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REPORT OF THE FIFTH SESSION

OF THE

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Cologne, 1 - 6 December 1969

This report includes the following Appendices:

Appendix I	List of Participants
Appendix II	List of Documents
Appendix III	Basic alumina (for analysis of olive oils)
Appendix IV	Methods of Analysis for Quick Frozen Foods
Appendix V	Guide to the Layout of Codex Sampling Methods
Appendix VI	Report of the first ad-hoc group on sampling
Appendix VII	Report of the second ad-hoc group concerned with the Status of Sampling Provisions in Codex Standards.

The following is a key to the abbreviated references to methods:

- A.O.A.C Official methods of Analysis of the Association of Official Agricultural Chemists
- I.F.J.U Methods of Analysis of the International Fruit Juice Union
- I.S.O Methods of the International Organization for Standardization
- I.W.O Methods of Analysis of the International Wine Office

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CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Report of the Fifth Session

PART I

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its fifth session from 1 to 6 December in the Industrie-und Handelskammer, Unter Sachsenhausen 10 - 26, Cologne, under the chairmanship of Prof. Dr. R. Franck. The session was opened by Frau Helga Merkel, Abteilungsleiterin, Bundesministerium für Jugend, Familie und Gesundheit, who stressed the importance of the work of the Codex Committee on Methods of Analysis and Sampling since, she pointed out, it cut across the whole field of food standardization. Frau Merkel wished the Committee success in its work. There were 59 delegates and observers present, representing 22 countries and 13 international organizations. The list of participants appears as Appendix I and the list of documents which the Committee had before it as Appendix II to this Report.

ADOPTION OF AGENDA

2. The Committee agreed to adopt the Revised Agenda (CODEX/ANALYS/69/A/2 - revised) with the following amendments:

- (a) to discuss a proposal concerning specific extinction method in UV for olive oils under Agenda Item 5(a);
- (b) to postpone the discussion of Agenda Item 6 until the Minutes of the Drafting Group, which met in Wädenswil (November 28-29, 1969) to review IFJU methods applicable to the standards for fruit juices, were available in the working languages of the Committee;
- (c) to discuss document ISO/TC34/WG1 (Secr. 9) 17, dealing with the standard layout for ISO methods of sampling, under Agenda Item 11 and to consider this item at an early point during the session;
- (d) to reinstate Agenda Item 12 of the Provisional Agenda (CODEX/ANALYS/69/A/2) dealing with technical procedures of sampling foods.

APPOINTMENT OF RAPPORTEURS

3. Dr. H. Egan of the United Kingdom Delegation and Ir. J. Gosselé, the Belgian Delegate, agreed to act as rapporteurs and were so appointed by the Committee.

PART II

MATTERS ARISING FROM THE REPORT OF THE SIXTH SESSION OF THE CODEX ALIMENTARIUS COMMISSION

(a) Amendments to the Guidelines for Codex Committees

4. The Committee was informed that, with some amendments, the Codex Alimentarius Commission, at its Sixth Session, agreed to the Guidelines for the Elaboration of Codex Methods of Analysis proposed by this Committee. The text of paragraph 13(c) of the Guidelines relating to methods of analysis and sampling is given on

pages 55 to 58 of the second edition of the Procedural Manual of the Codex Alimentarius Commission (ref. no. Cx 8/7 - 2nd edition, 1969).

5. In connection with microbiological methods of analysis, the Committee noted that the Executive Committee at its 13th Session had requested clarification on these methods of analysis. It was also noted that section 13(c)(v), dealing with microbiological methods, clearly stated that the methods were intended for the verification of hygiene provisions. In discussion, the Committee distinguished between microbiological methods the purpose of which related to the assessment and control of bacteriological hazards and those which related to technological standards, e.g. vitamin content. The Committee agreed with the text as adopted by the Commission but felt that the latter type of method should be considered by the Codex Committee on Methods of Analysis and Sampling. It also noted that there might be provisions listed under the section on hygiene in Codex commodity standards, which did not involve microbiological methods of determination. It was agreed that such methods, e.g. determination of aflatoxins, filth, mould count, insect debris and other objectionable matter came within the terms of reference of this Committee. It was further agreed that clarification of these points was desirable and that the views of the Committee should to this end be made available to the Codex Committee on Hygiene for information and comment.

(b) General Principles for the Establishment of Codex Methods of Analysis

6. The Committee noted that the Commission, at its sixth Session, considered a proposal of the delegation of Canada to replace the word "proven" by the word "judged" in the last sentence of paragraph 1 of the above General Principles (see page 59 of the 2nd edition of the Procedural Manual of the Codex Alimentarius Commission and paragraph 91, ALINORM 69/67) but could not reach a decision at that time and decided to re-examine this matter in more detail, should it appear to be necessary.

7. The Committee recognized that it had no laboratory facilities of its own and could therefore only judge the equivalence of methods on the basis of work done by other bodies, but that collaborative studies might from time to time be organized by member countries. The Committee agreed that the text did not need to be amended as proposed by the delegation of Canada.

(c) Methods of Analysis for Powdered Sugar

8. The Committee noted that the Commission at its Sixth Session requested it to consider methods of analysis for powdered sugar which were to be elaborated by ICUMSA. It was noted that the methods already endorsed for the quality criteria of white sugar would also be recommended by ICUMSA for powdered sugar. The Secretariat pointed out that these methods of analysis might be before this Committee at its next session. It was agreed to consider this further when the methods became available.

(d) Uniform Numbering and Layout of all Codex Documents

9. The Committee was informed that the Secretariat had taken steps to bring uniformity into the preparation of the Codex documents (see paragraph 93 of

ALINORM 69/67) and that the Executive Committee had made recommendations to the Commission in this connection at its 14th Session (ALINORM 70/3 paragraph 33 and Appendix I). Some delegations expressed their concern about the complexity of the numbering of the present documents. It was pointed out that this was inevitable since many of the documents prepared for sessions of other Codex Committees were also working documents for this Committee. The Secretariat informed the Committee that this question would be discussed at the Seventh Session of the Codex Alimentarius Commission.

PART III

METHODS OF ANALYSIS IN STANDARDS FOR FATS AND OILS

OLIVE OILS

(a) Solvent Residues

10. In considering the determination of solvent residues in olive oils the Committee was informed by the Secretariat that the Sixth Session of the Codex Committee on Fats and Oils had deleted reference to solvent residues in olive oil (ALINORM 70/11 paragraph 15 (d) (ii)). However, the Codex Committee on Methods of Analysis and Sampling was of the opinion that the question of analysis for residues of extraction solvent should, if possible, be considered for fats and oils generally by the Codex Committee on Food Additives and by the Joint FAO/WHO Expert Committee on Food Additives.

11. The Secretariat stated that the opinion of the Committee would be made known to the Secretariat of the Joint FAO/WHO Expert Committee on Food Additives but that, at this time, no prediction could be made as to whether or not the Expert Committee would wish to make a detailed consideration of the methods of analysis concerned. It was understood that any method developed by the Expert Committee would have to be placed before the Codex Committee on Food Additives and subsequently endorsed by the Codex Committee on Methods of Analysis and Sampling. (See Procedural Manual, 2nd edition, Guidelines for Codex Committees, paragraph 13 (c) (i)).

12. The Committee noted that some methods proposed for the determination of solvent residues (JAOAC(1967)50, 717-726) had been subjected to collaborative testing and were found to be satisfactory; however, it was necessary also to consider residues of other solvents e.g. chlorinated solvent residues. The Committee invited Governments to furnish information on this subject.

(b) Teaseed Oil Test

13. The Committee reconsidered the teaseed oil test which it had endorsed at its Fourth Session (ALINORM 69/23 paragraphs 76 and 77), in the light of the amendment proposed by the Codex Committee on Fats and Oils at its Sixth Session. The Committee agreed that the Note given in CODEX/FATS AND OILS/40 Appendix 6 should be replaced by the following text:

"A pink colour, indicating less than 10% teaseed oil, shall not be regarded as positive evidence of the presence of this adulterant."

(c) Specific extinction in the ultra-violet

14. The Committee considered the amendment proposed by the Codex Committee on Fats and Oils at its Sixth Session concerning specification of the alumina to be used. The French proposal for the more precise definition of the type of Brockmann grade of the alumina to be used in preparing the sample for this test and to be included under the heading "Reagents" of the method (CODEX/FATS AND OILS/40, Appendix 8) was accepted (see Appendix III).

MUSTARDSEED OIL

(a) General Methods of Analysis

15. The Committee agreed that, although the Sixth Session of the Codex Committee on Fats and Oils had made no specific recommendation for methods of analysis for the determination of contaminants and assessment of the quality and identity of mustardseed oil, the methods of analysis endorsed for the General Standard for edible fats and oils at the Sixth Session would also be applicable to mustardseed oil. The Committee agreed not to discuss these methods again and decided to endorse the methods included in ALINORM 69/23 (paragraphs 57-59) for the standard for edible mustardseed oil.

(b) Determination of Allyl Isothiocyanate Content

16. The Committee endorsed the method given in the Indian Standard IS:548-1964 pages 56-57 with the following amendments, subject to verification by the Secretariat with the relevant Indian Analytical Reagent Standards for nitric acid and ethanol.

"17.2.4 Nitric acid, Analytical Reagent Grade - 69% m/m HNO₃"

"17.2.1 Ethanol 90-95% v/v, neutral to phenolphthalein".

MARGARINE

Determination of Water Content

17. The Committee had before it the proposed method of analysis for water content which appeared as Appendix IX A - Draft Standard B-9 (1968) to Report of the 11th Session of the Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products (pages 124-125). This method had been drafted for determination of water in butter by the International Dairy Federation (IDF) but had not yet been accepted by these bodies for inclusion in the butter standard. It was further explained that the varying composition of margarine, especially with respect to emulsifiers, called for a different method. The Committee also considered another proposal to use a method for the determination of water involving the use of sand or pumice and also an alternative method using a temperature of 105° in the drying procedure. It was pointed out that, while the drying temperature in the IDF procedure for butter was 102[±] 2° C, the standard ISO drying temperature for this product was 103[±] 2° C.

18. The Committee agreed to this latter temperature in principle but it was divided on the question whether or not the same method as for butter should be used for determining the water content of margarine. The method drafted for butter was not adopted but it was agreed that the matter should be further discussed in the light of comparative results for margarine obtained using both methods. The Netherlands delegation agreed to provide such results for the Sixth Session of the Committee.

PART IV

METHODS OF ANALYSIS IN STANDARDS FOR FRUIT JUICES

19. The Committee took note of the recommendation made by the Joint ECE/Codex Alimentarius Group of Experts on Standardization of Fruit Juices at its Seventh Session to include the methods of the International Federation of Fruit Juice Producers in the standards (ALINORM 70/14 paragraph 21). It was also informed that a list of these methods had been prepared by a Drafting Group meeting at Wädenswil (28-29 November 1969) under a request made by the Joint ECE/Codex Group of Experts (ALINORM 70/14 paragraph 24). The Committee had before it documents

- CODEX/ANALYS/69/B/1 Government Comments on Methods of Analysis and Sampling for Fruit Juices (1969)
- CODEX/ANALYS/69/A/8 Minutes of a Meeting of the Drafting Group on Methods of Analysis for Fruit Juices (Wädenswil, 28-29 November 1969)
- CODEX/ANALYS/68-2/1 Digest of Comments from Governments and Organizations on the Synopsis of Methods of Analysis for Fruit Juices (1968)
- CODEX/FRUJU/69/2 Methods of Analysis and Sampling for Fruit Juices (including CODEX/ANALYS/67-2) Synopsis of Methods of Analysis for Fruit Juices)
- CODEX/FRUJU/69/4 Paper by the Swiss Delegation on the Cadmium Content of Fruit Juices.

The Committee decided to base its discussion on document CODEX/ANALYS/69/A/8.

(a) Paragraph "Description" of the standards

20. Concerning the provision for a test for fermentability it was pointed out that this would not necessarily give a clear indication of the absence or presence of chemical preservatives. However, as the Joint ECE/Codex Group of Experts on Fruit Juices had inserted a provision for fermentability into the standards, it was agreed that a test was required which related only to whether or not the juice was fermentable; and that the provision for the claim that the juice had been preserved exclusively by physical means, and therefore should be free from chemical preservatives would require appropriate methods of analysis. The Chairman of the Sub Commission on Methods of Analysis of the IFJU stated that such a method for fermentability was available in the collection of methods of his Organization.

(b) Minimum content of fruit ingredient

21. The Committee noted that the Joint ECE/Codex Group of Experts had not proposed methods of analysis for this criterion, and referred the matter back to the Commodity Committee.

(c) Soluble solids

22. The Committee noted that standards for fruit juices and nectars contained a provision for soluble solids to be determined by refractometer but that the Drafting Group of the Joint ECE/Codex Group of Experts had recommended a gravimetric method (IFJU-8, rev. 1968, Method A). The Committee, in conformity with paragraphs 1 and 2(d) of the General Principles for the Establishment of Codex Methods of Analysis, endorsed the refractometric (indirect) method of the IFJU-8, rev. 1968, Method B.3 for all standards, except for apple juice. It was noted that the standard for apple juice contained a provision for added sugar. The Committee agreed that the refractometric method, which estimated total soluble solids including added sugar, was not applicable to the standard for apple juice. Since no method had been proposed for the determination of added sugars in the presence of natural sugars, the matter was referred back to the Commodity Committee for further consideration. A preference for a method of drying at 70°C was indicated by the Committee in connection with a method for the estimation of total solids.

(d) Sugars

23. The Committee endorsed the IFJU method (IFJU-4, rev. 1968 - Method Luff-Schoorl) for all standards, except lemon juice, but requested the IFJU to make available to it the results of collaborative studies carried out on this method. Reservations were made by the delegations of Canada, Sweden and the United States on this method.

(e) Honey

24. The Committee noted that no method of determination of this ingredient had been proposed for apricot, peach and pear nectars.

(f) Salt

25. The Committee discussed whether it was sufficient to determine the chloride content of tomato juice as the basis of determination for added salt (sodium chloride) or whether the determination of sodium was also necessary. The Committee agreed that the determination of chloride was sufficient and endorsed the IFJU method (Cl⁻: IFJU-37, 1968 - potentiometric) for tomato juices.

(g) Essential oils

26. In the absence of an IFJU method the Committee agreed with the recommendation of the Drafting Group to endorse the AOAC method (19.110, 20.073, 20.074) for orange, lemon and grapefruit juices.

(h) Ethanol

27. The Committee endorsed the IFJU method (IFJU-2, rev. 1968) for all fruit juice standards except tomato juice. In this connection it was pointed out that while the method was sufficiently accurate for the requirements of the standards (3 to 5 g ethanol per kg), an oxidation method should be used for the determination of smaller quantities of ethanol. Regarding the need to work at higher temperatures in tropical countries, the Committee noted that tables were available which could be used to convert the results of the analysis to the equivalent result obtained at 20°C. The delegation of the United States pointed out that such tables were available in the publications of the AOAC; the representative of IWO mentioned that a manual including alcoholometric tables at higher temperatures would be published in 1970 by the International Organization of

Legal Metrology. The Committee requested the IFJU to amend its method to take into account operating temperatures higher than 20°C.

(i) Apparent viscosity

28. The Committee endorsed the AOAC method (Lamb and Lewis, JAOAC, 50, 207, 1967) for the apparent viscosity of apricot, peach and pear nectars, as recommended by the Commodity Committee.

(j) Hydroxymethylfurfural (HMF)

29. The Committee endorsed the IFJU method (IFJU-12, rev. 1968) for apricot, peach and pear nectars, with the modification by Postel (Deutsch. Lebensm. Rundsch. 64, 1968, 318).

(k) Volatile acids

30. It was pointed out that the IFJU method proposed by the Joint ECE/Codex Group of Experts was subject to error due to a slight carry-over of lactic acid (where present) into the distillate and that the method of the IWO contained provisions to eliminate this error. It was also stated that the IWO method was more reliable and more precise than the proposed IFJU method. In view of the fact that the standards in question did not permit the addition of lactic acid and since the limit for volatile acids included any such acids which may be present naturally in the juices (e.g. some lactic acid), the Committee endorsed the IFJU method (IFJU-5, rev. 1968) for apple, orange and grapefruit juices.

(l) Additives

31. The Committee agreed that no Codex Methods were required for citric and malic acids in apricot, peach and pear nectars since no maximum levels for these additives were included in the standard and since these acids were normally found in nectars and fruit juices.

32. Although no maximum limits were stated for l-ascorbic acid, the Committee agreed to endorse the IFJU method (IFJU-17, 1964, P. Spanyol and co-workers method as published in Zeitschr. f. anal. Chemie. 195, 268 (1963) and Zeitschr. Lebensmittel-Unters. u. Forsch. 123, 93-102 (1963) and which measures both the ascorbic and the dehydro-ascorbic acid) for apricot, peach and pear nectars and for apple and grape juices, because added ascorbic acid can be used both as an anti-oxidant and, at higher levels, as a nutritive adjunct.

(m) Contaminants

33. The Committee noted that the various individual metallic contaminants (arsenic, lead, copper, iron, cadmium and zinc) were of interest in connection with numerous Commodity Standards and accepted a proposal that generally applicable methods should be used in all these standards. However, until the Committee has had the opportunity to discuss such general methods in detail, it was agreed that the methods suggested by the various Commodity Committees as detailed below should be endorsed but only temporarily, with the view to replacing them later with general methods. In doing this

the Committee drew the Commission's attention to the fact that various AOAC methods had been subjected to collaborative study and had already been endorsed for such contaminants in other standards where the Commodity Committees had not proposed any methods. The Committee also considered that atomic absorption spectrophotometric methods would in future be of increasing importance, and some reservations were expressed regarding the use of polarographic methods.

<u>Arsenic</u> (All standards)	IWO method A 196 (diethyldithiocarbamate) to be published in IFJU
<u>Lead</u> (All standards)	IFJU-14. 1964
<u>Cooper</u> (All standards)	IFJU-13, 1964
<u>Zinc</u> (All standards)	AOAC (1965) 24.078-24.084
<u>Iron</u> (All standards)	IFJU-15, 1964 (with dry ashing)
<u>Tin</u> (All standards)	ISO diphenylfluorone draft method (ISO/TC 34/SC3/WG3/ No. 120F), subject to further comments in the light of ISO studies currently in progress. The delegation of the Netherlands reserved its position on this method.

The Committee noted that no method was required for cadmium, as the provisions for a maximum level of cadmium had been deleted from the standards.

34. The delegations of the United States, Australia, Canada, Irish Republic and Sweden, had reservations to the adoption of the IFJU methods in para 33 because

- (a) of lack of adherence to the criteria for the selection of Codex methods as stated in paragraphs 2(b) and 2(g) p.60, of the Procedural Manual, 2nd Edition (not uniformly applicable to various groups of commodities; reliability not statistically established in comparative or collaborative studies in several laboratories - no record of such);
- (b) the method for arsenic is not yet available to the Committee;
- (c) the document for discussion should have been CODEX/FRUJU/69/2, not CODEX/ANALYS/69/A/8 which was directed towards IFJU methods only;
- (d) AOAC and NMKL methods mentioned in CODEX/FRUJU/69/2 are general methods which have been studied on a collaborative basis and the data for which are a matter of record.

35. Concerning the method for total metal content precipitable by K hexacyanoferrate (II) (all standards), the Committee endorsed, but only temporarily, the IFJU method to be published, based on method 30/22/23 of Schweitz. Lebensm. Buch, Chapter 30 (Wein).

(n) Determination of sulphur dioxide in fruit juices and sugars

36. The Committee's attention was drawn to the need for a referee method for determination of sulphur dioxide at the level of 10 mg/kg as indicated in the standards for apple juice and grape juice; and also at lower levels, as for verifying the absence of SO₂ in fruit juices such as citrus juices. The Committee considered the comparative study made on sugars, fruit juices and dried fruits by the Netherlands using the Monier Williams method and the Tanner method (CODEX/ANALYS/69/C/5) and expressed a preference for the Tanner method at the low sulphur dioxide concentrations concerned. The Committee also noted that an ISO method was being elaborated (Franz Paul method combined with Zonnefeld-Meyer method ISO/TC 34/SC3/GT3 (127 bis)) and that a comparative study of SO₂ determination in wines by the methods of Paul and of Tanner had been made by the Max von Pettenkofer Institute, in the Federal Republic of Germany. The Committee noted that the Franz Paul method, though satisfactory, gave slightly higher results than the Tanner method. It was noted also that an iodometric method would not be suitable in the case of fruit juices containing essential oils (e.g. citrus juices). The Committee endorsed the Tanner method IFJU No. 7-(1968) for all fruit juices.

37. The Committee also agreed to replace the Monier Williams method by the Tanner method for determining the sulphur dioxide level in sugars at Step 9 and to draw the Commission's attention to this proposed amendment.

(o) Mineral impurities insoluble in HCl (apple, grape and tomato juices)

38. The Committee considered that the exact concentration of HCl to be used was not critical and endorsed AOAC method (1965) 28.005.

(p) Hygiene provision

39. The Committee noted that the provision included in all standards for micro-organism capable of development was under Codex Committee on Food Hygiene's responsibility and that the provision for mould filaments had been deleted from the standards.

(q) Organoleptic test

40. The Committee was of the opinion that this test should be deleted for the present time as there were no methods of organoleptic testing included in Codex Standards and that this came under the general item "Methods of Sensoric Analysis" which would be given further discussion (see paras 88-90).

(r) Relative density (all standards)

41. The Committee considered the need for this determination as a referee method for conversion of all analytical results given by volume into results by weight (as presented in the standard) and endorsed IFJU-I, rev. 1968. The Committee also agreed to draw the Commission's attention to the fact that quantities should for preference be expressed per litre.

(s) Ash, alkalinity, pH, total titrable acid, non volatile acids, organic acids, tartaric acid, benzoic acid, sorbic acid, colouring agents

42. As these determinations are not directly related to the criteria given in standards, the Committee was of the opinion that no referee methods were necessary.

It was recognized, however, that a combination of several of these factors could be useful, e.g. for determining the degree of purity of a juice, and could be taken into consideration at an appropriate time in the future.

(t) Carbon dioxide (apple and grape juice)

43. The Committee endorsed IFJU-42, 1966 (Hennig and Lay Method).

(u) Minimum fill of containers (all standards)

44. The Committee agreed to draw the attention of the Joint ECE/Codex Group of Experts to the necessity of proposing a method for the determination of minimum fill of containers. It also agreed that, in this respect, a general method should be elaborated for all commodities.

(v) Degree of concentration of fruit juices

45. The Committee noted that no methods for measuring the degree of concentration of fruit juices had been proposed and referred this matter to the Joint ECE/Codex Group of Experts.

Considerations on IFJU methods

46. The representative of IWO made a general objection concerning the use of the following methods of analysis for fruit juices (grape juices in particular): relative density, soluble solids, volatile acidity, methods of measurements for absolute density, alcoholic titre and SO₂. The delegation of France made a reservation on these methods.

47. The representative of IFJU agreed that, where a method endorsed or to be endorsed by the Codex Committee on Methods of Analysis and Sampling had not been given sufficient comparative or collaborative studies, these studies should be undertaken as soon as possible. The representatives of AOAC and IWO agreed to contribute to such studies. The representative of IFJU agreed as a principle that, where a method which at present is not included in the IFJU Manual of Analysis has been endorsed by the Committee, this method would be included in this Manual at a later stage.

PART V

METHODS OF ANALYSIS IN STANDARDS FOR QUICK FROZEN FOODS at Step 8

48. The Committee had before it the following working papers:

- Net Weight Determination of Frozen Fruits and Vegetables
(CODEX/ANALYS/68/14)
- Thawing Procedure for Quick Frozen Fruits and Vegetables
(QFF/MAS/4 (1969))
- Cooking Procedure for Quick Frozen Vegetables
(QFF/MAS/5 (1969))

- Determination of the Alcohol-insoluble Solids Content of Quick Frozen Peas (CODEX/ANALYS/68/15)
- Determination of Ash Insoluble in Hydrochloric Acid (ISO Rec. R 763)
- Test Procedure for "Ash Insoluble in HCl" for Quick Frozen Strawberries and Similar Products ((QFF/MAS/1 (1969))

(a) Net weight determination (Quick frozen Fruits and Vegetables)

49. The delegation of the Federal Republic of Germany pointed out that the style in which this method was written was not suitable for an international referee method, and drew particular attention to the last paragraph of the section on Definition which would more properly form part of the standards for quick frozen foods. The Committee endorsed the method proposed by the Joint ECE/Codex Group of Experts (CODEX/ANALYS/68/14) with the deletion of paragraphs 2, 3 and 4 of the section on Definition (see Appendix IV).

(b) Thawing and Cooking Procedures (Quick frozen Fruits and Vegetables)

50. The Committee agreed that these methods (QFF/MAS/4/1969) needed editing to bring them into line with the layout of international referee methods of analysis. For example it was agreed that, with the exception of the first two sentences, section 3 dealing with the Principle of the cooking procedure appeared redundant. The Committee was of the opinion that the procedures for thawing and cooking did not constitute methods of analysis in the true sense but represented procedures of preparation of samples for further examination of the product. It was agreed that the methods should be amended editorially by the Secretariat and referred back to the Commodity Committee with the above remarks. (See Appendix IV)

(c) Determination of alcohol-insoluble solids content in quick frozen peas at Step 8

51. In discussion it was pointed out that the AOAC had studied collaboratively the method described in CODEX/ANALYS/68/15 and had found it acceptable at the level of 11% alcohol-insoluble solids. The results of the collaborative tests are given in JAOAC 52, page 11, 1969. The Committee endorsed the method, noting that it would be of interest to see the results of collaborative testing at the levels prescribed in the standard (19-23%). The delegation of Australia pointed out that the term "methylated spirit" needed clarification and proposed to replace it by ethanol denatured with 5% methanol". The Committee agreed to this amendment. (See Appendix IV)

(d) Mineral impurities - sand (Ash insoluble in HCl) (Quick frozen Strawberries at Step 6 and Quick frozen Raspberries at Step 5)

52. The Joint ECE/Codex Group of Experts had submitted two methods to this Committee for consideration (ISO Rec. R 763, which contained a reservation by the Netherlands, and QFF/MAS/1, 1969). It was agreed that a choice of methods depended on the intended meaning of the provision for mineral impurities; for example, while the ISO method measured ash insoluble in HCl, the method described in QFF/MAS/1, 1969 measured

extraneous matter as well as HCl insoluble intrinsic silica.

53. The Committee noted that both the above methods were still subject to review and while agreeing that in principle the method of the ISO was more appropriate, referred the matter back to the Commodity Committee for a choice of method. The delegation of Austria pointed out that in homogenizing the sample, heavy particles such as sand might tend to settle to the bottom of the container. In connection with the ISO method, it was proposed by the delegation of the Federal Republic of Germany to include demineralised water as an alternative to distilled water, to increase the ashing temperature to 1000° and to delete the words "at least" in paragraph 5.3. (See also paragraphs 58, 59, 63 and 67).

PART VI

METHODS OF ANALYSIS IN STANDARDS FOR PROCESSED FRUITS AND VEGETABLES

54. The Committee had before it the following working documents:

- CODEX/ANALYS/69/A/4
- ALINORM 69/23
- ALINORM 70/20
- CODEX/ANALYS/69/C/1

Canned Pears and Mandarin Oranges at Step 5

Drained weight determination and syrup measurements

55. The Committee endorsed the relevant methods described in Appendix IV, ALINORM 69/23 (Determination of drained weight Method I p.1, and Syrup measurements p.6) for canned pears and mandarin oranges, noting that methods had been previously endorsed for other processed fruits and vegetables.

Processed Tomato Concentrate at Step 5

(a) Soluble tomato solids

56. In discussion on the determination of natural soluble tomato solids, the delegation of the United States pointed out that the provision in the standard was based on the methods of analysis proposed by the Commodity Committee (see JAOAC 50, 1967 page 690) so that a change in the method of analysis would necessarily involve a corresponding change in the provision for natural soluble tomato solids. The Committee noted that the methods of analysis section of the standard on processed tomato concentrates implied a definition of natural soluble tomato solids but that, in fact, the proposed method measured total tomato solids. The Committee endorsed the above AOAC method but suggested that the title be referred back to the Commodity Committee for clarification.

(b) Determination of salt

57. The Committee endorsed the AOAC method (AOAC(1965)6.103, 6.104, 6.105) but noted that more detailed reference should be made to the relevant parts of the official methods of the AOAC to clarify such matters as the preparation of the tomato concentrate and the strength of nitric acid.

(c) Determination of mineral impurities

58. In discussion the delegation of Poland pointed out that there was a need to elaborate general methods for the determination of this provision, and subsequently to standardize the various differing provisions for mineral impurities. In discussing the proposed AOAC method, the question was raised as to which section (a) Sand and/or (b) Alkali-soluble silica, would be applicable. It was pointed out that the section (b) dealing with the combined alkaline filtrate and washings was necessary for the further determination of such substances as metals and that therefore it was not relevant to the method of determination of mineral impurities.

59. The Committee endorsed the AOAC method (AOAC (1965) 6.005) with the understanding that reference would be made to the relevant parts of the official methods of AOAC to clarify the standard reagents. (See also paras 52, 53, 63 and 67)

(d) Mould count

60. The Committee noted that the standard for processed tomato concentrate contained a mandatory provision relating to the presence of mould filaments in the section on hygiene. It was further noted that the actual limit for the number of positive fields was in the nature of a guide rather than a mandatory provision of the standard. It was pointed out that the degree of homogenization leads to an increase in the number of positive fields observed. This method could not therefore be regarded as being suitable as an international referee method but could serve as a guide as stated in the standard.

61. At the same time it was agreed that the matter should also be referred to the Commodity Committee for further guidance regarding the extent to which the mould count was also a technological consideration.

Raisins at Step 5

Moisture determination

62. It was pointed out that the proposed AOAC method (AOAC(1965) 20.009, 20.003 (c)) was a general method for fruit products including dried fruits. The Delegation of the Netherlands pointed out that in other similar methods sand was used in place of asbestos during drying process; in this connection it was stated that asbestos, in the AOAC method, was intended specially for raisins and other fruits rich in sugar. The Committee endorsed the AOAC method for the determination of moisture content in raisins but indicated the need to draw special attention to the manner of sample preparation and the kind of drying oven to be used.

Canned strawberries at Step 6

63. In discussing methods of determination of mineral impurities in canned strawberries (CODEX/ANALYS/69/C/1), the representative of ISO pointed out that this

method was the same as the ISO method R 762, 1968. The Committee agreed to draw this matter to the attention of the Commodity Committee with a request for clarification of the meaning of the provision for mineral impurities, and for a proposal of an appropriate method. (See also paras 52, 53, 58, 59 and 67)

Canned pineapple at Step 8

64. The Committee discussed the "size of sample" requirement of the standard and (following its discussion on sampling plans) agreed that this matter did not represent a statistical problem but an editorial one which should be clarified by the Secretariat in consultation with the Chairman of the Commodity Committee.

PART VII

METHODS OF ANALYSIS IN STANDARDS FOR FISH AND FISHERY PRODUCTS

Methods of Analysis for Canned Shrimp and Prawns at Step 6

65. With reservations by the delegation of the Federal Republic of Germany regarding the draining times concerned, the Committee agreed on the methods for determining drained weight and net content set out in Appendix IV to ALINORM 70/18

66. It was noted that the specification of circular sieve did not conform to ISO standards and agreed that the nearest ISO size, (above or below to be decided by the Commodity Committee) should be substituted with a footnote to say that the nearest corresponding US size could also be used. It was agreed that the methods for size determination, including a count of shrimp or prawns, should be referred to the Commodity Committee as it was not considered to be a method of analysis requiring endorsement. The method for the determination of water capacity of the container set out in Appendix IV to ALINORM 70/18 was agreed and it was also agreed that this should form the basis for a general standard for the capacity of open top cans.

PART VIII

METHODS OF ANALYSIS IN STANDARDS FOR EDIBLE FUNGI AND FUNGUS PRODUCTS, DRIED EDIBLE FUNGI AND FRESH FUNGUS "CHANTERELLE" at Step 8

67. The Committee had before it CODEX/ANALYS/69/A/6 "Methods of Analysis for Edible Fungi and Fungus Products, Dried Edible Fungi and Fresh Fungus "Chanterelle". The Committee discussed the method ISO R 763 for the determination of mineral impurities insoluble in hydrochloric acid and agreed to refer this matter to the Coordinating Committee for Europe for clarification as to what impurities were to be covered by the above provision in the standard; it also agreed that methods of determination of water, salt and sugars contents should be proposed by the above Committee. The delegation of Poland agreed to provide drafts of appropriate methods. The Secretariat was requested to bring these matters to the attention of the Commission. (See also paras 52, 53, 58, 59 and 63).

PART IXSAMPLING PLANS FOR PREPACKAGED FOODS AND TECHNICAL PROCEDURE OF SAMPLING FOODS

68. The Committee recognized at the outset the need for well-defined sampling plans and acceptance procedures and reaffirmed the view expressed at its fourth session that a systematic and comprehensive approach to the subject of sampling was necessary (ALINORM 69/23 paragraphs 80(f) 80(g)).
69. The representative of ISO reported that since the fourth session of this Committee the ISO/TC 34 Working Group WG-1 - Sampling had met and had
- (i) prepared a draft guide to the layout of sampling methods (document ISO/TC 34/WG-1 (Secretariat-9)17 - see Appendix V) to be accompanied by a series of explanatory notes, but not setting out individual sampling plans;
 - (ii) started work on a vocabulary relating to sampling with special reference to agricultural products; and
 - (iii) considered the Codex document on the General Statement on Sampling in the field of Food (ALINORM 65/25(1)), based on an ISO document.
70. The Committee welcomed this work and agreed that the draft guide to the layout of sampling methods should be drawn to the attention of Commodity Committees as a guide; it should however, be realized that the guide and notes for the guide were still being developed by ISO; it should also be circulated to Governments with a request for comments.
71. In addition the Committee had before it the Technical Procedure of Sampling Foods (ALINORM 69/23, Appendix VI) which covered some of the same points as the ISO document referred to in (i) above and the Government Comments thereon (CODEX/ANALYS/69/B/2).
72. In view of the need to coordinate the various approaches to the sampling problem outlined above, the Committee appointed an ad hoc Group on Sampling, consisting of Dr. Young (Chairman), Dr. Agthe, Dr. Smith, Mr. Zaboklicki and Mr. Zoltán to meet during the Session. The report of the Group, as amended by the Committee upon acceptance, appears as Appendix VI. The report describes how the Technical Procedure for Sampling Foods will be used in the development of the notes for the ISO document, on the layout of sampling methods and suggests the terms of reference and responsibilities of a proposed FAO/WHO consultant. In accepting the report, the Committee strongly recommended that such a consultant be appointed.
73. The Committee considered the problem of the status of the sampling provisions at present included in Codex Standards. A further ad hoc Group consisting of Dr. Horwitz (Chairman), Dr. Smith, Dr. Woidich, Dr. Young and Mr. Zaboklicki was appointed to consider this problem during the session; the report of this Group was accepted by the Committee and is reproduced as Appendix VII. The ad hoc Group

proposed that the Sampling Plans would be suitable only for production control and could be included in Codex Standards as a guide to manufacturers. It was pointed out that the Sampling Plans were included in a number of processed fruit and vegetables standards and had been considered as a mandatory provision. It was suggested by the delegation of Canada that governments in their acceptance might indicate that they accept the Sampling Plans as a guideline for production control rather than as a mandatory part of the standard.

74. Regarding the Sampling Plans in standards at an early stage, these should be referred back to the appropriate Codex Committee with the suggestion that after considering the points raised in paragraph 3 of Appendix VII they request that appropriate plans be drawn up for their purposes. The Committee agreed that this procedure should apply to the sampling for canned shrimps and prawns at Step 6 (ALINORM 70/18, Appendix IV) and Raisins at Step 5 (ALINORM 70/20, Appendix VIII). It was recognized that the latter might be a special case, since it dealt with bulk sampling. It was also recognized that special consideration might apply in the case of expensive commodities such as quick frozen gutted pacific salmon at Step 8 (ALINORM 70/18, Appendix II), the sampling plan for which was endorsed by the Committee on the above basis with the understanding that it should be revised in the future on a more general basis.

75. Since plans based on consumer acceptance criteria are missing from Codex Standards, the Committee agreed that this could be perhaps one of the tasks of the FAO/WHO consultant as indicated in the terms of reference proposed for its assignment (See Appendix VI para 3.2.v.)

PART X

STANDARD LAYOUT FOR CODEX METHODS OF ANALYSIS

76. At its last session the Committee had agreed to a Standard Layout for the presentation of Codex methods of analysis (ALINORM 69/23, Appendix VII). Governments had been requested to comment on the layout and these comments were given in document CODEX/ANALYS/69/B/4. In addition to the above documents, the Committee also had before it the AOAC Style Manual. The Committee agreed to amend the layout in the light of comments received from governments and to accept the nomenclature of Chemical Abstracts in addition to IUPAC nomenclature, noting that these symbols of both organizations are becoming more and more similar. In this connection it was pointed out that the Commission had adopted the units of the Système International for all Codex Standards. The delegation of the United Kingdom agreed to assist in preparing the final text, revised in the light of comments received. The Committee accepted the standard layout and requested the Secretariat to take steps to publish it.

PART XI

METHODS OF ANALYSIS FOR THE RECOMMENDED EUROPEAN REGIONAL STANDARD FOR HONEY at Step 9

77. The Committee had before it the following working documents:

- (a) CODEX/ANALYS/68/10 Microscopic Examination of Honey
- (b) CODEX/ANALYS/68/11 Quantitative Determination of Sugars in Honey by Gas Chromatography
- (c) CODEX/ANALYS/68/13 UV-Spectrophotometric Determination of HMF Content in Honey
- (d) CODEX/ANALYS/69/C/4 Determination of Diastase Activity in Honey
- (e) CODEX/ANALYS/69/B/5 Synopsis of Government Comments on (a) and (b)

At its fourth session the Committee had discussed the methods in documents (a) and (b), above and agreed that they could be of application to the honey standard but that the merits of the pollen analysis needed to be evaluated and the procedure of the GLC method adequately described (ALINORM 69/23 paragraph 96). The delegation of the Netherlands had offered to make available to the Committee a description of a method for preparing soluble starch used in the determination of diastase activity (ALINORM 69/23 paragraph 9). Concerning the photometric determination of HMF content in the Regional Standard for Honey, it was understood that the method had been endorsed with the view of replacing it by a spectrophotometric method in the future (ALINORM 69/23, Appendix III, page 11 - foot note). For these reasons the Committee discussed the methods of analysis described below in connection with the honey standard in spite of the fact that it was in the process of being sent to governments for acceptance at Step 9.

(a) Pollen analysis

78. The representative of APIMONDIA introduced the subject, pointing out that the Commission Internationale de Botanique Apicole of the Union Internationale des Sciences Biologiques had been working on this subject since 1952 and had recently published quantitative methods of pollen analysis. He also mentioned that training in this field did not represent any difficulties for persons with a knowledge of systematic botany and that this method, if considered together with other methods, was indispensable for a conclusion on quality and origin of honeys.

79. The Committee noted that provisions were given in the standard for compositional criteria depending on the origin of the honey and for labelling according to floral or botanical origin or according to the geographical or topographical region where the honey was produced. The Committee was of the opinion that pollen analysis, while useful, was not sufficiently developed to be suitable as a referee method of analysis. In particular, it considered that the Scope Section of the method should be very detailed, stressing the capabilities and limitations of the technique. The Committee recognized that the documentation on European honeys was extensive but that a comprehensive pollen atlas was necessary and that documentation was less complete for other parts of the world. The Committee also considered that, for international trade, a world atlas of pollens contained in honeys should be available before the amendment of the standard could be considered.

80. The representative of APIMONDIA agreed to consider these points and to provide delegations with their method. The Committee agreed to have this question on the agenda of its Sixth session, but it was pointed out that the publication of this atlas needed fundamental research which would probably take a considerable time.

(b) Determination of sugars by gas chromatography

81. The Committee noted that this selective and quantitative method of determining the sugars present in honeys gave results comparable to the enzymatic determination of sugars. It also considered that a less precise method, such as thin-layer chromatography might be sufficient for such a determination. The Secretariat was requested to ask the delegation of Austria for such a method and to provide the Committee with it. The representative of APIMONDIA agreed to consider the various methods discussed.

(c) Ultra-violet spectrophotometric determination of HMF

82. The Committee agreed that this method had undergone adequate collaborative studies and should be recommended in replacement of the method contained in the present standard. It further agreed to request the Commission to set in motion a procedure for the amendment of the standard as stated above.

(d) Diastase activity of honey

83. The delegation of the Netherlands pointed out that the method at present included in the Recommended European Regional Standard for Honey (CAC/R§ 12-1969) was not fully satisfactory and that the new proposed method presented to the Committee was the official BENELUX method. The Committee noted that directions for the preparation of the soluble starch, including determination of its moisture content and blue value, were given in this method. The Committee therefore agreed that the establishment of an international reference centre for the supply of such a reagent (as agreed at the fourth session, ALINORM 69/23 paragraph 9) was no longer necessary. The Committee agreed to send the BENELUX method (with appropriate translations) to governments for comments.

PART XII

METHODS OF ANALYSIS IN THE STANDARD FOR NATURAL MINERAL WATERS at Step 8

84. The Coordinating Committee at its Seventh Session made available references to a number of publications dealing with the analysis of natural mineral waters and requested the Codex Committee on Methods of Analysis and Sampling to examine these methods. The Committee discussed the working paper (CODEX/ANALYS/69/A/5) prepared by the FAO Secretariat dealing with the various criteria of the standard on natural mineral waters which may require suitable methods of analysis for their verification. In discussion the delegation of Switzerland pointed out that the direct method of determination of mineral content by drying or ashing was not suitable for the determination of the actual mineral content and proposed methods for the determination of individual or total cations and anions.

85. The delegation of the United Kingdom drew the Committee's attention to an ambiguity in the paragraph under section A of the definition of natural mineral water; the onus, placed on the competent authority in the country of origin to recognise a water (which should be specified as "potable" since the definition also covered natural waters used for baths) as a "natural mineral water", could be taken to refer to either sub-paragraph (i) or (ii) of the Definition. The Committee requested clarification

○ on this point.

86. The Committee noted that the forthcoming publication of Vol. VIII of the German Handbuch der Lebensmittelchemie contained appropriate methods of analysis for mineral waters but did not come to any decision regarding a choice of methods and referred the matter to the Coordinating Committee for Europe with a request that methods be proposed. The delegation of Switzerland agreed to prepare a working document for this purpose.

PART XIII

DETERMINATION OF SODIUM CONTENT OF DIETARY FOODS WITH LOW SODIUM CONTENT, at Step 5

87. The Committee had before it document CCDF/69/6 in which two methods for the determination of sodium, based on flame photometry, were proposed by the FAO Secretariat and agreed to by the Codex Committee on Foods for Special Dietary Uses. The Committee considered that one of these methods, that of the French Biscuit Manufacturers' Association,^{1/} had been especially drafted for foods with very low sodium content and was at present under consideration for adoption by the Association des Fabricants des Aliments Diététiques de la CEE (IDACE). The delegation of the United States requested that the results of collaborative studies on this method should be made available to the Committee. The Austrian delegation said that atomic absorption spectrophotometric methods should also be considered. The Committee agreed to send document CCDF/69/6 to governments for comments with the understanding that the IDACE method would be made available in the working languages of the Commission.

PART XIV

UNIFORMITY OF METHODS OF SENSORY ANALYSIS

88. The Committee received a verbal report from the delegation of Poland, who indicated that only a few comments had been received on the paper prepared for the 1968 session of this subject (MA/68/2). A number of delegations pointed out that their countries were active in the field of sensory testing which they regarded as a matter of great importance. The representative of ISO stated that its Technical Committee 34 was also working in this field and that a liaison between that Committee and the Codex Committee on Methods of Analysis and Sampling was desirable. On a request of the delegation of Australia the representative of the Federal Republic of Germany undertook to prepare a list of references to publications dealing with sensory analysis.

89. In reply to a question by the Secretariat, the representative of ISO stated that while the sensory methods would be generally applicable to the relevant criteria in Codex Standards, they were not always detailed enough for the purpose of modern sensory testing methods.

90. The Committee expressed its thanks to the delegation of Poland for the above paper and agreed to await the results of work done by ISO before taking further action.

○ ^{1/} Bulletin d'Information Technique, CTU.

PART XV

METHODS OF ANALYSIS OF GENERAL APPLICATION TO THE CODEX ALIMENTARIUS

91. The delegation of Poland had prepared a paper on the above topic (MA/68/1) which had been sent to governments for comments. The delegation of Poland pointed out that very few comments had been received. It was agreed that, for the time being, the items relating to microbiological tests (5) and pesticide residues (7.11) in this paper should be put in square brackets. The Committee agreed that the above paper contained a useful schematic representation of the type of general methods which the Codex Alimentarius (section on Methods of Analysis) would eventually have to contain. It was agreed that a further request to comment should be made to governments, with a final date of 1 August 1970; and that the paper prepared by Poland should be re-examined at the Sixth session in the light of all the comments received.

PART XVI

METHODS OF ANALYSIS FOR PRESERVATIVES AND ANTIOXIDANTS IN FOOD at Step 4

92. The Committee had before it papers CODEX/ANALYS/67/8 and 10 originally prepared by the delegation of the Netherlands and papers MA/68/11 and 12 as revised in 1969 and CODEX/ANALYS/69/B/6 containing government comments. Because of time limitation the Committee was again unable to discuss these papers in detail. It was unanimously agreed that methods of analysis for additives in food was an important task of the Committee, but that this had been put aside in the past because of the need to endorse methods for other criteria in Codex Standards proposed by Codex Commodity Committees. The Committee agreed that methods for food additives needed urgent attention since there were food additive provisions in Codex standards at advanced steps without an indication of the analytical method to be used.

93. In addition to the additive provision in Codex standards the Committee agreed that it was equally important to elaborate detection methods for food additives, which have been found unacceptable. The representative of WHO agreed to make available a list of such additives. The delegation of the Netherlands undertook to prepare a further paper for the next Session, including detection methods for non-permitted antioxidants and preservatives (as drawn up by WHO) which might be used in foods.

94. The Committee decided that the methods contained in CODEX/ANALYS/67/8 and 10 should not be advanced to Step 5.

PART XVII

METHODS FOR THE DETECTION AND IDENTIFICATION OF COLOURS IN FOOD

95. The Committee had before it paper (CODEX/ANALYS/68/9) prepared by the delegation of the United Kingdom and a circular letter issued by the Secretariat (CL 1969/20), together with comments from governments (CODEX/ANALYS/69/B/7 with an addendum and other comments summarized by the United Kingdom delegation). The delegation of the

United Kingdom stated that the method contained in the paper, which was based on paper chromatography, detected both permitted and non-permitted water-soluble food colours. It was pointed out that it was also necessary in some cases to provide quantitative procedures for food colours for which there were limits in Codex standards, although some of these may be only limit tests.

96. The Committee agreed that the methods contained in CODEX/ANALYS/68/9 should not be advanced to Step 5 but that the present paper be revised in the light of recent developments in this field, including thin layer chromatography. The Netherlands' delegation referred to the ECE method. The delegation of the United Kingdom agreed to prepare a revised document for the Sixth session of the Committee and the delegations of Austria, the Netherlands and the U.S.A. agreed to provide the United Kingdom delegation with information on these methods.

PART XVIII PROCEDURAL MATTERS

FUTURE WORK

97. The Committee was in agreement with a statement of the delegation of the United Kingdom that consideration of methods of analysis for food additives should be regarded as being of high priority and should therefore be placed at the beginning of the Agenda for the Sixth session of this Committee. It was agreed that preservatives, antioxidants, colours, artificial sweetening agents and metallic contaminants should be so considered and that any papers prepared for these subjects should be submitted to the Secretariat by 1 August 1970.

98. The United Kingdom delegation stressed the importance of considering general methods for the determination of metallic contaminants, applicable to all commodities. The delegation of Canada supported this and agreed to prepare a working document for the next session; the United Kingdom delegation offered assistance for this. It was also pointed out that the determination of solvent residues and any residues arising from impurities in solvents should be considered in the future by this Committee. The Committee agreed that no other work should be undertaken in view of the already heavy work load facing it.

OTHER BUSINESS

99. A number of delegations also stressed the importance of receiving documents in good time before the session so that they can be submitted to appropriate experts for their opinion. (See also para 100)

DATE AND PLACE OF NEXT SESSION

100. In order to facilitate the early distribution of working documents, particularly those arising from sessions of Commodity Committees which take place immediately prior to the Codex Committee on Methods of Analysis and Sampling, the Committee was of the opinion that the Sixth session should take place not earlier than two months after the last meeting of Commodity Committees which have matters to refer to this Committee. The last week in January 1971 was suggested but it was pointed out by the Secretariat that this was subject to confirmation by the Commission and that with the proposed arrangement certain difficulties might be experienced in the timely distribution of the report of the Sixth Session of the Committee to the participants of the Eight Session of the Commission.

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LIST OF DOCUMENTS

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| CODEX/ANALYS/69/A/1 Rev. | - Revised Provisional List of Documents |
| CODEX/ANALYS/69/A/2 Rev. | - Revised Provisional Agenda |
| CODEX/ANALYS/69/A/3 | - Methods of Analysis for Fats and Oils |
| CODEX/ANALYS/69/A/4 | - Methods of Analysis for Processed Fruits and Vegetables |
| CODEX/ANALYS/69/A/5 | - Methods of Analysis for Natural Mineral Waters |
| CODEX/ANALYS/69/A/6 | - Methods of Analysis for Edible Fungi |
| CODEX/ANALYS/69/A/7 | - Methods of Analysis for Fish and Fishery Products |
| CODEX/ANALYS/69/A/8 | - Minutes of the Meeting of the Drafting Group on Methods of Analysis of Fruit Juices (28-29 Nov. 1969) |
| CODEX/ANALYS/69/B/1 | - Government comments on Methods of Analysis and Sampling for Fruit Juices |
| CODEX/ANALYS/69/B/1 Add. 1 | |
| CODEX/ANALYS/69/B/2 | - Synopsis of Government comments on the Technical Procedure for Sampling |
| CODEX/ANALYS/69/B/3 | - Synopsis of Government comments on the Sampling Plans for prepackaged foods - and, extracts from the Reports of the Commodity Committee |
| CODEX/ANALYS/69/B/3 Add. 1 | |
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| CODEX/ANALYS/69/B/4 | - Synopsis of Government comments on the Standard layout for a Standard method of analysis |
| CODEX/ANALYS/69/B/4 Add. 1 | |
| CODEX/ANALYS/69/B/5 | - Synopsis of Government comments on Methods of Analysis for Honey |
| CODEX/ANALYS/69/B/5 Add. 1 | |
| CODEX/ANALYS/69/B/6 | - Synopsis of Government comments on methods for the detection and identification of preservatives and antioxidants |
| CODEX/ANALYS/69/B/7 | - Synopsis of Government comments on the methods for the detection and identification of colours added to food |
| CODEX/ANALYS/69/B/7 Add. 1
and Summary of Comments by
the U.K. Delegation (not numbered) | |
| CODEX/ANALYS/69/C/1 | - Determination of mineral impurities in canned strawberries (Methods proposed by the United Kingdom) |
| CODEX/ANALYS/69/C/4 | - Determination of diastase activity in honey (paper prepared by the Netherlands) |
| CODEX/ANALYS/69/C/5 | - Collaborative study on the determination of SO ₂ in Sugars (prepared by the Netherlands) |
| CODEX/ANALYS/69/C/5 Add. 1 | - Monier-Williams Method (Official Method of the ICUMSA) |
| ALINORM 68/23 | - Report of the Third Session of the Codex Committee on Methods of Analysis and Sampling, Berlin, 24-27 October 1967 |
| ALINORM 69/6 | - Report of the Sixth Session of the Codex Committee for Europe, Vienna, 4-8 November 1968 |

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- ALINORM 69/11 - Report of the Fifth Session of the Codex Committee on Fats and Oils, London, 16-20 September 1968
- ALINORM 69/14 - Report of the Fifth Session of the Joint ECE/Codex Alimentarius Group of Experts on Standardization of Fruit Juices, Rome, 25-29 March 1968
- ALINORM 69/18 - Report of the Third Session of the Codex Committee on Fish and Fishery Products, Bergen, 7-11 October 1968
- ALINORM 69/20 - Report of the Fifth Session of the Codex Committee on Processed Fruit and Vegetables, Washington, 13-17 May 1968
- ALINORM 69/23 - Report of the Fourth Session of the Codex Committee on Methods of Analysis and Sampling Berlin, 11-15 November 1968
- ALINORM 69/25 - Report of the Fourth Session of the Joint ECE/Codex Alimentarius Group of Experts on the Standardization of Quick Frozen Foods, Geneva, 2-6 September 1968
- ALINORM 69/27 - Sampling Plans for Prepackaged Foods (ex Sampling Plans for Processed Fruits and Vegetables), Revised Draft Standard prepared by U.S.A., August 1968
- ALINORM 69/67 - Report of the Sixth Session of the Codex Alimentarius Commission, Geneva, 4-14 March 1969
- ALINORM 70/3 - Report of the Fourteenth Session of the Executive Committee
- ALINORM 70/10 - Report of the Seventh Session of the Codex Committee on Cocoa and Chocolate
- ALINORM 70/11 - LIM I - Extracts of the Draft Report of the Sixth Session of the Codex Committee on Fats and Oils
- ALINORM 70/20 - Report of the Sixth Session of the Codex Committee on Processed Fruits and Vegetables, Washington, 12-16 May 1969
- SP 10/75, SP 10/101, CODEX/ANALYS/67-2 - Synopsis of Methods of Analysis for Fruit Juices prepared by the delegation of the Federal Republic of Germany
- CODEX/ANALYS/68-2(1) - Digest of comments received from Governments and Organizations on the Synopsis of Methods of Analysis for Fruit Juices

Document No.

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January 1968

CODEX/FRUJU/69/4
CL 1967-27 and
CODEX/FRUJU/69/2

QFF/MAS/1(1969)

ISO Rec. R 763

QFF/MAS/4(1969)

QFF/MAS/5(1969)

CODEX/ANALYS/68/14

CODEX/ANALYS/68/15

MA/68/2, January 1968

MA/68/11 Revised
September 1969

SP/10/101
CODEX/ANALYS/67-10

MA/68/12 Revised
September 1969

SP/10/101
CODEX/ANALYS/67-8

CODEX/ANALYS/68/10

CODEX/ANALYS/68/11

CODEX/ANALYS/68/13

- Matters arising from the Report of the Third Meeting of the Codex Committee on Methods of Analysis and Sampling, Methods of Analysis for Preservatives in Fruit Juices
- Comments by Swiss Delegation on Cadmium
- Methods of Analysis and Sampling for Fruit Juices
- Test procedure for "Ash insoluble in HCl" for quick frozen strawberries and similar products, prepared by the U.S.A. delegation
- Determination of the ash insoluble in HCl (Fruits and Vegetables products)
- Thawing procedure for Quick Frozen Fruits and Vegetables, submitted by the U.S.A. delegation
- Cooking procedure for Quick Frozen Vegetables submitted by the U.S.A. delegation
- Net weight determination of frozen fruits and vegetables, submitted by the U.S.A. delegation
- Determination of the alcohol insoluble solids content of quick frozen peas, submitted by the U.S.A. delegation
- Unification of Methods of Sensoric Analysis, proposed by Poland
- Methods of Analysis for Preservatives
- Preservatives, Report prepared by the Netherlands delegation
- Methods of Analysis for Antioxydants
- Antioxydants, Report prepared by the Netherlands delegation
- Microscopic examination of honey
- Quantitative determination of sugars in honey by gas chromatography
- U.V. - Spectrophotometric determination of HMF content in honey

Document No.

MA/68/1

CL 1969-20 and
CODEX/ANALYS/68/9

CCDF/69/6

ISO/TC 34/WG (Secr. 9) 17(*)
(Not numbered)

- General part of the section "Methods of Analysis" of the Codex Alimentarius. Proposal of Poland
- Methods for the detection and identification of colours added to food, submitted by the U.K. delegation
- Determination of sodium content in dietary foods with low sodium content, Note by the Secretariat
- Guide to the layout of sampling methods
- AOAC Style Manual

(*) Distributed at Cologne.

BASIC ALUMINA
(see para.14)

(Proposal of the Delegation of France
accepted by the Committee)

REAGENTS (see ALINORM 68/23, Appendix IV, page 11)

1. Spectrophotometrically pure cyclohexane: minimum transmittance at 220nm: 40% and minimum transmittance at 250nm: 95% by comparison with distilled water.
2. Basic alumina of known activity index
 - 2.1 Basic alumina of Brockmann activity, index I (0% H₂O) is obtained by heating for x 1/ hours, at y 1/ °C basic alumina (chromatographic quality) of particle size 30 μ to 130 μ (mean 80 μ).
 - 2.2 To 100 g of this product add z 1/ ml of distilled water to produce basic alumina of Brockmann activity index ... (II or III) 1/.
 - 2.3 Method used to check the activity index of the alumina:
 - 2.3.1 Place 30 g of the basic alumina (as obtained above) in a chromatographic column, 45 cm long and 35 mm diameter;
 - 2.3.2 Through this column, pass, under the conditions laid down in the method, a mixture of 95% virgin olive oil, having a specific extinction coefficient below 0.18 at 270nm, and of 5% groundnut oil having a specific extinction coefficient equal to or above 4 at 270nm, previously treated with decolourizing agent (absorbant earth).
 - 2.3.3 If this mixture shows a specific extinction coefficient greater than 0.11, the activity of the alumina is acceptable. Should the elution of conjugated trienes not have taken place using this alumina, an alumina at a higher level of hydration should be used.

1/ Details to be supplied later by the Delegation of France.

METHODS OF ANALYSIS FOR QUICK FROZEN FOODS

A. NET WEIGHT DETERMINATION OF FROZEN FRUITS AND VEGETABLES

The method of analysis described hereunder has been endorsed for quick frozen fruits and vegetables.

1. SCOPE

This method is applicable to all frozen fruits and vegetables.

2. DEFINITION

The net weight is the weight of the product itself, including any packing medium, as determined by the procedure described.

3. PRINCIPLE

The weight of the container including the product therein is determined. The weight of the container itself is determined. The net weight is calculated from the difference of these two weights.

4. APPARATUS

- 4.1 Balance of adequate capacity having a sensitivity of 0.25g (or 0.01 oz), for containers not in excess of 2 kg (or 5 lb).
- 4.2 Balance of adequate capacity having a sensitivity of 0.70g (or 0.025 oz), for containers in excess of 2 kg (or 5 lb).

5. PROCEDURE

- 5.1 Set balance on firm, level support and adjust indicator to zero.
- 5.2 Remove container from low temperature storage and with a towel remove frost and ice from outside of the container.
- 5.3 Weigh unopened container immediately and record as gross weight (G).
- 5.4 Open container and remove contents including product particles, frost or ice crystals, that may be adhere to the container.
- 5.5 Blot off free water with a towel and air dry empty container at room temperature.
- 5.6 Weigh the dry, empty container and record as tare weight(T).

7. CALCULATION

Calculate the net weight of the sample by means of the following formula:

$$\text{Net weight} = G - T$$

Where:

G = the gross weight found under 5.3.

T = the tare weight found under 5.6.

B. THAWING PROCEDURE FOR
QUICK FROZEN FRUITS AND VEGETABLES (*)

1. SCOPE

This procedure is generally applicable to all quick frozen fruits and vegetables. If a product requires special treatment not fully covered by the method outlined herein such thawing procedures should be outlined in the appropriate commodity standard.

2. PRINCIPLE OF THE METHOD

2.1 There are two general methods for thawing frozen fruits and vegetables -- namely a) air thawing and b) water thawing. The latter method, water thawing, is faster and in some instances more desirable than air thawing. Some products also thaw much faster than others. Frozen peas or broccoli thaw much faster than leafy greens. Consequently, no specific time can be allotted in which to accomplish adequate thawing. Through experience the analyst will learn to judge the best procedure and time requirement for each commodity.

2.2 Extreme care should be taken during the thawing process in order that the product is not damaged or exposed to abuse that will alter or degrade the true characteristics of the product. Frozen fruits are more susceptible to abuse during thawing than frozen vegetables. Light colored fruits (such as peaches and apricots) and red cherries oxidize quite readily and should be examined for color while some ice crystals still remain in the product. Some fruits show breakdown in texture or "bleed" when thawed more than necessary. Consequently, rapid thawing under controlled conditions is most desirable in preparing the product for laboratory examination.

3. APPARATUS

3.1 Water bath with temperature controls and pump to circulate water.

3.2 Electric Fan - optional for air thawing.

3.3 Plastic bags or suitable container with tight closure - optional for sub samples from large containers.

(*) This method was referred back to the Commodity Committee (See para 50 of this Report).

4. SAMPLING

Entire package or sample unit is used intact, except that in the case of bulk or industrial size containers it is not practical to use the entire container and a representative sub-sample of approximately 1 to 2 kilo is adequate for testing and organoleptic examination.

5. PROCEDURE

5.1. Frozen Fruits and Berries

5.1.1 Consumer-size Packages

Thaw in unopened containers at ambient temperature until the product is sufficiently free from ice so that individual units may be easily separated and handled. The thawing process may be hastened by placing the cartons on a table in such a manner that they are separated by air spaces and directing a stream of air from a fan on the packages.

If packed in tightly sealed containers they may be placed in a water bath at a temperature not to exceed 30° C. to expedite thawing.

5.1.2 Bulk or Industrial Packages

If the entire container is used the thawing procedure is the same as outlined in 6.1.1 for consumer-size packages.

If a representative sub-sample is used (in many cases this is the only practical way) place the sub-sample in a suitable container, which may be a plastic bag or a metal can, and thaw as prescribed above using either air or water thawing.

5.2 Frozen Vegetables

5.2.1 Air Thawing

Allow the product to thaw in unopened containers at ambient temperature. A blast of air from an electric fan may be used to hasten the process.

5.2.2 Water Thawing

5.2.2.1 In Unopened or Sealed Containers - Thaw as specified for frozen fruits, paragraph 5.1.2, by immersing the tightly sealed package in a water bath not to exceed 30° C.

5.2.2.2 By Direct Contact - Most frozen vegetables can be thawed by direct contact with water without affecting the characteristics of the end product. An exception is frozen corn, or products

containing corn, which should be air-thawed. Remove the frozen product from the package and place it directly in water at a temperature not exceeding 30° C. As soon as the product is thawed sufficiently to permit easy separation of the individual units, drain on a suitable screen (8 mesh per inch) to remove excess water and place product on tray for examination.

5.2.3 Precautions

5.2.3.1 Special Products

Certain products, such as frozen corn, should always be air thawed; or, thawed in water in sealed containers in order to prevent leaching of soluble solids or product material.

5.2.3.2 Odor and Flavor

If there is an indication of off flavors or off odors in the product when the packages are opened, do not use water thawing (product in contact with water) as a preparatory step to cooking as the off flavor may be partially removed during such thawing. Place such suspect samples in cooking receptacle while still frozen.

C. COOKING PROCEDURE FOR QUICK FROZEN VEGETABLES (*)

1. SCOPE

This method is applicable to all quick frozen vegetables.

2. PRINCIPLES

Many frozen products, particularly frozen vegetables, require preparation by cooking in order to complete product examination. In some cases cooking is required for proper evaluation of texture, tenderness or maturity. In other cases there may be a question regarding the flavor of the prepared product.

Most frozen vegetables are blanched or partially precooked during preparation for freezing. The freezing process also softens the tissues still further and consequently frozen vegetables require only from one-third to one-half as much cooking time as compared to the fresh product. Therefore care should be taken so as not to overcook the product beyond what would be expected under normal culinary preparation for serving. At the same time the cook should be sufficiently long to tenderize the product beyond a raw or uncooked state. (**)

(*) This method was referred back to the Commodity Committee (see para 50 of this Report).

(**) See para 50 of this Report.

3. SAMPLING

Ordinarily those containers used for testing other product characteristics can be used for cooking. A separate set of samples for cooking purposes only is not generally taken. With this in mind, complete testing can be performed by careful segregation and planning during the process of product evaluation.

In the case of bulk or institutional type containers, sufficient product is available so that a portion of each container can be allocated for visual examination and chemical or physical tests, and a separate portion (frozen state) for cooking.

In the case of retail size packages (for example, 500 grams or less) all of the container may require thawing for product examination and testing. In such instances the packages may be partially or completely air thawed and checked for suspect off-odors and flavors. Portions of suspect samples should be cooked for further odor and flavor evaluation. If the odor of the thawed product is normal, one can proceed with product examination and cook representative portions of the samples for whatever checks may be required. If the packages are very small it may be desirable to draw additional containers for cooking purposes only. However, this will depend upon the product and the feasibility of drawing a larger than normal sample.

4. COOKING PROCEDURE

4.1 Basic Steps

The following steps will assure uniformity in the cooking of frozen vegetables for test purposes:

- 4.1.1 Place approximately 225 g (8 oz) of product in a two-litre (two quart) sauce pan containing about 180 ml. of water. It is desirable to have the water boiling at the time the product is placed in the pan.
- 4.1.2 Bring rapidly to a boil and continue to heat sufficiently to maintain a "rolling boil".
- 4.1.3 Start timing the cook from the moment the water returns to a boil after the vegetable is added.
- 4.1.4 During the cooking process keep a reasonably tight fitting cover on the pan to avoid excess loss of moisture.
- 4.1.5 Continue the cooking for a period of time as specified in the directions on the package label. In the absence of such instructions follow the schedule as indicated in 5.2 for the appropriate product.
- 4.1.6 At the end of the cooking period, decant any excess water and place the cooked product on a tray.
- 4.1.7 Allow to cool sufficiently to be comfortably warm and make the required organoleptic tests.

4.2 Seasonings

In preparing frozen vegetables for home use it is customary to season the product with salt, sugar, butter, vinegar, spices, etc. However, for the purposes of laboratory testing it is recommended that seasonings be used sparingly as they may cover up undesirable flavors.

5. NOTES ON PROCEDURE

5.1 Cooking time may vary within range specified depending upon variety, maturity and size of pieces.

5.2 Recommended cooking times

<u>PRODUCT</u>	<u>TIME</u> (minutes)
Asparagus, Small and Medium Sizes	5 to 7
Asparagus, Large and Very Large Sizes	7 to 9
Green Beans	8 to 10
Wax Beans	7 to 9
Lima Beans (more mature)	15 to 18
Lima Beans (less mature)	12 to 15
Broccoli	5 to 7
Brussels Sprouts	8 to 10
Carrots	6 to 8
Cauliflower	3 to 5
Corn (whole kernel)	2 to 4
Corn-on-the-Cob	5 to 8
Leafy Greens - Turnip, Mustard & Kale Collards	20 to 25 40 to 45
Mixed Vegetables	9 to 12
Okra	8 to 10

PRODUCT

TIME
(minutes)

Peas	3 to 5
Blackeye or Field Peas	40 to 45
Peas and Carrots	7 to 10
Spinach (leaf)	3 to 5
Spinach (chopped)	2 to 4
Squash (summer)	5 to 7
Succotash (corn and lima beans)	8 to 10

D. DETERMINATION OF THE ALCOHOL INSOLUBLE SOLIDS CONTENT
OF QUICK FROZEN PEAS

The method of analysis described hereunder has been endorsed for:
Quick frozen peas.

1. SCOPE

This method is applicable to quick frozen peas.

2. DEFINITION

The alcohol insoluble solids content is defined as the percentage by mass of substances as determined by the procedure described.

3. PRINCIPLE OF THE METHOD

The alcohol insoluble solids in peas consist mainly of insoluble carbohydrates (starch) and protein. A weighed quantity of the sample is boiled with slightly diluted alcohol. The solids are washed with alcohol until the filtrate is clear. The alcohol insoluble solids are dried and weighed. The amount present is used as a guide to maturity.

4. REAGENTS

4.1 Ethanol (95% v/v) OR denatured ethanol (ethanol denatured with 5% v/v methanol)

4.2 Diluted ethanol (80% v/v) OR diluted denatured ethanol (80% v/v) (dilute 8 litres of reagent under 4.1 to 9.5 litres with H₂O)

5. APPARATUS

- 5.1 Analytical balance.
- 5.2 Beaker, 600-ml, if sample is boiled or 250-ml standard taper ground glass joint flask with reflux condenser if refluxed.
- 5.3 Buchner funnel.
- 5.4 Drying dish with lid, flat bottom.
- 5.5 Hot plates or boiling waterbath for refluxing or boiling.
- 5.6 Clamps or weights to prevent agitation of package in waterbath during thawing.
- 5.7 Desiccator with active desiccant.
- 5.8 Drying oven, well ventilated and thermostatically controlled adjusted to operate at $100 \pm 2^{\circ}\text{C}$.
- 5.9 Filter paper, Whatman No. 1 or equivalent
- 5.10 Macerator or blender (e.g. Atomix, Turmix, Waring or equivalent).
- 5.11 Plastic bag, of capacity to hold entire sample for thawing.
- 5.12 Pliceman on glass rods bent so as to facilitate cleaning flask or beaker.
- 5.13 Waterbath, with continuous flow at room temperature or regulated at room temperature for thawing.

6. PREPARATION OF TEST SAMPLE

Place frozen peas or frozen peas with sauce in plastic bag and tie off. Immerse sample in waterbath with continuous flow at room temperature or regulated at room temperature. Avoid agitation of package during thawing by using clamps or weights if necessary. When completely thawed, remove package from bath. Blot off adhering water from the plastic bag. Transfer the peas from package to a sieve the meshes of which are made by so weaving wire as to form square openings of 2.8 mm by 2.8 mm (a) (b). If sauce is present, wash with gentle spray of water at room temperature until the sauce is removed. Without shifting the peas, incline the sieve as to facilitate drainage, and drain two minutes. Wipe the bottom of the sieve. Weigh 250 g peas into blender, add 250 ml distilled water and macerate to a smooth paste. If there is less than 250 g sample, use the entire sample of peas with an equivalent quantity of distilled water and macerate to a smooth paste.

(a) Ref. ISO Recommendation R 565

(b) Such sieve can be replaced by US sieve with No. 8 standard screen (size of opening 2.38 mm).

PROCEDURE

- 7.1 Dry a filter paper in flat-bottomed dish, lid off, for 2 hours at $100 \pm 2^{\circ}\text{C}$. Cover dish, cool in a desiccator, and weigh accurately. (The filter paper should be larger than the base of the funnel and folded at the circumference to facilitate subsequent removal without loss of solids).
- 7.2 Weigh $20 \text{ g} \pm 0.010 \text{ g}$ paste into a 250-ml ground-joint flask, add 120 ml denatured ethanol or ethanol, and swirl to mix. If boiling rather than refluxing is preferred, weigh $40 \text{ g} \pm 0.010 \text{ g}$ paste into a 600-ml beaker. Add 240 ml denatured ethanol or ethanol, stir, and cover beaker.
- 7.3 Reflux on a steam or water bath for 30 minutes or bring solution in the beaker to a boil and simmer slowly for 30 minutes on a hot plate.
- 7.4 Immediately filter with suction on a Buchner funnel through the dried and weighed filter paper.
- 7.5 Decant most of the supernatant liquid through the filter paper. Wash the solids in the flask or beaker without delay with small portions of 80% denatured ethanol or ethanol until the washings are colourless, decanting through the filter paper each time. Do not allow solids to become dry during the washing. Transfer solids to the filter paper, spreading the solids evenly.
- 7.6 Remove the filter paper containing the residue from the funnel, transfer to the dish used in preparing the filter paper and dry uncovered in an air oven for 2 hours at $100 \pm 2^{\circ}\text{C}$.
- 7.7 Cover the dish, cool in a desiccator, and weigh accurately. The weight of the dry residue is the difference between the weight under 7.1 and this final weight.

8. CALCULATION AND EXPRESSION OF RESULTS

8.1 Method of calculation

Calculate the alcohol insoluble solids content of the sample by means of the following formula:

8.1.1 If 20 g sample is refluxed:

$$\text{Alcohol insoluble solids content (\%)} = 10 \underline{M}$$

Where:

$$\underline{M} = \text{the mass in g of dry residue.}$$

8.1.2 If 40 g sample is refluxed:

$$\text{Alcohol insoluble solids content (\%)} = 5 \underline{M}$$

Where:

$$\underline{M} = \text{the mass in g of the dry residue.}$$

8.2 Repeatability of results

The difference between results of duplicate determination (results obtained simultaneously or in rapid succession by the same analyst) should not exceed 0.6 g alcohol insoluble solids for 100 g of the product,

8.3 Expression of results

Report as g alcohol insoluble solids per 100 g of the product.

GUIDE TO THE LAYOUT OF CODEX SAMPLING METHODS

Note. In a later draft the following list of headings will be accompanied by more detailed indications or examples of the subject matter appropriate to each. It is not envisaged that all the headings will necessarily be required in documents dealing specifically with the sampling of particular commodities or groups of related commodities.

Title

1. Introduction
2. Scope and field of application
 - 2.1 Scope
 - 2.2 Field of application, e.g.
 - Intended purpose of the product (raw material, direct consumption, etc.)
 - Size of consignment or lot
 - Bulk or packed material
 - Means of transport or storage
 - Kind of examination (physical, chemical, sensory, bacteriological, etc.)
 - Purpose of examination (consumer protection, hygiene control, etc.)
 - Level of distribution (whole sale, retail trade, etc.)
3. Definitions
4. Administrative arrangements
 - 4.1 Sampling agent(s)
 - 4.2 Representation of parties concerned
 - 4.3 Signing and countersigning of sampling report
5. Identification of the lot and general inspection (prior to sampling as such)
6. Sampling equipment
 - 6.1 Apparatus for sampling for chemical tests
 - 6.2 Apparatus for sampling for microbiological examination
 - 6.3 (Onwards) Apparatus for sampling for other purposes

7. Sample containers
8. Principle of method of sampling
9. Procedure (for each field of application)
 - 9.1 Sampling plan
 - 9.2 Drawing of primary samples
 - 9.3 Production of composite sample (bulk sample)
(e.g. by combining of primary samples, blending and reduction to desired quantity)
 - 9.4 Production of replicate samples (for laboratory, contractual or other uses)
10. Packing, sealing and marking of samples
11. Preservation, storage and transport of samples
12. Sampling report, including:
 - Place of sampling
 - Date of sampling
 - Time of sampling
 - Names and descriptions of sampling agent(s) and witnesses
 - Identification of sampling method used, with indication of deviations, if any
 - Nature and number of units constituting the lot, together with details of markings, or reference to document giving them
 - Number of samples selected and their identification (markings, with special reference to batch numbers, etc.)
 - Destination to which samples are sent
 - Condition of packages and surroundings
 - Atmospheric conditions
 - Any other relevant information on the nature and condition of the lot, and on the conditions of sampling
13. Appendices
 - e.g. Specifications and drawings of apparatus, shown by way of example
 - Model report
 - Bibliographical references
 - Cautionary notes
 - Reference to statutory regulations

REPORT OF THE FIRST AD HOC GROUP ON SAMPLING OF THE
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

(as amended by the Committee)

1. Membership of the Group. This was as follows:

Dr. Agthe (WHO)
Dr. Smith (Canada)
Dr. Young (U.K.), Chairman
Mr. Zaboklicki (Poland)
Mr. Zoltán (Hungary)

2. Terms of Reference of the first ad hoc Group

This was understood to cover the formulation of proposals to the Codex Committee:

- (a) for future action in relation to the "Provisional Standard for the Technical Procedure of Sampling Foods" (doc. ALINORM 69/23, Appendix VI), and
- (b) for the terms of reference, or programme of work, of the consultant who, it had been suggested, should be engaged by FAO to provide assistance to the Committee and to other Codex Committees, in the elaboration of suitable standard methods of sampling and acceptance of food products.

3. Meeting of the Group

The Group met on Wednesday, 3 December 1969.

3.1 Technical Procedure for Sampling Foods

The Group considered the Provisional Standard, and noted that the subjects therein were all included in the draft "Guide to the Layout of Sampling Methods" doc. ISO/TC 34/WG 1 (Secretariat-9) 17, which the Committee had commended. It confirmed the view, therefore, that there was little point in elaborating the Provisional Codex Standard further as a separate document, if the Committee were in due course to adopt the ISO proposals; and that its contents should be used instead towards the drafting of the Notes which, in the completed Guide, were to accompany the ISO Standard Layout. The Group noted that the TC 34/WG 1 Secretariat was willing to convene a meeting of an expert panel in February 1970 to consider possible amendments to the draft layout in the light of any comments, and to start preparation of the notes in question; and it recommended that the contents of the Codex document ALINORM 69/23 Appendix VI be made available to it for use in that work, without delay.

3.2 Consultant on Sampling

The Group discussed the assistance that a consultant, appointed for this purpose, might give to the programme of work along the lines suggested by the delegate of Canada at the 1968 meeting of the Committee (see ALINORM 69/26, clause 80(g)), and proposed that the consultant's assignment should require of him the following tasks or responsibilities as a sampling consultant:

- (i) to take account, in his work, of relevant documentation available in the field of sampling;
- (ii) to explore the practical application of the proposed ISO layout for methods of sampling, and to put this to a constructive test as a means of assessing the value (adequacy) of existing Codex or ISO sampling documents;
- (iii) to suggest suitable revisions (editorial or other) of existing Codex methods of sampling in the light of these studies, bearing in mind in particular the aims of the Codex Alimentarius Commission in relation to Codex standards which should provide user protection and fair practices in international trade, rather than quality control in or immediately following the production line (which is the function of factory standards);
- (iv) to attend during his period of service and in an observer/adviser capacity, meetings (if any) of ISO/TC 34/WG 1 (or of its expert panel) and/or of Codex Commodity Committees facing sampling problems;
- (v) to prepare a number of sampling plans and of sampling cum acceptance procedures, designed for a series of typical cases, which may serve as models to Codex Commodity Committees in the elaboration of their own particular sampling and acceptance plans, although such plans must always be specially designed to take account of the individual set of criteria involved. This work would be based on the Notes in the draft ISO Guide to the Layout of Sampling Methods to be elaborated by ISO, and on the General Statement on Sampling in the field of Food (ALINORM 65/25(1)); and might usefully include plans applicable to the sampling of products for the verification of criteria intended for health protection;
- (vi) to report on the above work to the sixth session of the Codex Committee on Methods of Analysis and Sampling.

The Group considered that candidates for the post should be persons with a knowledge of food and agriculture, and of statistics, who had experience of sampling, including the development of sampling and acceptance plans. They should be able to read, and to understand well, French and English documents, and to speak and write fluently in one at least of these languages. It was suggested that the successful candidate would require one week in Rome at FAO Headquarters to survey the problem at the start of his service, followed by one or two

weeks for consultation with experts in other centres abroad associated with Codex work, as an introduction to the problems to be faced. Further similar visits, including time spent at ISO or Codex meetings in the course of the assignment, might also be required.

3.3 Preparatory work by FAO

In anticipation of the recruitment of the sampling consultant, the Group proposed that preparatory work be undertaken by FAO as follows:

- (i) collection of relevant documentation to serve as reference documents, or as working documents for the consultant, in the field of sampling;
- (ii) preparation of a list of experts, nominated by member countries, who are willing and able to receive a visit from the consultant for a discussion and to assist him in gaining useful information on local conditions relevant to his work;
- (iii) compilation of a list of all instances, to be obtained from the chairmen of Codex committees, where a sampling problem exists.

The Group expressed the hope that the consultant might be in office by 1st April 1970, to enable the full benefits of his work to be applied in advance of the ISO and Codex meetings in that year.

REPORT OF THE SECOND AD HOC GROUP CONCERNED WITH THE
STATUS OF SAMPLING PROVISIONS IN CODEX STANDARDS

1. TERMS OF REFERENCE OF THE SECOND AD HOC GROUP

- 1.1 To consider the applicability of the sampling plan for processed fruits and vegetables.
- 1.2 To consider whether the proposed sampling plan for processed fruits and vegetables can be broadened in scope to encompass certain prepackaged processed food as recommended by the Commission at its Sixth Session.
- 1.3 To indicate appropriate procedures for Commodity Committees to apply when inserting referee sampling plans for use in cases of dispute into their various commodity standards.

2. REPORT

- 2.1 The Group examined the comments of governments on the Sampling Plans for Prepackaged Foods (ALINORM 69/27 and para. 182 of ALINORM 69/67) and suggested that, in view of the fact that it was a sampling plan for production control, it could be included with the standards as a guide to manufacturers concerned with such production control.
- 2.2 Similarly, this Sampling Plan can be included with other standards for prepackaged processed food as a control over similar criteria as a guide for production control.
- 2.3 In the opinion of the Group appropriate sampling procedures for insertion into commodity standards can be developed using the ISO guide to the layout and drafting of sampling methods. In addition committees must consider carefully the purpose for which a sampling plan is needed and develop for this purpose a suitable procedure. Criteria influencing the choice of a suitable procedure might be:

Nature of the variables and
Information concerning the homogeneity of the lots.

With this information, the Committee should then make decisions as to the acceptable

- (1) Probability of accepting a bad lot
- (2) Probability of rejecting a good lot.