic of Germany was of the opinion that defining methods and the relevant criteria should be considered at the same time in order to avoid situations arising such as that seen in relation to the determination of moisture content indicated in para 9 above.

11. As regards crude fibre in infant formula, the Committee noted the view of the CCFSDU that the determination of this criterion was not necessary in relation to the determination of carbohydrates. The delegation of Spain was of the opinion that from a nutritional point of view, the determination of crude fibre in infant food is important and that a method should be developed.

#### Codex Committee on Vegetable Proteins (CCVP)

12. The delegation of Spain pointed to the need for appropriate methodology for the characterization and estimation of individual proteins. The representative of ISO informed the Committee that ISO is developing methodology for the determination of vegetable proteins in meat products.

#### Codex Committee on General Principles (CCGP)

13. The Committee was informed that the Codex Committee on General Principles had considered the question (a) whether it is appropriate to establish more than one method of analysis for each parameter in a standard; and (b) whether it is appropriate to establish methods of analysis for parameters which are not in a standard. The Committee noted the views of the CCGP and agreed that they corresponded to present procedures relating to Codex methods, at least, on the basis of the way in which the above questions were put to the CCGP. The Committee noted, however, that the issue raised in question (b) above had arisen from work on natural mineral waters.

## Other Matters

14. Analysis of Food Additives in Foods - The Committee noted that the delegation of Egypt, at the Thirteenth Session of the Commission, had expressed the view that methods of analysis should be elaborated for the determination of additives in food. The Secretariat pointed out that this field had indeed been somewhat neglected in the past and also that more work on the determination of contaminants should be undertaken. The delegation of the United States of America indicated that AOAC methods for the determination of aflatoxins and other mycotoxins existed and that these had been endorsed by several International Organizations. These methods could be made available to the Committee. The Committee noted the Codex procedures for the elaboration of specific or general methods for the determination of food additives in foods. It was agreed that Codex Commodity Committees, the Codex Committee on Food Additives and the CCMAS itself, should give this problem due consideration in the future.

15. Determination of Lard in Foods - The representative of ASMO pointed to the religious and social reasons for which the consumption of this animal fat in foods is prohibited. Thus, for the mutual interest of the producer and purchaser in an Islamic country, it is necessary to develop a quantitative and/or qualitative method for the determination of any amount of lard in food. The representative of ISO indicated that ISO had studied this problem but that no reliable method could yet be developed.

## DEFINITION AND CLASSIFICATION OF CODEX METHODS OF SAMPLING AND CRITERIA FOR THEIR SELECTION

16. The Committee had before it a paper prepared by the Codex Secretariat (CX/MAS 81/2) and a paper prepared by the United Kingdom (CX/MAS 81/3). Paper CX/MAS 81/2 was prepared by the Secretariat as no report from the inter-session Working Group on Sampling, set up during the last session of the CCMAS, was available. In introducing the paper, the Secretariat drew attention to the following points:

- (a) To what extent is international harmonization of sampling procedures desirable or feasible;
- (b) Should Codex deal with sampling procedures only in relation to consignments of food moving in international trade;
- (c) Is the purpose of Codex sampling methods to specify the conditions under which a consignment of food may be considered to be in conformity with the relevant standard or legal provisions;
- (d) Should Codex draw up advisory practical sampling procedures which are less rigorously statistically based but still acceptable or should Codex draw up statistically based 'reference' or 'referee' sampling procedures with high confidence levels to be resorted to in cases of dispute.

17. The Secretariat paper also indicated the sort of work the Committee might wish to undertake in order to achieve the aims of the Codex.

18. During the discussions, the delegation of the United Kingdom pointed out that sampling consisted basically of two components: (a) the physical procedures for taking samples; and (b) the statistical aspects, including interpretation of results of analysis in relation to lots or consignments. This latter aspect was considered to be the more difficult one to resolve and which often involved non-statistical considerations.

19. The delegation of the United States of America posed the question of principle as to whether the determination of compliance should be related to the lot or the sample. In

this connection, the system adopted by the CCPR was noted in which compliance is determined on the analytical sample prepared from the sample drawn from a consignment in accordance with a practical sampling scheme. The United States delegation raised the question whether this sort of legal approach could lead to a situation where an acceptable consignment could be declared not to be in conformity with the relevant legal requirements on the basis of a non-complying sample which may not be representative of the consignment. This sort of approach, especially if the manufacturer of the food were denied the right of challenging the results of analysis, ignored the principle that the greater the size of the sample the more reliable is the decision reached in relation to a consignment or lot.

20. The point was made by the delegation of the Federal Republic of Germany that the task of Codex was to assist Governments in making decisions in relation to consignments or lots. The fact should be accepted that decisions based on sampling cannot be made with an absolute guarantee of making correct decisions and that, in any event, more than one sampling plan may be required for checking compliance. The delegation suggested that information should be collected from Governments in order to ascertain how sampling for food control purposes is carried out in the various countries. In this respect, the delegation of Hungary pointed out that some information was already available on this matter (see para 36 of this Report).

21. The Committee proceeded to discuss the types of sampling procedures required by the Codex. Several delegations put forward suggestions for classes of sampling plans covering different aspects such as microbiological criteria, net weight and compositional criteria. The delegation of Hungary suggested sampling plans should be drawn up to cover the following criteria: those relating to health (microbiological criteria, toxic components, etc.); nutritional and other similar ingredients; criteria related to storage; criteria relating to weights and measures, etc.

22. Considering the number of parameters which have to be considered in drawing up appropriate sampling plans, it was agreed that several classes of sampling plans would have to be elaborated. In selecting a given sampling plan from among a number of available sampling plans, that plan should be used which would provide a sample size sufficient to cover the criteria which would be subject to checking for compliance.

23. The Committee then considered the document prepared by the United Kingdom (CX/MAS 81/3) which also dealt with the definition and classification of Codex methods of sampling, criteria for their establishment as well as with other questions. This document had been drafted as a proposed addition to the "General Principles for the Establishment of Codex Methods of Analysis and Sampling" given in Appendix II to ALINORM 79/23.

24. Following discussion of the UK paper, the Committee decided to set up a Working Group consisting of the countries listed below, to consider the remarks made during the Session. The following countries and International Organizations took part in the deliberations of the Working Group: Denmark, France, Hungary, Switzerland, United Kingdom (Dr. Wood, Chairman), United States of America, EEC and ISO.

25. The amended version of paras 3 and 4 to the above "General Principles" proposed by the Working Group and adopted by the Committee is given as Appendix II to this Report. It was agreed that Governments be requested to comment on Appendix II and that it be reconsidered in the light of comments with a view to finalizing paras 3 and 4 of the 'General Principles' relating to sampling at the next Session. The Commission should be informed of this development.

## DEVELOPMENT OF VOCABULARY USED IN SAMPLING

26. The Committee had before it document CX/MAS 81/4, prepared by the Hungarian Secretariat, containing definitions of terms used in sampling.

27. The Committee noted that International Organizations, such as ISO, EOQC and ASQC were active in the development of vocabulary used in sampling. The representative of ISO indicated that ISO had already available on this subject a standard (ISO 3534, Statistical Vocabulary and Symbols) and a draft Standard (ISO/DIS 7002, Agricultural Food Products - Layout for a standard method of sampling from a lot). The latter draft standard, which included a vocabulary of the most commonly used terms in sampling, is the more relevant text for purposes of the Codex. It had been sent to Members of ISO/ TC 34 and Associate Members of ISO for comments and is now available for public comment.

28. The Committee agreed that, in order to avoid duplication of effort, Codex should not proceed with the development of a vocabulary of its own. The Committee, therefore, agreed that the Codex Secretariat should request comments on the draft standard ISO/DIS 7002 and that these comments should be sent to ISO as an input from Codex Members and interested International Organizations in the finalization of the document. The Codex Secretariat should also extract terms which are used in Codex Standards and which are not present in ISO/DIS 7002 and refer them to ISO for possible inclusion in the revised document.

29. The representative of ISO informed the Committee that the end of September 1981 was the last date for receiving comments on the draft standard and requested that every effort should be made by the Codex in order to ensure that their comments should reach ISO before that date.

30. The Committee expressed its appreciation to ISO for undertaking the task of considering any input generated through the Codex channels, and agreed that, once developed, the ISO vocabulary used in sampling should be considered by the Committee. The present arrangements did not mean an priori endorsement of the terms used in the sampling of foodstuffs adopted by ISO.

## GUIDELINES FOR ADMINISTRATIVE ASPECTS OF SAMPLE TAKING

31. The Committee had before it a document prepared by Hungary on the above subject matter (CX/MAS 81/5). The document was introduced by the delegation of Hungary which pointed out that the document consisted of two parts: the first part, giving technical and other details on how samples were actually taken and the second part, which dealt with questions of principle and approach taken in various countries in connection with sampling.

32. During the discussion of the Hungarian paper, the Committee was informed that similar work was in progress within the EEC and ISO. A part of the Hungarian paper had been based on work by ISO, IDF and AOAC. A number of delegations were of the opinion that it would be appropriate to proceed with the development of the technical and administrative guidelines for sampling in the same way as was decided in connection with the development of sampling vocabulary (see paras 26-30 of this Report).

33. The Committee agreed to proceed in the manner suggested in para 32 above, and requested the Secretariat to ensure that ISO would receive appropriate input from Members of the Codex and from interested International Organizations. (Reference to ISO Document: )

34. As regards the second part of the Hungarian paper dealing with questions of principle and approach relating to sampling, it was agreed that this represented useful information for future reference when considering details of sampling plans and procedures. In addition, the Hungarian paper might also be of interest to ISO.

35. The Committee expressed its appreciation to ISO for undertaking the task of considering comments from Codex Members and interested International Organizations and agreed that the future ISO Standard which would eventually be adopted should be considered by the Committee. As in the case of the vocabulary used in sampling, the present arrangements with ISO did not represent an a priori endorsement of that standard.

36. The Committee agreed that, regarding the issues raised in the second part of CX/MAS 81/5, information on sampling and enforcement procedures for foodstuffs should be obtained by circulating an appropriate questionnaire to Codex Contact Points, the questionnaire being based on the questionnaire currently being prepared within the EEC (see also para 20 of this Report).

#### SAMPLING PLAN FOR THE DETERMINANT ON OF NET CONTENTS

37. As no document was available on this subject, the Committee decided to deal with the question of net contents only from a point of view of what further action should be undertaken (see para 5 of this Report). There was general agreement that it was necessary for Codex to develop sampling plans for net contents. The Committee decided to set up an ad hoc Working Group on Sampling for Net Contents to discuss the question of sampling in relation to net content. The report of the Working Group is given as Appendix III to this Report.

38. The Committee received a verbal report from the Chairman of the Working Group, Dr. W. Dubbert (USA). He indicated that the Working Group had discussed the subject of sampling plans for the determination of a net content in detail and had also discussed other more general issues relating to sampling. The Group had reached conclusions on how work should proceed on sampling for net content and had also reached some conclusions concerning sampling in general.

39. The Committee was not in a position to discuss the report of the Working Group as it had not been possible to prepare it in time for distribution during the Session. However, the Committee agreed that the report be appended to the report of the present Session, and that a questionnaire on sampling for net content be distributed by the Codex Secretariat to Codex Contact Points and other interested parties as suggested by the Working Group.

40. The Committee agreed that the Working Group on Sampling for Net Content should continue its work and report to the next Session of the Committee. Dr. W. Dubbert (USA), agreed to act as contact point for the Working Group.

#### REVIEW OF CODEX METHODS OF ANALYSIS

41. The Committee had before it document CX/MAS 81/7, prepared by Australia listing all methods of analysis contained in Codex draft standards pending adoption by the Commission and document CX/MAS 81/7-Add.1, also prepared by Australia, containing recommendations on how methods of analysis might be reviewed. In introducing the paper, the delegation of Australia expressed the opinion that it seemed appropriate for the Committee to initiate a full review of all Codex methods in the light of new developments and in the light of changes in the classification and definition of

Codex methods. The initial responsibility for such a review, in the opinion of Australia, rests with the responsible Codex Commodity Committees. In order to facilitate this work, the CCMAS could develop an overall policy and prepare guidelines for Commodity Committees.

42. The Committee was informed that, in fact, the Codex Committees on Fats and Oils and Sugars had already initiated a review of methods recommended by them. The representative of ISO pointed to the use by some Codex Committees of double references to existing identical official methods and expressed the view that such a practice should be encouraged.

43. The delegation of Australia pointed to the Codex principle that methods selected need not be the most sophisticated available. On the other hand, it appeared that for some years Codex methodology has not kept up with developments in the field of analysis. In reviewing Codex methods, this should be kept in mind. In this connection, the suggestion was made that, rather than avoiding sophisticated methods as Codex reference methods, countries where difficulties existed in the adoption of such methods might be given appropriate assistance through the UN system, in order to improve their analytical capabilities.

44. The delegation of France indicated that, in response to a request by the Coordinating Committee for Europe, it had assembled information on the basis of which methods of analysis could be recommended for natural mineral waters. The Committee recalled the views of the Codex Committee on General Principles (see para 13 of this Report) and noted the opinion of the Secretariat that the Regional European Standard for Natural Mineral Waters contained provisions which required methods of analysis not only for their verification but also for their definition. The delegation of Norway was strongly against the inclusion of provisions in Codex standards which required analytical proof of the absence of ill-defined substances in foods. The Committee suggested that the delegation of France and other interested delegates may wish to organize an informal meeting during the Session to study the material available in preparation for the next Session of the Coordinating Committee for Europe.

45. On the suggestion of the delegations of Australia, United Kingdom, the Federal Republic of Germany and the United States of America, the Committee adopted the following statement which would assist Codex Commodity Committees in undertaking a review of the methods included in their commodity standards:

The Committee RECOMMENDS that:

- (a) BEFORE the next meeting of the Codex Committee on Methods of Analysis and Sampling (CCMAS) all Commodity Committees report on the necessary measures taken to update all analytical methods elaborated, recommended or endorsed by them, prior to 1 January 1979. Details of all supporting collaborative studies should be supplied. Methods endorsed after 1 January 1979 should be reported upon at a subsequent meeting of the CCMAS.
- (b) COMMODITY Committees should report on all methods where it is recommended that no update should occur. This would include instances where no further work on such methods was known or where an earlier or traditional method was considered adequate, and in these cases, the facts should be reported.
- (c) WHERE recommendations are for updating to a later edition of a printed volume, journal or series, the Commodity Committees should ensure that the

later reference is to the same method and is identical except for editorial revisions, otherwise supplementary collaborative studies should have been conducted. These studies should be reported.

- (d) COMMODITY Committees should also report on the availability of sampling plans necessary for the analysis of materials with which they are concerned. The Commodity Committees should also report where such plans do not exist, or where their use has not been considered necessary to date.

## GENERAL METHODS FOR THE DETERMINATION OF METALLIC CONTAMINANTS

46 The Committee had before it document CX/MAS 81/8 containing a status report prepared by the Canadian delegation on general methods for the determination of metallic contaminants and Room Document No. 4 prepared by The Netherlands on a rapid method for the determination of mercury in fish. The status of the methods, as agreed by the Committee, is summarized in Appendix IV to this Report.

### Mercury

47. The Committee noted that the Official Methods of Analysis of the AOAC (1980) for the determination of mercury, incorporated appropriate precautionary measures to minimise the possibility for explosion when using the teflon lined reaction vessel for the digestion of foodstuffs with nitric acid and agreed that it would be suitable as a Codex reference method (Type II) for the determination of mercury in fish and sea foods.

48. The Committee noted the satisfactory results obtained by the method of Pearce *et al.* (JAOAC, 1976, 59,655) for the rapid determination of mercury in fish (see Room Document No.4) and felt it desirable to develop such rapid methods where possible for the determination of the different trace elements even though such methods may not be adopted as Codex methods.

### Lead

49. The Committee was informed that the considerable variation observed in determination of very low levels of lead in foods was due, in part, to environmental contamination, avoidance of which needed great care, both in the preparation of reagents and in the layout of laboratories. Objections were raised by certain delegations to the acceptance of the closed system digestion-anodic stripping method, since it is a complicated method. The Committee noted that other methods like the graphite furnace method were also used for determination of lead in foods.

50. The Committee noted that, in spite of the drawbacks, the anodic stripping method gave fairly good results in collaborative studies carried out in the United States of America for determination of lead at lower concentrations (less than 1 mg/kg) and considered it suitable as an alternative approved method (Type III). This action of the Committee may be reviewed later, when better methods which have been collaboratively tested are available. The delegations of The Netherlands, Norway and the Federal Republic of Germany expressed their reservation concerning the suitability of this method.

51. The Official method of analysis of the AOAC (1975), or the equivalent AOAC (1980) was considered by the Committee to be suitable as a Codex reference method (Type II) for samples containing more than 1 mg/kg (1 ppm) of lead. It was pointed out that in some instances, it was possible to use this method to check compliance of food with maximum levels less than 1 mg/kg through concentration procedures.

### Arsenic

52. The Committee noted that the AOAC (1980) colorimetric method for determination of arsenic in foods gave satisfactory results and agreed that it would be suitable as a Codex Reference Method (Type II) until the AAS method could be further developed.

53. As regards the acceptability of the Hydride Generation-Atomic Absorption method as a Codex reference method (Type II) for the determination of arsenic in foods, as recommended in the Canadian paper (see para 46 of this Report), the delegation of The Netherlands indicated that the method did not yield equally good results when tested by different workers and that the method had the same drawbacks as the method mentioned under lead (para 49 of this Report). The delegation of the Federal Republic of Germany expressed similar reservations concerning the acceptance of the method. In contrast to the recommendation by Canada, the Committee agreed to consider it as an alternative approved method (Type III) until the matter raised by the above delegations was resolved.

#### Cadmium

54. The Committee was informed by the delegation of Hungary about the availability of a suitable polarographic method for the determination of cadmium in foods. It is expected that documentation on this method would be made available for consideration by the Committee at its next Session.

55. The Committee noted that a working party of the EEC had adopted an atomic absorption method similar to the AOAC method for the determination of lead and cadmium in meat products. This method had been collaboratively tested.

56. In contrast to the recommendation contained in the Canadian paper, the Committee agreed that the AOAC (1980) atomic absorption method for cadmium was suitable as a Codex reference method (Type II) and that the AOAC (1980) anodic stripping voltametry method could be used as an alternative approved method (Type III). The delegations of The Netherlands and the Federal Republic of Germany expressed their reservation concerning the latter method.

#### Copper

57. The Committee agreed that the AOAC (1980) atomic absorption method was suitable as a Codex reference method (Type II).

#### Zinc

58. The Committee agreed that the AOAC (1980) atomic absorption method was suitable as a Codex reference method (Type II) and the more recent closed system digestion atomic absorption method of the AOAC (1980) as an alternative approved method (Type III).

59. The delegation of The Netherlands expressed reservation concerning the closed system digestion atomic absorption method since the combined wet destruction ashing technique involved in the procedure posed difficulties.

#### Tin

60. The Committee agreed that the AAS method of the AOAC (1980) was suitable as a reference method (Type II) for determination of tin present in foods at levels of 10 mg/kg (10 ppm) or more. The Committee noted that the above method had not been before Governments at Step 3 of the Codex Procedure but, as there was unanimous agreement with the method and as this method was urgently required by Governments

and the Codex, it was unanimously agreed that it be referred to the Commission for adoption.

#### ENDORSEMENT OF METHODS OF ANALYSIS INCLUDED IN CODEX STANDARDS

61. The Committee had before it document CX/MAS 81/9 containing methods of analysis requiring endorsement. A summary of the methods endorsed or temporarily endorsed is given in Appendix V to this Report. The following paragraphs represent a summary of the salient points raised during the discussions. The Committee noted that difficulties experienced with standard methods should be communicated to the International Organization concerned in an appropriately documented form.

##### Contaminants in Foods

62. In view of the fact that the Committee had elaborated general methods based on appropriate information for the determination of a number of contaminants in food (see paras 46-60), it was decided that the endorsement of the methods for these contaminants included in the Codex draft standards before the Committee at the present Session, should be postponed. The general methods included in Appendix IV should be referred to the relevant Codex Commodity Committees for consideration and inclusion in their standards as appropriate.

##### Endorsement of Provisions in Commodity Standards

63. During the consideration of the various methods of analysis proposed by Codex Commodity Committees the following points were raised by delegations or by the Committee:

(a) Cocoa butter confectionery

i. Fat-free milk solids

The Committee was informed that the OICC/AOAC method could not be used for products which had been heat treated and difficulties were reported in using this method. Reservations were expressed by the delegations of The Netherlands and the Federal Republic of Germany concerning the OICC/AOAC method.

(b) Nectars of Certain Citrus Fruits, Pulpy Mango Nectar, Guava Nectar.

i. General

The delegation of Australia was of the opinion that all IFJU methods should be reviewed and that, in the meantime, IFJU methods should be only temporarily endorsed.

ii. Sugars

The delegation of the United Kingdom expressed the opinion that the same method should be used for sugar determination in fruit juices as for sugar products. The Committee questioned whether the IFJU method No.4 had been collaboratively tested and was informed that this was so and that results of the collaborative test would be made available by the Federal Republic of Germany. The Committee noted that the IFJU method measured total sugars rather than added sugars. The Commodity Committee was requested to clarify this matter. The delegation of the United States of America indicated that AOAC GLC methods, which were specific for a number of sugars, were available.

(c) Concentrated Pineapple Juice

- i. General  
See statement by Australia (para 63, (b)i.).
  - ii. Soluble solids  
The Committee considered that the IFJU method No.8 B should be commented upon by Governments and that IFJU should review this method in the light of comments.
  - iii. Acid  
The representative of ASMO questioned whether the title "Acid" was appropriate. It was agreed that the definition referred to total acidity in concentrated pineapple juice.
- (d) Guava Nectar
- i. General  
See statement by Australia (para 63, (b)i.).
  - ii. L-Ascorbic Acid  
The Committee was informed that collaborative studies were in progress by IFJU on an enzymatic method and that a direct titration method using iodine was available for use as a routine method,
- (e) Minarine
- i. Milk fat  
Some delegations thought that the existing Codex method CAC/RM 15-1969) which was an empirical method, was difficult to perform and did not give satisfactory results. There was a need to develop a more specific GLC method. It was noted that such a procedure was being considered by EEC for cocoa products. Other delegations expressed the opinion that GLC methods based on butyric acid, while more reliable, were equally difficult to interpret in relation to milk fat content.
  - ii. Sodium chloride  
The Committee agreed to refer Codex method CAC/RM 19/1969 to the Commodity Committee with a request that it should consider the newly adopted general Codex potentiometric method for chloride determination.
- (f) Bouillons and Consommés
- i. Creatinine, total nitrogen, amino nitrogen  
In view of the fact that results of collaborative studies for the relevant AIIBP methods had not been published and were, therefore, not available to the Committee, it was agreed to give only temporary endorsement to the AIIBP methods concerned.

#### Review of Methods of Analysis in Standards for Fat3 and Oils at Step 9

64. The Committee noted, with satisfaction, that the Codex Committee on Fats and Oils had started a review of the methods of analysis it had previously recommended for fats and oils. It also noted that many of the methods being proposed were IUPAC methods and that IUPAC did not publish the results of its collaborative studies. It was

considered that this would pose difficulties in accepting these methods as Codex methods. It was noted that the results of the review by the CCFO would be submitted to this Committee for endorsement.

#### PLANNING AND STATISTICAL EVALUATION OF COLLABORATIVE STUDIES

65. The Committee had before it document CX/MAS 81/10 prepared by the Federal Republic of Germany. Noting that the Inter-Agency Meeting, held in Budapest from 8-9 May 1981, had also discussed the question of the harmonization of collaborative testing, the Committee agreed to discuss the above paper when considering the report of the Inter-Agency Meeting (see para 66 of this Report).

#### CONSIDERATION OF THE REPORT OF THE THIRD INTER-AGENCY MEETING ON METHODS OF ANALYSIS

66. The Committee had before it a Report of the above meeting (Room Document No.1). The report was introduced by the representative of ISO who informed the Committee concerning the deliberations of the Inter-Agency Meeting. The Meeting had received reports by the various International Organizations on progress made in the development of methods for cocoa products, edible ices, fats and oils, foods for special dietary uses, fruit juices, processed fruits and vegetables, processed meat products, sugars, starch hydrolysis products and cereal products. The Meeting had noted that good cooperation existed among the interested International Organizations as regards the development of methods required by Codex. In order to facilitate this work, ISO had presented an inventory of methods so far elaborated by that Organization. This inventory would be up-dated and made available to interested persons and to the participants of the Fourteenth Session of the Codex Alimentarius Commission. Other Organizations had been invited to prepare similar inventories. The Meeting had also discussed the need for standardizing terminology used in analysis and sampling and the need for harmonizing the approach to international collaborative tests. It had also agreed that there was a need to continue holding sessions of the Inter-Agency Meeting prior to sessions of the CCMAS.

67. As regards the question of harmonization of collaborative testing, the delegation of the Federal Republic of Germany indicated that document CX/MAS 81/10 did not cover all aspects relating to the organization of collaborative testing and the statistical evaluation of results. Other aspects could be added to a document eventually prepared on this subject. The delegation of Spain read out a detailed statement concerning collaborative testing in relation to standard ISO 5725 and the document CX/MAS 81/10 and expressed the view that there was a need for IUPAC to give attention to the harmonization of collaborative studies. The delegation of Spain was requested to make its comments available in writing to interested parties and the Codex Secretariat. The delegation of the United States of America stressed the need to harmonize the approach to collaborative tests since such tests were expensive and called for international collaboration as well as a common approach to the interpretation of results. One of the problems in interpreting the results from collaborative tests was the significance of outliers, especially at the  $\mu\text{g}/\text{kg}$  level. The delegation of the United States of America expressed the hope that IUPAC would undertake the task of harmonizing collaborative tests.

68. The Committee agreed that it was important that the procedures for carrying out collaborative tests and for the interpretation of results be discussed internationally with a view to the elaboration of an internationally acceptable protocol of collaborative testing. It decided that the outcome of the IUPAC conference should be placed before the next

session of the Inter-Agency Meeting and before the Codex Committee on Methods of Analysis and Sampling.

#### OTHER BUSINESS

69. The representative of the EEC introduced a report on the approximation of standard methods of analysis for foodstuffs within the European Economic Community (Conference Room Document No.5). The document gave an outline of methods for a number of classes of food commodities indicating the principle of the methods concerned. The Committee noted that a number of the methods were in line with those recommended by Codex. The Committee expressed its appreciation to the representative of EEC for this informative document and also noted that the EEC would, as in the past, be invited to Inter-Agency Meetings.

#### DATE AND PLACE OF THE NEXT SESSION

70. The Committee was informed that the proposed date and place for the Thirteenth Session of the Codex Committee on Methods of Analysis and Sampling is 6-10 December 1982 in Budapest. This proposed date would be considered by the Fourteenth Session of the Codex Alimentarius Commission. The opinion was expressed that it might be more practical to hold the next Session of the CCMAS early in 1983 so that more time would be available for the preparation of working documents. In this regard, the timely distribution of adequate documentation was stressed by many delegations.

71. Regarding the place for the Session of the CCMAS, the Chairman informed the Committee that the Government of Hungary would, in principle, be in agreement to hold a Session of the CCMAS in a developing country should such a country make concrete proposals regarding the hosting of a Session of the Committee. It was noted that this matter would be pursued further between the Government of Hungary and the Codex Secretariat.

LIST OF PARTICIPANTS  
LISTE DES PARTICIPANTS  
LISTA DE PARTICIPANTES

Chairman of the Session

Dr. R. LÁSZTITY  
University Professor  
Technical University  
Müegyetem rkp. 3  
H-1111I Budapest  
Hungary

President de la Session

President de la reunión

AUSTRALIA

AUSTRALIE

R.C. NORRIS

Australian Government Analyst  
Department of Science and Technology  
P.O. Box 65  
Belconnen A.C.T. 2616, Australia

D. BARVIR

Ing. State Inspection of Food Quality  
18000 Pobrezni 10  
Prague 8 - Karlín, Czechoslovakia

AUSTRIA

AUTRICHE

H. WOIDICH

Univ. Prof. Dr.  
Lebensmittelversuchsanstalt  
Blaasstr. 29  
Wien A-1190. Austria

D. PROCHÁZKA

Ing. State Inspection  
Podjavorinsky 4  
Bratislava 80100, Czechoslovakia

CANADA

J.P. BARRETTE

Chief (R+D)  
Laboratory Services Division  
Food Production and Inspection Branch  
Agricultura Canada  
Ottawa, Ontario. K1A 0C5, Canada

DENMARK

DANEMARK

DINAMARCA

P. KNUTHSEN

M.Sc., Chem. Eng.  
National Food Institute  
Mørkhøjs Bygade 19  
2860 Søborg, Denmark

H.B.S. CONACHER

Chief, Food Research División  
Health Protection Branch  
Tunney's Pastora  
Ottawa, Ontario K1A 062, Canada

FINLAND

FINLANDE

FINLANDIA

P.L. PENTTILA

Inspector of Foods  
National Board of Trade and Consumer  
Interests  
Haapaniemenkatu 4B  
00530 Helsinki 53, Finland

CZECHOSLOVAKIA

TCHECOSLOVAQUIE

CHECOSLOVAQUIA

J. RAJAMA

Research Officer  
Technical Research Center of Finland  
Food Laboratory  
Biologin Kuja 1  
02150 Espoo 15, Finland

FRANCE

FRANCIA

Ms. J. CASTANG  
Inspecteur General  
Service de la Répression des Fraudes  
et du Contrôle de la Qualité  
44 Blvd. de Granalla  
75015 Paris, France

Ms. C. SOULES  
Directeur Central de Laboratoire  
Service de la Répression des Fraudes  
et du Contrôle de la Qualité  
44 Blvd. de Grenelle  
75015 Paris, France

B. NINARD  
Directeur du Departement des Etudes  
Hydrogiques et Thermales du  
Laboratoire National du Ministère de  
la Sante  
1 rue Lacreteille  
75015 Paris XV<sup>e</sup>, France

GERMANY  
ALLEMAGNE, Rep. Fed.,  
ALEMANIA, Rep. Fed.,

W. KRÖNERT  
Director Profesor  
Head of Food Chemistry Division  
Federal Health Instituto  
Thiel Alle 82-84 Postfach  
0-1000 Berlin 33, Fed., Rep. of  
Germany

U. THIELE  
BASF-A6  
D-2HU  
D-6700 Ludwigshafen , Fed., Rep. of  
Germany

HUNGARY  
HONGRIE  
HUNGRIA

B. BORSZÉKI  
Senior Technical Officer  
Hungarian Office for Standardization  
Üllői ut 25  
H-1450 Budapest 9, Hungary

I. OLÁH  
Head of Department for Agriculture and  
Food Industry  
Üllői ut 25  
H-1450 Budapest 9, Hungary

L. KÖRMENDY  
Scientific Consultant  
Hungarian Meat Research Inst.  
Gubacsi ut 61B  
H-1017 Budapest

K. LINDNER  
Professor of the College of Commerce  
and Catering  
Alkotmány u 9-11  
H-1054 Budapest, Hungary

K. KISMÁRTON  
Deputy of División  
Hungarian Office for Standardization  
Üllői 25  
H-1450 Budapest, Hungary

M. UZONYI  
Head of Laboratory  
Hungarian State Control Station for  
Dairy Products  
Bartók B. ut 102  
H-1113 Budapest, Hungary

P. MOLNÁR  
Vice Director  
Centre of Food Control and  
Investigation  
Guszev u.25  
H-1051 Budapest, Hungary

F. ÖRSI  
Assistant Profesor  
Technical University  
H-1521 Budapest, Hungary, PF 92

JAPAN  
JAPON

T. HORIBA  
Technical Officer  
Agricultural and Forestry Products  
Inspection Instituto  
4-7 Konan-4-chome  
Minato-ku  
Tokyo

JORDAN  
JORDANIA

S. EMEISH

Head of Laboratories  
Directorate of Jordan Standards  
Ministry of Industry and Commerce  
P.O. Box 2019  
Amman H.K., Jordan

KUWAIT

Yacoub AL-MUTAWA  
Head of Public Health Laboratory  
Department of Health and Planning  
Ministry of Health  
Kuwait

Abdulwahab AL-MUTAWA  
Director of Food Inspection  
Department of Municipality  
P.O. Box 10  
Kuwait

NETHERLANDS  
PAYS-BAS  
PAISES BAJOS

W.J. de KOE  
Ministry of Public Health and  
Environmental Higiene  
Dr. Reyersstraat 10-12  
Leidschendam, Netherlands

P.L. SCHULLER  
National Instituto of Public Health  
P.O. Box 1  
Bilthoven, Netherlands

L.G.M.Th. TUINSTRA  
State Instituto for Quality Control of  
Agricultural Products  
Bornsesteeg 45, 5700 PD Wageningen  
Netherlands

P.W. HENDRIKSE  
Unilever Research Laboratory  
Vlaardingen POB 114  
3130 AC Vlaardingen, Netherlands

NORWAY  
NORVEGE  
NORUEGA

O.R. BRAEKKAN  
Prof., Head, Vitsmin Research Instituto  
Directorate of Fisheries  
POB 187  
5001 Bergen, Norway

S. NOSSEN  
Ministry of Agricultura  
Processed Fruits and Vegetable Control  
División  
Gladengv. 3B  
Oslo 6, Norway

A.O. HOUGEN  
Norwegian Food Research Inst.  
P.O. Box 50  
N-1432 As NLH, Norway

H. BLOKHUS  
Chief Inspector  
Directorate of Fisheries  
Division for Inspection and Quality  
Control  
P.O. Box 185  
5001 Bergen, Norway

POLAND  
POLGNE  
POLONIA

Ms. K. MAZURKIEWICZ  
Ministry of Foreign Trade and Shipping  
Quality Inspection Office  
Lurawia Str. 32/34  
05-015 Warsaw, Poland

W. MARTINEK  
Ministry of Foreign Trade and Shipping  
Quality Inspection Office Laboratory  
Reymonta Str. 11/13  
60-791 Poznan, Poland

Ms. T. LAWADZKA  
Ministry of Health and Public Welfare  
State Instituto of Hygiene  
Chocimska Str. 24  
00-791 Warsaw, Poland

SPAIN  
ESPAGNE  
ESPANA

R. GARCIA FAURE  
Dr. Ing., Laboratorios Agracios  
Av. Puerta de Hierro s/n  
Madrid-3, Spain

J.J. SANCHEZ SAEZ  
Dr. Chemist  
Comision Interm.Ord.Alim.  
National Centre Food and Nutrition  
Majadahonda, Madrid, Spain

R. REYES RODRÍGUEZ  
Veterinaire Service da Defense contra  
Fraudes  
Mo da Agricultura  
P. Infanta Isabel 1  
Madrid, Spain

L. BRIZ ESCRIBANO  
FORDPA  
Ministero da Agricultura  
Jose Abascal-4  
Madrid-3, Spain

SWITZERLAND  
SUISSE  
SUIZA

P. DOSSIER  
Head of Codex Section  
Federal Office of Public Health  
Haslerstrasse 16  
CH-3008 Berna, Switzerland

R. GERBER  
Dr. Phil., Section Head,  
Federal Office of Public Health  
Haslerstrasse 16  
CH-3008 Berna, Switzerland

P. KOCH  
Dr. Phil., Nat. Physicist  
Swiss Office of Metrology  
CH-3084 wabern/Berne, Switzerland

P. VENETZ  
Ing.Chim., Soc.D'Assistance Technique  
pour Produits Nestlé S.A  
CH-1814 La Tour de Peilz  
Switzerland

THAILAND  
THAILANDIA

V. WANARAKS  
Director of Inspection Division  
Food and Drug Administration  
Ministry of Public Health  
Bangkok, Thailand

UNITED KINGDOM  
ROYAUME-UNI  
REINO UNIDO

R. WOOD  
Principal Scientific Officer  
Ministry of Agricultura Fisheries and  
Food  
Great Westminster House  
Horseferry Road  
London SW1P 2AE, UK

R. SAWYER  
Superintendent Food and Nutrition  
Laboratory of the Government Chemist  
Cornwall House  
Stamford Street, London SE1 9NQ, UK

C.D. USHER  
Analytical Chemist  
Food Manufacturers Federation Inc.  
6 Catherine Street  
London WC2B 5JJ, UK

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

W. HORWITZ  
Acting Director, Science Policy Staff  
Bureau of Foods HFF-7  
Food and Drug Administration  
Washington D.C.20204, USA

W. DUBBERT  
DVM Acting Director of Staffs Technical  
Services  
Meat and Poultry Inspection Program  
Food Safety and Quality Service  
US Department of Agriculture  
Washington D.C.20250, USA

P. KHAN  
Vice President Quality and Food  
Protection  
ITT-Continental BKG Co. POB 731  
Rye, N.Y .10580, USA

J.A. YERANSIAN  
Corporate Research Manager  
Analytical Chemistry Laboratory  
General Foods Technical Center  
White Plains  
New York 10625, USA

Ch. H. BROKAW  
Director  
Product Integrity and Development  
Coca-Cola USA  
P.O. Drawer 1734 Atlanta  
6A 30301, USA

Ms. G.E.S. COX  
Chief Executive Officer  
Cox and Cox Investments  
12006 Auth Lane  
Silver Spring, MD 20902, USA

J.S. WINBUSH  
Director  
Division of Mathematics  
Food and Drug Administration  
200 C Street S.W.  
Washington D.C.20204, USA

YUGOSLAVIA  
YUGOSLAVIE

Ms. D. NOVICEVIC  
Savezni Zavod za Standardizaciju  
Beograd, Yugoslavia

INTERNATIONAL ORGANISATIONS  
ORGANISATION INTERNATIONALES  
ORGANIZACIONES INTERNACIONALES

AOAC (Association of Official Analytical Chemists)

W. HORWITZ  
Director, Editor, Official Methods of Analysis  
AOAC Box 540  
Benjamin Franklin Station  
Washington D.C. 2004, USA

EEC (Commission of the European Communities)

Ms. O. DEMINE  
Administrateur  
Direction Générale du Marché Intérieur  
et des Affaires Industrielles  
200 rue de la Loi  
Bruxelles 1030, Belgium

ISO (International Organization for Standardization)

K.G. LINGNER  
Senior Technical Officer  
ISO Central Secretariat  
1 rue de Varembe  
CH-1211 Geneva 20, Switzerland

J. KANIZSAY  
Chief of the Secretariat of ISO/TC 34  
and of the National Codex Committee  
Hungarian Office for Standardization  
Ollói ut 25  
H-1450 Budapest, Hungary

G. CASTAN  
Directeur de Département Association  
Francaise de normalisation Tour  
Europe  
92080 Paris La Défense, France

H.W. SCHIPPER  
Head, Foods and Agriculture Dept.  
Nederlands Normalisatie Instituut  
Kalfjeslaan 2  
2623 AA Delft, Netherlands

ICC (International Association of Cereal Chemists)

R. LÁSZTITY  
Vice President, ICC  
Schwechat, Smiedgasse 3-8  
Austria

NMKL (Nordic Committee on Food Analysis)

O.R. BRAEKKAN  
Prof. Head, Vitamin Research Institute  
Directorate of Fisheries  
P.O. Box 187  
5001 Bergen, Norway

ASMO (Arab Organization for Standardization and Metrology)

S. EMEISH  
Head of Laboratories  
Directorate of Jordan Standards  
Ministry of Industry and Commerce  
P.O. Box 2019  
Amman H.K., Jordan

FAO/CODEX SECRETARIAT  
SECRETARIAT FAO/CODEX  
SECRETARIA FAO/CODEX

L.G. LADOMERY  
Food Standards Officer  
Joint FAO/WHO Food Standards  
Programme  
FAO  
00100 Rome, Italy

N. Rao-MATURU  
Food Standards Officer  
Joint FAO/WHO Food Standards  
Programme  
FAO  
00100 Rome, Italy

Ms. J.V.I. WORRELL  
Joint FAO/WHO Food Standards  
Programme  
FAO  
00100 Rome, Italy

HUNGARIAN SECRETARIAT  
SECRETARIAT HONGROIS  
SECRETARIAT HONGARO

Or. D. Törley  
Assistant Professor, Technical  
University  
Budapest Müegyetem rkp.3  
H-1111 Budapest, Hungary

GENERAL PRINCIPLES FOR THE ESTABLISHMENT OR  
SELECTION OF CODEX SAMPLING PROCEDURES

INTRODUCTION

The following text is intended to be added as paragraphs 3 and 4 to the General Principles for the Establishment of Codex Methods of Analysis and Sampling contained in Appendix II to ALINORM 79/23:

"3. Purpose of Codex Methods of Sampling

The sampling methods are intended for use as international methods designed to avoid or remove difficulties which may be created by diverging legal, administrative and technical approaches to sampling and by diverging interpretation of results of analysis in relation to lots or consignments of food, in the light of the relevant provision(s) of the applicable Codex standards.

4. Methods of Sampling

(A) Types of Sampling Plans and Procedures

(a) Sampling Plans for Commodity Defects:

These are normally applied to visual defects (e.g. loss of colour, mis-graded for size, etc.) and extraneous matter. They are normally attribute plans, e.g. as given in CAC/RM 42-1969.

(b) Sampling Plans for Net Contents:

These are sampling plans which apply to pre-packaged foods generally and are intended to serve to check compliance of lots or consignments with provisions for net contents.

(c) Sampling Plans for Compositional Criteria:

Such plans are normally applied to analytically determined compositional criteria (e.g. percentage of fat in milk products, etc.). They are predominantly based on variable procedures with unknown standard deviation, e.g. as given in US MIL-STD 414.

(d) Specific Sampling Plans for Health-related Properties:

Such plans are generally applied to heterogeneous conditions, e.g. in the assessment of microbiological spoilage, microbial by-products or sporadically occurring chemical contaminants.

(B) General Instructions for the Selection of Methods of Sampling

(a) Official methods of sampling as elaborated by international organizations occupying themselves with a food or a group of foods are preferred. Such methods, when attracted to Codex standards, may be revised using Codex recommended sampling terms [to be elaborated].

(b) The appropriate Codex Commodity Committee should indicate, before it elaborates any sampling plan, or before any plan is endorsed by the Codex Committee on Methods of Analysis and Sampling, the following:

- (i) The basis on which the criteria in the Codex Commodity standards have been drawn up (e.g. whether on the basis that every item in a lot, or a specified high proportion, shall comply with the provision in the standard or whether the average of a set of samples extracted from a lot must comply and, if so, whether a minimum or maximum tolerance, as appropriate, is to be given);
  - (ii) whether there is to be any differentiation in the relative importance of the criteria in the standard and, if so, what is the appropriate AQL value each criterion should attract, and hence, the basis for judgement when a lot is in conformity with a standard.
- (c) Instructions on the procedure for the taking of samples should indicate the following:
- (i) The measures necessary in order to ensure that the sample taken is representative of the consignment or of the lot;
  - (ii) the number of individual items forming the sample taken from the lot or consignment;
  - (iii) the administrative measures for taking and handling the sample.
- (d) The sampling protocol may include the following information:
- (i) The statistical criteria to be used for acceptance or rejection of the lot on the basis of the sample;
  - (ii) the procedure to be adopted in cases of dispute.
- (e) Guidelines for the administrative and legal measures to be taken when lots fail to comply with the requirements of standards."

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

1. At the Twelfth Session of the Codex Committee on Methods of Analysis and Sampling (CCMAS) held in Budapest from 11-15 May 1981, an Ad Hoc Working Group was convened primarily to review the status of ongoing work towards harmonizing, within Codex, of procedures regarding acceptance sampling for the determination of net contents.

2. The Membership of the Working Group was as follows:

Denmark	P. Knuthsen
Finland	P. L. Penttila
France	J. Castang
	C. Soules
Hungary	M. Uzonyi
	P. Molnar
	L. Kömendy
	A. Ambrus
Netherlands	W.J. de Koe
Norway	Arne Hougen
	H. Blokhus
Spain	R. Reyes
	J. Briz
Switzerland	P. Koch
United States of America	C.H. Brokaw (Rapporteur)
	P. Khan
	J. Wimbush
	B. Dubbert (Chairman)
ISO	H.W. Schipper
SEC	O, Demine
FAO	L.G. Ladomery (attended in part only)

3. The representative of FAO suggested that the Working Group include in its consideration the following points:

- (a) How should the Working Group proceed further concerning the matter of acceptance sampling for net contents. This point should be considered from the context of large-consignments of foods moving in international trade.
- (b) What other Codex Committees should be involved in the furtherance of harmonizing principles and practices related to acceptance sampling for net contents?
- (c) Should the CCMAS consider the matter of sampling for net contents in relation to the way net contents are declared on the label?
- (d) Can the Working Group consider other aspects of sampling in addition to net contents?

4. The Chairman reviewed the activities of the Working Group convened at the 11<sup>th</sup> Session of the CCMAS, specifically the questionnaire described in paragraph 10, Appendix III, Report of 11th Session CCMAS, ALINORM 79/23. This questionnaire was circulated between the 11th and 12th Sessions of the CCMAS but only one response was received. Another response was forwarded though not received.

5. It was agreed that the mechanism of a questionnaire is still the best means available to the Group in determining what sampling policies exist among Member Countries and how they view the various sampling plan philosophies currently in use or being proposed. To that end, it was further agreed that the previously distributed questionnaire would be revised and amended as necessary and forwarded on the following target schedule:

July 1, 1981	Chairman of Working Group forwards redrafted questionnaire and attachments to Working Group members for comment on form and content.
October 1, 1981	Working Group comments due to be sent to Chairman.
January 1, 1982	Final form of questionnaire and associated documents forwarded to Codex Secretariat in Rome.
February 15, 1982	Codex Secretariat forwards questionnaire and documents to Codex Member Nations.
May 15, 1982	Responses due from Member Nations to Rome, with copy to Chairman of Working Group.
October 1, 1982	Summary of questionnaire results mailed to Working Group members.
December, 1982	Working Group reports to 13th Session of CCMAS and determines future action.

6. The Working Group felt that liaison should be established with appropriate Codex Commodity and other Codex Committees (including those on Labelling and General Principles) which service the Commodity Committees. However, such contact, it felt, should be postponed until the results of the questionnaire are known and some proposal for acceptance sampling has been further considered by the CCMAS.

7. The Working Group did not have time to discuss labelling matters but would plan to do so according to the proposed liaison contacts noted in paragraph 6. above.

8. The Working Group devoted its time only to considering aspects of acceptance sampling for net contents.

GENERAL METHODS FOR THE DETERMINATION OF METALLIC CONTAMINANTS  
IN FOOD SUBMITTED TO THE COMMISSION AT STEP 5 OF THE PROCEDURE (see  
Paras 46-60)

- Mercury - Official methods of analysis of the AOAC XIII (1980) 25.110-25.116 is advanced as a Codex Reference Method for the determination of mercury in fish and sea food (Type II) to Step 5 with the recommendation that Steps 6 and 7 be omitted.
- Lead - Official method of Analysis of the AOAC (1975) XII 25.066-25.064 or the equivalent AOAC (1980) XIII, 25.061-25.067 is advanced as a Codex Reference Method (Type II) with the recommendation that Steps 6 and 7 be omitted.
- Closed system digestion - anodic stripping method, AOAC (1980) XIII 1st Supplement 25.A03 - 25.A04 is advanced as a Codex Alternative Approved Method (Type III) to Step 5.
- Arsenic - Colorimetric (silver diethyl dithiocarbamate) method of AOAC (1980) XIII 25.012 - 25.013 is advanced as a Codex Reference Method (Type II) to Step 5 with the recommendation that Steps 6 and 7 be omitted.
- Hydride Generation - Atomic absorption method, AOAC XIII - 1st supplement 25.A01 - 25.A05 is advanced as a Codex Alternative Approved Method (Type III) to Step 5.
- Colorimetric (molybdenum blue) method of AOAC (1980) XIII 25.010-25.011 is advanced as a Codex Alternative Approved Method (Type III) to Step 5.
- Cadmium - . AOAC (1980) XIII 25.026 - 25.030 atomic absorption method is advanced as a Codex Reference Method (Type II) to Step 5 with the recommendation that Steps 6 and 7 be omitted.
- Anodic Stripping Voltammetry method, AOAC XIII (1980) 1st supplement 25.A01 - 25.A04 is advanced as a Codex Alternative Approved Method (Type III) to Step 5.
- Copper - AOAC (1980) XIII, 25.044 - 25.048 atomic absorption method is advanced as a Codex Reference Method (Type II) to Step 5 with the recommendation that Steps 6 and 7 be omitted.
- Zinc - AOAC (1980) XIII 25.150 - 25.153 atomic absorption method is advanced as a Codex Reference Method (Type II) to Step 5 with the recommendation that Steps 6 and 7 be omitted.
- AOAC (1980) XIII, First Supplement 25.A03 - 25.A05 Closed System digestion AA method is advanced as a Codex Alternative Approved Method (Type III) to Step 5.
- Tin - AAS method AOAC (1980) XIII, 25.136 - 25.138 is advanced as a Codex Reference Method (Type II) for the determination of tin at levels of 10 mg/kg in food to Step 5 with recommendation that Steps 6 and 7 be omitted.

## APPENDIX V

### SUMMARY OF METHODS ENDORSED OR TEMPORARILY ENDORSED (E = ENDORSED : TE = TEMPORARILY ENDORSED)

#### PROCESSED FRUITS AND VEGETABLES

##### Draft Standard for Canned Palmito at Step 5 (ALINORM 81/20, Appendix VI)

			<u>Type</u>
8.2	Determination of drained weight	E	I
8.3	Determination of water capacity of containers	E	II

##### Draft Standard for Canned Mangoes at Step 5 (ALINORM 81/20, Appendix VII)

8.2	Determination of minimum drained weight	E	I
8.3	Syrup measurements (Refractometric method)	E	I
8.4	Method for determination of water capacity of containers	E	II

#### COCOA PRODUCTS AND CHOCOLATE

##### Draft Standard for [Cocoa Butter Confectionary] at Step 6 (ALINORM 81/10, Appendix III)

8.1	Determination of total ash	E	I
8.2	Determination of percentage of cocoa butter: a) Total fat	E	I
8.3	Determination of milk fat	E	I
8.4	Determination of fat-free milk solids	E	I
8.5	Determination of moisture content	E	I

#### FISH AND FISHERY PRODUCTS

##### Canned Pacific Salmon, Proposed Revision at Step 5 (ALINORM 81/18 Appendix II)

6.2	Determination of net contents	E	
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#### FRUIT JUICES

##### Draft Standard for Nectars of Certain Citrus Fruits Preserved Exclusively by Physical means at Step 8 (ALINORM 81/14, Appendix I)

8.1	Taking of sample and expression of results as m/m	E	
8.2	Test for fermentability	E	I
8.4	Determination of sugars	TE	I
8.6	Determination of soluble solids	E	I
8.7	Determination of ethanol	E	I
8.8	Determination of essential oils	E	I
8.13	Determination of iron	E	II
8.15	Determination of sulphur dioxide	E	II
8.16	Determination of water capacity and fill of containers	E	II

##### Proposed Draft Standard for Pulpy Mango Nectar Preserved Exclusively by Physical means at Step 5 (ALINORM 81/14, Appendix III)

Methods as for Nectars (ALINORM 81/14, Appendix I)			<u>Type</u>
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Proposed Draft Standard for Concentrated Pineapple Juice Preserved Exclusively by Physical means at Step 5 (ALINORM 81/14, Appendix IV)

8.1	Taking of sample and expression of results as m/m	E	
8.2	Test for fermentability	E	I
8.3	Soluble solids	E	I
8.4	Acid	E	I
8.5	Determination of water capacity and fill of containers	E	II

Proposed Draft Standard for Concentrated Pineapple Juice with Preservatives for Manufacturing at Step 5 (ALINORM 81/14, Appendix V)

Methods as for Pineapple juice preserved by physical means (ALINORM 81/14, Appendix IV)

Proposed Draft Standard for Guava Nectar preserved Exclusively by Physical means at Step 3 (ALINORM 81/14, Appendix VI)

Methods as for Nectars (ALINORM 81/14, Appendix I).

FATS AND OILS

Draft Standard for Minarine at Step 8 (ALINORM 81/17, Appendix III)

9.1	Estimation of milk fat content	TE	I
9.2	Determination of fat content	E	I
9.3	Determination of loss of mass on drying	E	I
9.4	Determination of vitamin A content	E	II
9.5	Determination of vitamin D content	E	II
9.6	Determination of vitamin E	E	II
9.8	Determination of iron	E	II

QUICK FROZEN FOODS

Standards for Quick Frozen French Fried Potatoes (ALINORM 79/25, Appendix VIII)

8.5	Determination of Moisture Content	E	I
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Standards for Quick Frozen Corn-on-the-cob and Whole Kernel Corn (ALINORM 81/25, Appendix IV and V)

8.4	Determination of net weight	E	I
8.6	Determination of total soluble solids content	E	I

SOUPS AND BROTHS

Recommended International Standard for Bouillons and Consommés (ALINORM 78/9, Appendix II; ALINORM 79/33, Appendix I)

			<u>Type</u>
9.2	Determination of creatinine		II
9.3	Determination of total nitrogen	TE	II
9.4	Determination of amino nitrogen		II
9.5	Determination of Na Cl	TE	II