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ALINORM 74/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX ALIMENTARIUS COMMISSION
Tenth Session, Rome 1974

REPORT OF THE EIGHTH SESSION OF THE
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Budapest, 3-7 September 1973

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Eighth Session from 3 to 7 September 1973 in Budapest at the courtesy of the Government of Hungary. The session was presided over by Mr. A. Miklovicz, Chairman of the Hungarian Codex Committee. The session was opened by Dr. K. Karcsai, General Secretary of the Hungarian National FAO Committee, who welcomed the participants and stressed the importance of reaching agreement on appropriate methods of analysis and sampling as part of international standardization of food. The session was attended by delegates from 20 countries and 9 International Organizations. The List of Participants including officers from FAO is contained in Appendix I to this Report.

ADOPTION OF THE AGENDA

2. The Committee adopted the agenda with some re-arrangement of the order of the items, as follows:

- i. Item 4 was postponed pending the arrival of the FAO Secretariat;
- ii. Items 5, 6 and 8 were postponed until after the consideration of the statistical sampling plans for the determination of the net contents.
- iii. Item 10 was taken immediately after Item 3.

APPOINTMENT OF RAPORTEURS

3. Dr. W. Horwitz of the delegation of the USA and Mr. G. Janssens of the delegation of France agreed to act as rapporteurs and were so appointed by the Committee.

TERMS OF REFERENCE AND PROCEDURE FOR SUBMITTING METHODS OF ANALYSIS FOR CONSIDERATION BY THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING (CCMAS)

4. The Committee considered a working paper (CX/MAS 73/6) containing proposals by Denmark and the USA concerning certain procedural matters. The delegation of Denmark requested the Committee to direct its work primarily to dealing with general methods, leaving methods specific to commodities to be handled by the Commodity Committees in order to avoid delays in approval of Codex methods and possibly standards. Several delegations were not in favour of this proposal. Therefore, an ad hoc Working Group consisting of representatives from Australia, Denmark and the United States was appointed by the Committee to review this matter with instructions to avoid any radical changes in the Terms of Reference of the Committee and in the General Principles for the Establishment of Codex Methods of Analysis.

5. The delegation of the United States proposed the development of an administrative scheme to permit the orderly submission, review, and approval of methods of analysis

and sampling. The ad hoc Working Group was requested to report on this matter also. The report of the ad hoc Working Group is given as Appendix II to this Report.

6. In considering the report of the ad hoc Working Group, the following points were raised:

a. Revising adopted methods included in Codex commodity standards, or indeed replacing them with other methods may not constitute an amendment of the Codex standards concerned. This is a matter which the Commission may wish to consider in due course.

b. In the opinion of several Delegates collaborative testing carried out in different countries should be preferred to collaborative testing carried out in only one country, in order to take into account differences in reagents, standard solutions, equipment, etc. used.

c. The Committee noted that the ad hoc Working Group had not considered the question of sampling.

7. The Committee agreed with the conclusions contained in the report of the ad hoc Working Group but decided that there was no need for an absolute requirement for collaborative testing to be done at an international level, although such collaborative testing should be preferred. The report of the Working Group was adopted by the Committee with the amendment concerning the desirability but not requirement for collaborative tests to be carried out internationally. The delegation of Denmark pointed out that it accepted as reasonable the report of the present ad hoc Working Group, but indicated that the Government of Denmark may not necessarily agree with the conclusions of the Group concerning the present terms of reference of the Codex Committee on Methods of Analysis and Sampling. The Committee expressed its appreciation to the members of the ad hoc Working Group.

STATISTICAL SAMPLING PLANS FOR THE DETERMINATION OF NET CONTENTS

8. The Committee had before it documents CX/MAS 73/13 and 73/14 and had reference to documents CX/FL 72/8 and 73/12 on the subject of the statistical interpretation of the term "net contents" in relation to lot inspection. The Committee was informed of the decision of the Codex Committee on Food Labelling that, in interpreting the declaration of net content on the label, the average content should be taken as the criterion, based on the examination of an adequate sample, provided there was no unreasonable shortage in individual containers (see para 52, ALINORM 72/22 and para 75, ALINORM 74/22).

9. Since a number of sampling plans were available to the Committee differing in number of units examined per lot, simplicity, destructiveness, and number of commodity classifications, the Committee decided to set up an ad hoc Working Group consisting of the delegations of Australia, Canada, France, Hungary, the Netherlands, Switzerland, the UK and the USA. The ad hoc Working Group was requested to prepare guidelines for a sampling plan based on all the available documentation applicable to the determination of net contents in different types of food. The report of the ad hoc Working Group is given as Appendix III to this Report.

10. The Committee received a verbal report by the Chairman of the ad hoc Working Group, Mr. G.E. Anderson, who pointed out that the Group had reached agreement on certain statistical assumptions, requirements, and definitions to be adopted in order to verify compliance with a declaration of net contents but that the Group had not been able to reach agreement on certain important issues referred to in detail in the report of the ad hoc Working Group which required policy decisions. These issues would have to be considered in the light of comments from the governments and other

interested bodies as they involved administrative considerations. The Chairman of the ad hoc Working Group was further of the opinion that the documents containing a number of articles on the problems of sampling (CX/MAS 73/13 and 73/14) should, in an amended form, be distributed to interested parties. Furthermore, it was essential for the ad hoc Working Group to continue its work beyond the present session so as to be able to prepare Operating Characteristic Curves (OCC) on the basis of which the difference between the two basic statistical approaches contained in the report of the ad hoc Working Group could be more readily ascertained. The Secretariat was of the opinion that it would be desirable to issue a questionnaire requesting appropriate information from governments concerning the report of the ad hoc Working Group. In this connection the opinion was expressed that the questionnaire should be drawn up in such a way as to afford those replying to it the possibility of giving adequate information and should not be only of the "yes" or "no" type.

11. It was pointed out that the statistical approaches considered by the ad hoc Working Group applied equally to certain non-food items but that the consideration of such items was not within the scope of work of the Commission. It was agreed nevertheless that the questionnaire should draw attention to the implication for non-food items of decisions regarding foods, since these were in many countries subject to the same legislation and administrative procedures.

12. The Committee expressed its thanks to the members of the ad hoc Working Group and agreed that:

- a. the report of the ad hoc Working Group should be appended to its report;
- b. a questionnaire should be drawn up by the Chairman of the ad hoc Working Group in collaboration with the Secretariat and the other members of the ad hoc Working Group and distributed to governments;
- c. documents CX/MAS 73/13 and 73/14, with any amendments and changes as appropriate, be distributed to governments;
- d. governments be requested to send their comments on the eleven points of agreement relating to statistical approaches, assumptions, requirements, and definitions contained in the report of the ad hoc Working Group (see Appendix III);
- e. information and comments be requested on the unresolved issues contained in the report of the ad hoc Working Group (see Appendix III);
- f. the ad hoc Working Group, with Mr. G.E. Anderson acting as rapporteur, should continue its work by correspondence or other appropriate means not necessitating the holding of unscheduled Codex meetings until the next session;
- g. Codex Commodity Committees should be requested to comment on the report of the ad hoc Working Group concerning sampling plans for the determination of net contents in conjunction with the reports of the Codex Committee on Food Labelling (ALINORM 72/22 and ALINORM 74/22);
- h. Comparative Operating Characteristic Curves be drawn by the delegations of Canada, Switzerland and the USA; and
- i. the ad hoc Working Group should consider the information received from governments, Commodity Committees, and other sources and prepare a working document for the next session of this Committee.

GENERAL PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF SAMPLING

13. The Committee had before it document CX/MAS 73/3 containing a proposal by the UK for General Principles on the basis of which sampling procedures should be selected by the Committee. The Committee noted that, although the UK proposal had been distributed for comments prior to the session, no comments had been received. A number of delegations were of the opinion that it might not be timely to consider the question of General Principles for the establishment of Codex Methods of Sampling and that this matter should be taken up at a later session. The delegation of Australia once more stressed that technical methods of sampling and statistical sampling plans proposed by Codex Commodity Committees should be submitted to the Committee for consideration and endorsement (see para 13(c), Guidelines for Codex Committees, Codex Alimentarius Commission Procedural Manual, Third Edition).

14. The Secretariat was of the opinion that the concept of Codex methods of sampling would probably have to be reconsidered at some future date. The Committee requested the Secretariat to prepare a paper in collaboration with the delegation of the UK setting out all previous decisions and adopted texts relating to the definition, general principles, acceptance etc. of Codex methods of analysis and sampling. The Committee expressed its appreciation for the work done by the delegation of the UK but decided to await the conclusions of the ad hoc Working Group on sampling for the determination of net contents as revised on the basis of government replies and requested the delegation of the UK to continue as rapporteur in regard to the general principles on sampling.

STANDARD LAYOUT AND GUIDE TO THE DRAFTING OF STANDARD METHODS OF SAMPLING

15. The Committee considered the ISO working document, ISO/TC 34/WG.1 (Secretariat-20) 40 E "Standard Layout and Guide to the Drafting of a Standard Method of Sampling from a Lot" and was informed that it had not yet been finalized by ISO/TC 34 pending comments from members of the Codex.

16. During the discussion the delegation of Australia drew the Committee's attention to the fact that ISO Standard Layout and Guide made reference to the use of definitions of terms as defined in the "Draft Vocabulary of Sampling Terms and Definitions" (ISO/TC 34/WG1 (Secretariat-13) 26 bis E) prepared by ISO/TC 34. In the opinion of the delegation of Australia the definitions of ISO/TC 69 were more appropriate. The Committee noted that within ISO there were three Technical Committees dealing with definitions and terms in the field of sampling and also noted that the European Organization of Quality Control (EOQC) had published a Glossary of Terms used in Quality Control.

17. The Committee decided that before taking any action concerning the adoption of the ISO Standard Layout and Guide, ISO itself should consider the finalization of their document. In the meantime, the draft standard layout and guide would serve the purposes of this Committee. ISO was also requested to look into the matter of the definition of terms in sampling in an endeavour to coordinate efforts in this field. It was also agreed that the comments received from Poland and other countries on the ISO documents should be made available to the Committee and to ISO/TC 34.

TENTATIVE DRAFT CODE OF SAMPLING FOR INFANT FORMULA

18. The Committee had before it a paper prepared by the FAO Secretariat containing the tentative draft Code CX/MAS 73/4. In introducing the paper, the Secretariat explained that the purpose of the document was to serve as an example aimed at eventually drawing up a sampling procedure suitable for food for infants and children.

The present draft should not be considered more than an attempt to demonstrate the type of information which would have to be included in a Codex sampling procedure drawn up on the basis of ISO Standard Layout and Guide. The Secretariat further pointed out that in doing so, the document necessarily had to contain not only statistical sampling plans together with acceptance criteria but also actual physical procedures for obtaining samples.

19. The Secretariat was of the opinion that the Codex Committee on Foods for Special Dietary Uses may not be the most appropriate body to consider the actual physical procedures for obtaining samples, and that the Codex Committee on Methods of Analysis and Sampling should continue to elaborate technical procedures for drawing samples as a general document.

20. A number of delegations pointed out that some details in the tentative draft prepared by the Secretariat were not appropriate, e.g. acceptance criteria for a standard with a single limit, maximum or minimum, were not necessarily the same as for standards with both maximum and minimum limits. It was further pointed out that plans which involved high consumer risk such as those proposed by the Secretariat may not be suitable for a product such as Infant Formula or other products where public health aspects were involved. It was also pointed out that the sampling procedure would probably not apply either at the producer or at the retail level.

21. The Committee agreed that the above document should be kept for future reference and authorized the Secretariat to take steps to obtain the views of the Codex Committee on Foods for Special Dietary Uses on the need to draw up a sampling procedure for Infant Formula and to indicate the details that would be necessary to draw up an appropriate sampling plan. Such details would include, for example, the level of confidence at which a sampling plan should be carried out, any tolerances for the range of limits laid down for the nutrients provided for in the standard, and other such relevant information. The delegation of the Federal Republic of Germany offered to explore the possibility of his country preparing a paper for the next session of the Codex Committee on Foods for Special Dietary Uses.

WORK PRIORITIES IN THE FIELD OF SAMPLING

22. The Committee agreed that the elaboration of sampling plans for the determination of net contents be given first priority and that sampling procedures for Infant Formula may, pending advice from the Commodity Committee, also be considered at a future session (see para 21). It requested the Secretariat to obtain suggestions from Codex Commodity Committees regarding future work priorities in the field of sampling and prepare a paper on this subject for the next session. The consideration of General Principles for the Establishment of Codex Methods of Sampling (see paras 13-14) was also considered an item for future consideration.

MATTERS ARISING FROM REPORTS OF CODEX COMMITTEES

23. The Secretariat informed the Committee that the Codex Committee on Pesticide Residues, at its 6th session, had decided to continue work on the problem of sampling for the enforcement of pesticide residue tolerances without awaiting the outcome of the work of the Codex Committee on Methods of Analysis and Sampling. In the opinion of the Pesticide Residue Committee the tolerances for pesticide residues represented a specific and specialized field requiring particular sampling procedures (see para 16, ALINORM 72/24A). The Committee noted that any sampling procedures elaborated by the Codex Committee on Pesticide Residues would most likely be referred to this Committee for consideration. The Committee was also informed that matters arising from the Codex Committee on Food Labelling and the Executive Committee had already been dealt with under other Agenda Items.

ENDORSEMENT OF METHODS OF ANALYSIS

Standard for Infant Formula and Standards for Foods for Infants and Children

24. Moisture (loss on drying) - The delegation of the Netherlands pointed out that the drying time at the temperature specified in the AOAC method was too long for acid infant food and that similar difficulties would be expected with products containing added crystalline lactose. The Committee decided to maintain the temporary endorsement of the AOAC method (AOAC XI,7.003), pending the results of collaborative tests.
25. Ash - The delegation of the Netherlands drew the Committee's attention to the method of the USA Infant Formula Council and pointed out that ashing above 550°C led to lower values for ash. The delegation of the USA pointed out that the methods of the Infant Formula Council were nearly all AOAC methods but that some of the methods of the Council deviated slightly from the corresponding AOAC methods and that it was up to the Infant Formula Council to justify such deviations. Although the AOAC method for ash determination referred to animal feed, this was only a presentational question and the AOAC method was a general one applicable to any type of food. The Committee was informed that results of collaborative studies had not been submitted to the Codex Committee on Foods for Special Dietary Uses which was scheduled to be held from 11 to 15 February 1974. The Committee decided to temporarily endorse the AOAC, XI, 7.010 method awaiting information from governments concerning its applicability to foods for infants and children.
26. Crude fat - The Committee noted that in reply to its request, the Codex Committee on Foods for Special Dietary Uses had given a definition of "crude fat" (see para 6, ALINORM 74/26). The delegation of the Netherlands was of the opinion that three methods were needed to cover the various types of foods involved, i.e. those containing milk and sugar, cereals, and complete foods containing meat and vegetables. The Committee agreed that it was not in a position to select appropriate methods for the various types of foods for infants and children and accepted the offer of the Netherlands to organize a collaborative study in which the delegations of at least the USA, Australia, UK and the Federal Republic of Germany would participate. Three methods were to be used in the international collaborative tests and applied to four types of products.
27. The delegation of the Netherlands indicated that it would request comments from the collaborators on the collaborative design before organizing the actual tests. The Committee wished to record its satisfaction as regards the proposed first international collaboration organized by this Committee to establish the reliability of proposed Codex methods and expressed the hope that similar collaborative efforts would be made in connection with other methods which lack such studies. It expressed its thanks to Dr. W. Horwitz for his efforts aimed at achieving international collaboration in this field.
28. Crude Fibre - The Committee was informed that the joint AOAC/ISO method was still in process of finalization and that, when agreed, the method would be subjected to collaborative tests. The Committee decided to await the results of the joint AOAC/ISO collaborative tests.
29. Crude Protein - The Committee was informed that ISO was circulating a joint AOAC/ISO text and that a report would probably be available next year. The delegation of Australia pointed out that there was need to look for new catalysts such as the titanium oxide/copper described by P.C. Williams (1973) in J. Science Food Agriculture, as mercury was being phased out. The delegation of US informed the Committee that many organizations were looking into this matter.
30. Conversion factors for available calories and nitrogen - The Committee noted that the Commodity Committee had adopted the conversion factors contained in the report of a Joint FAO/WHO Ad Hoc Expert Committee on Energy and Protein Requirements (Annexes 2 and 3, WHO Techn.Rep.Ser.No.522).

31. Available carbohydrates - The Committee noted the conclusions of the Commodity Committee and decided to await proposals for specific methods.
32. Sodium/potassium - The Committee noted the method prepared by the US (see Appendix IV to this Report) and requested governments to comment on this revised method.
33. Vitamin E - The Committee did not change the previous temporary endorsement (see para 46, ALINORM 72/23).
34. Linoleic acid - The Committee noted that the Commodity Committee had specified this compound as cis,cis,9:12-octadecadienoic acid (see paras 11-12, ALINORM 74/26) and that the Codex Committee on Fats and Oils would consider the elaboration of a GLC method for fatty acids. The Committee requested the UK to continue work in this field.
35. Vitamin K - The Committee temporarily endorsed the chick-assay method of Schönheyder and noted that GLC and TLC methods were being developed to measure Vitamin K₁, the only form of this vitamin permitted in Infant Formula. The Committee agreed that in due course the chick-assay method would be either reconfirmed or replaced by an appropriate GLC or TLC method.
36. Identification of Ingredients - The Committee agreed with the conclusions of the Commodity Committee that there was no need to establish methods for the determination of optional ingredients unless these were specified in the standard.
37. Choline - The Committee was informed that the EEC had not yet elaborated a method for the determination of choline in food but that a method had been considered for animal feed.
38. Chloride - The Committee had before it a general method submitted by the US delegation to the Codex Committee on Foods for Special Dietary Uses (see Appendix I, CX/MAS 73/7). The US delegation informed the Committee that collaborative studies had indicated that the method was generally applicable to food with only slight modification to the section dealing with the preparation of sample and that the present text of the method was suitable for the determination of chloride in the Infant Formula. The Committee also had before it a method submitted by the French delegation (see Appendix II, CX/MAS 73/7). The Committee agreed that the US method containing the revised preparation of sample and the method proposed by the French should be circulated to the governments for comments together with any supporting data from collaborative tests.
39. Milk Solids and Cocoa Solids - The Committee was informed that the Codex Committee on Cocoa Products and Chocolate is considering methods for the determination of milk and cocoa solids (see Appendix IV, ALINORM 74/10) and that these methods would be placed before this Committee in due course.
40. Quality of Protein - The Committee noted that the Codex Committee on Foods for Special Dietary Uses was considering this question but that the FAO/WHO Protein Advisory Group, which met in December 1972, did not discuss methods for the determination of protein quality in foods for infants and children as it had already proposed methods for determining protein value (PAG Guideline No. 6). The Committee postponed further consideration of this question.
41. Residues of hormonal and antibiotic substances - The representative of the EEC informed the Committee that no EEC methods were yet available to determine these residues. The delegation of Australia pointed out that USDA methods were used in that country but that these methods required large samples and that the clean-up procedures were very difficult. He further informed the Committee that hormones and antibiotics in infant foods were extremely difficult to determine. The delegation of the USA indicated that the USDA methods were still under investigation and that the results of collaborative tests would be made available in due course. He requested

the representative of the EEC to furnish supporting data when publishing official EEC methods of analysis. The Committee decided to await specific recommendations for levels of particular hormones and antibiotics in food and any recommendation for methods to determine them, before reconsidering the above question.

42. Iodine - Although the method which the Committee had endorsed at its previous session was not under consideration, the Committee noted the opinion of the delegation of the Federal Republic of Germany that the method was found by analysts in that country not to be applicable to the quantity specified in the standard for Infant Formula. The delegation of the USA agreed to bring this matter to the attention of the AOAC.

Fruits and Vegetables and Quick Frozen Foods

43. Moisture in Raisins - The Committee considered a method proposed by the Commodity Committee as an alternative to the AOAC method previously endorsed. The delegation of the UK pointed out that methods of the type contained in Annex 1, Appendix VII to ALINORM 72/20A did not give satisfactory results as, among other things, these depended on the degree of packing of the product. The delegation of the USA informed the Committee that the method had been found satisfactory on the basis of collaborative studies. The Committee endorsed the alternative method proposed by the Commodity Committee.

44. Mineral Impurities in Raisins - The Committee endorsed the method proposed by the Commodity Committee contained in Annex II, Appendix VII to ALINORM 72/20A but decided that the "household detergent" should be specified. The delegation of Australia undertook to provide the Secretariat with the relevant details concerning the detergent to be used.

45. Sulphur Dioxide in Raisins - The delegation of the Netherlands, supported by the delegation of Austria, expressed its preference for the Tanner method which had been tested on raisins. The delegation of the USA was of the opinion that, while the Tanner method was acceptable as it was generally applicable to food, the AOAC method was a more rapid one. The delegation of the USA was further of the opinion that details of the collaborative tests carried out in the Netherlands on the Tanner method should be made available to the Committee. The Committee endorsed the AOAC method proposed by the Commodity Committee and was of the opinion that the Tanner method, which is a general method and therefore preferable, should be considered for endorsement as an alternative to the AOAC method if, as a result of collaborative tests, involving direct comparison of the two methods, it is shown to be equivalent to the AOAC method.

46. Mineral Oil in Raisins - The Committee considered the method proposed by the Commodity Committee (see Annex III to Appendix VII, ALINORM 72/20A). The delegation of the Netherlands was of the opinion that a blank determination should be included in the text to eliminate any possible errors arising from residues resulting from the evaporation of 400 ml petroleum ether. The delegation of the USA was of the opinion that the specification for the analytical grade petroleum ether of the American Chemical Society prevented any significant errors which could arise and that, therefore, there was no absolute need for a blank test. The question of the separation of mineral oil and vegetable fats using column chromatography was discussed. On the one hand it was argued that the presence of carbonyl groups in the IR spectrum would be sufficient to indicate whether or not separation was complete. On the other hand it was considered necessary to carry out a saponification step. The Committee temporarily endorsed the method and requested the Secretariat to amend the method to require a blank determination to be carried out. The delegations of the USA, Australia and the Netherlands agreed to carry out a collaborative test to see whether there was a need for a saponification step and to report back to the Committee.

47. Sorbitol in Raisins - The Committee considered the method proposed by the Commodity Committee contained in Annex IV of Appendix VII to ALINORM 72/20A. The delegation of the Netherlands was of the opinion that the method proposed by the Commodity Committee was rather cumbersome and that simpler enzymatic methods were available. The Committee endorsed the method proposed by the Commodity Committee and agreed that the enzymatic tests could be considered at a later stage.

48. Mineral Impurities in Canned Strawberries - The Committee endorsed the method proposed by the Commodity Committee (see Appendix XIV, ALINORM 72/20A).

49. Mineral Impurities in Quick Frozen Strawberries and Raspberries - The Committee endorsed the method proposed by the Commodity Committee (see Appendix II, ALINORM 74/25) but noted that this method differed from that under paragraph 48 above in that it did not include treatment by HCl. Although the Committee recognized that these methods were closely linked to the "mineral impurity" provision in the respective standards, it requested the Commodity Committees concerned to propose, as far as possible, one method for the determination of similar or identical provisions in standards and to consider whether treatment by HCl was necessary.

50. Berry Ingredient in Quick Frozen Raspberries - The Committee endorsed the method proposed by the Commodity Committee contained in Appendix III to ALINORM 74/25 noting that the method, in fact, defined "drained berry ingredient". The Secretariat was requested to clear up the inconsistency in sub-sections 3.3.5.1 and 8.3.2 concerning the weight of the sample unit to be taken.

51. Total Soluble Solids in Quick Frozen Raspberries - The Committee endorsed the Codex Alimentarius Commission Recommended Method CAC/RM 43-1971 proposed by the Commodity Committee.

52. Drained Weight of Table Olives - The Committee endorsed the Codex Alimentarius Commission Recommended Method CAC/RM 36-1970 proposed by the Joint Codex/IOOC Meeting on the standardization of Table Olives.

53. Salt in Table Olives - The Committee endorsed the potentiometric titration method proposed by the Joint Codex/IOOC Meeting (J.AOAC, No. 2, March 1971, 32.A01 to 32.A05). As regards the alternative method proposed by the Commodity Committee (see page 2, document CX/MAS 73/10) the Committee invited the Commodity Committee to furnish evidence of equivalence before consideration can be given to the endorsement of the alternative method.

54. Acidity and pH of Table Olives - The Committee endorsed the method proposed by the Joint Codex/IOOC Meeting noting that the standard for table olives provided for a number of additives which, being acids, would affect the interpretation of results following the determination of both pH and acidity.

Cocoa Products and Chocolate

55. The Committee had before it document CX/MAS 73/14 containing the results of a collaborative study on a Joint AOAC/OICC method for the determination of ash in cocoa products. The Committee was of the opinion that the Joint AOAC/OICC collaborative effort was an excellent example of cooperation between two international organizations and endorsed the Joint AOAC/OICC method. The delegation of Australia wished to be placed on record that, in conformity with the Commission's Guidelines relating to the elaboration of Codex methods of analysis and sampling, sampling procedures proposed by Codex Commodity Committees for inclusion in Codex standards had to be submitted to this Committee for endorsement.

Edible Fungi and Fungus Products

56. The Committee had before it a working paper, CX/MAS 73/8, containing methods of analysis requiring consideration by the Committee and comments from governments thereon.

57. Mineral Impurities in Fungi in Oil - The Committee endorsed the ISO method R.763 as amended in CL 1970/5, para B. It noted the comments received from the Federal Republic of Germany that an ashing temperature not above 550°C should be prescribed. It also noted the opinion of the UK that no published methods for mineral impurities for fungi in oil existed and that countries familiar with this product should provide information so that suitable methods can be developed.
58. Salt in Fungi in Oil - The Committee endorsed the J.AOAC method 32.A01 to 32.A05, March 1971 noting the comments of the Federal Republic of Germany, that in the case of fungi in oil, the oil should be carefully removed since its presence could influence the potentiometric titration. The UK made the same remarks as in para 57 above. The US pointed out the applicability of the endorsed method to such high fat products as butter and margarine.
59. Water in Dried Fungi, Freeze-Dried Fungi and Dried Fungus Shi-ta-ke - The Committee noted that the new AOAC method (AOAC,1970,31.006) was the same as the previously endorsed AOAC,1965.29.005 method and agreed that no action need to be taken on this matter.
60. Lactic and/or Citric Acids in Sterilized Fungi - The delegation of the Netherlands was of the opinion that the proposed AOAC method (AOAC,1970,22.058), which measured total titratable acidity, was not specific enough for the verification of this provision and proposed that an enzymatic method should be used. Other delegations were of the opinion that only final pH was necessary as an accessory for the sterilization process. The Secretariat was requested to bring this matter to the attention of the Coordinating Committee for Europe so that appropriate methods could be developed, if necessary.
61. Concentrated Fruit Juices - The Committee had before it a working paper, CX/MAS 73/9, listing those methods which needed consideration or elaboration by this Committee as well as a summary of methods already endorsed or temporarily endorsed in Step 8 and Step 9 standards for single strength fruit juices. The Committee was informed that the International Fruit Juice Union would meet at the end of September in Vienna and consider various methods for fruit juices including tests for fermentability (see Appendix II, Method of Pilnik and Piek-Faddegon, CX/MAS 73/9). The Committee decided to consider these methods after the meeting of IFJU. However, it agreed to consider the applicability of methods already endorsed for single strength fruit juices to concentrates. For this purpose a small ad hoc Working Group was set up consisting of the delegations of Austria, Switzerland, the USA and the representative of the IFJU.
62. The Committee received a verbal report from Mr. H.U. Pfister, Chairman of the ad hoc Working Group. He informed the Committee that the Working Group recommended that all methods endorsed for single strength juices were applicable to concentrates except those methods which determined provisions applicable to the concentrated juices as such, e.g. refractometric determination of soluble solids, etc. The Group further recommended that the standards for concentrated fruit juices should contain a suitable reference that the methods of analysis in question applied to the fruit juice concentrates after reconstitution to the appropriate Brix value. Thereafter the appropriate methods should either be inserted by reference to the single strength juices or quoted in their entirety.
63. The representative of IFJU indicated that his organization, at its next meeting in September in Vienna, would study a method based on isotopes to distinguish between single strength fruit juices and reconstituted fruit juices. The delegation of France proposed that an interlaboratory test be carried out to verify such methods.
64. The Committee agreed with the conclusions of the ad hoc Working Group and also agreed that it may be useful for a method such as proposed by the representative of IFJU and the delegation of France to be developed.

OTHER BUSINESS

65. The Committee was informed that the US and the Netherlands had amended their previous proposal for a method to determine preservatives in food, in keeping with the suggestions and recommendations made by this Committee during its seventh session and to include the findings of Ir. Gosselé, the delegate from Belgium. In addition, a second method (reported by Clement *et al.*, *Z.Anal.Chem.* 248,182,1969) had been examined by the Netherlands and found to be applicable. These two methods would be circulated to the participating countries (Austria, Belgium, the Netherlands, the UK and the USA) for their comments and a joint AOAC-CCMAS collaborative study would be conducted to test and compare these two methods. The Committee agreed to examine these methods at a future session.

66. The delegation of the Federal Republic of Germany agreed with the request of the Committee to update its previous paper on the work of international organizations dealing with methods of analysis. The representative of IUPAC offered to cooperate in this task. He indicated that IUPAC intended to organize a working group of representatives of organizations to harmonize specifications for food additives and pertinent test methods. The Secretariat was requested to enquire into the conclusions and report of the meeting convened in March 1973 in Paris under the auspices of FAO on work done by international organizations concerning standardization in all fields of agriculture, and to make available any information to the delegation of the Federal Republic of Germany.

67. The Secretariat was of the opinion that it may be desirable to draw up a protocol for conducting international collaborative tests. The Committee was informed that IUPAC intended to organize a symposium on the harmonization of collaborative studies and requested the representative of that Organization to prepare a report for the next session of the Committee.

FUTURE WORK

68. The Committee agreed that the following items should be considered at its next and subsequent sessions:

- a. General methods for preservatives in food;
- b. General methods for metallic contaminants - the delegation of Canada was requested to update its previous paper;
- c. Sampling plan for the determination of net contents;
- d. Sampling procedure for foods for infants and children;
- e. Other sampling procedures proposed by Commodity Committees; and
- f. Endorsement of methods of analysis and sampling proposed by Codex Commodity Committees.

DATE AND PLACE OF NEXT SESSION

69. The Committee was informed that the date of the next session of the Committee would be September 1974, as agreed by the Executive Committee (see ALINORM 73/3), and would be held in Budapest.

ALINORM 74/23
APPENDIX I

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APPENDIX II

REPORT OF THE AD HOC GROUP ON TERMS OF REFERENCE,
AND GENERAL PRINCIPLES FOR THE ESTABLISHMENT OF
CODEX METHODS OF ANALYSIS AND SAMPLING

This Ad Hoc Working Group, consisting of Dr. F.E. Peters, AUSTRALIA, Dr. M. Bergström-Nielsen, DENMARK, Dr. William Horwitz, UNITED STATES and Dr. James Yeransian, UNITED STATES, was appointed to review the procedures established for endorsement of Codex methods of analysis, particularly:

(a) The proposal of Denmark to permit the Codex Committee on Methods of Analysis and Sampling (CCMAS) to concentrate on general methods of analysis; and

(b) The proposal of the United States to formalize the administrative operations of the Codex Committees in their selection, review, and endorsement of methods of analysis, statistical selection of samples, and technical operations in obtaining an analytical sample.

The Ad Hoc Working Group found no fault with the present Terms of Reference and General Principles. On the contrary, the Group found that these statements were still excellent guides to the selection of methods of analysis and sampling appropriate for international referee methods. These statements, however, have not been studied and applied routinely by the Commodity Committees or by the Committee on Methods of Analysis and Sampling. The Ad Hoc Working Group therefore, recommended that the Committee on Methods of Analysis and Sampling bring to the attention of the Commission the need to remind the Commodity Committees that they should follow the "General Principles for the Establishment of Codex Methods of Analysis" if they are to avoid rejection of methods not selected in accordance with the criteria and considerations given on pages 59-60 of the second edition of the Codex Alimentarius Commission's "Procedural Manual".

"The methods of analysis and sampling contained in the Codex Alimentarius are international referee methods intended for use in case of disputes". To determine that a method meets this definition it is essential that data be available to demonstrate the reliability of the proposed or approved Codex methods through the use of interlaboratory collaborative studies, preferably international. Only if a method can be applied by different laboratories with assurance that they will obtain the same value on the same sample is there a reason for believing that application of the method will settle a dispute. Commodity Committees, countries and organizations have developed a practice of submitting methods of analysis to the CCMAS without the documentation which supports their decision of approval of the method which should be on the basis of reliability and applicability.

As a guide to Codex Commodity Committees, the following illustrates the type of documentation which should accompany a request for endorsement:

1. For All Methods

(a) References to publications describing the origin, development, and use of the method or a review article containing this information.

(b) The results of interlaboratory studies involving the analysis of a number of identical samples by a number of different laboratories, e.g. six separate samples by five different laboratories.

2. For Application of Presently Endorsed Methods to other Commodities

General methods already approved and endorsed for Codex standards for other commodities need only evidence of the extension of applicability, providing the original endorsements were sufficiently well grounded. (An example is a general method for chlorides submitted by the United States for foods for infants and children based on the endorsed method for salt in processed tomato concentrates, which in turn was based on an AOAC collaboratively studied method. Additional evidence has been supplied of the applicability of the proposed method to other commodities in document CX/MAS 73/7 App.1.)

3. For Application of Potentially General Methods

Potentially general methods, based upon a review of the literature, and a determination that they meet the criteria for selection of methods of analysis in the Codex Alimentarius Commission's "Procedural Manual", may be considered for adoption and supplied to Commodity Committees as a recommended method for that constituent in standards already approved, under consideration, and to be proposed. Commodity Committees may also recommend potentially general methods. (The documentation required may be illustrated by the methods in Standards for Foods for Infants and Children, and for Low Sodium Content discussed in paras 23-62, ALINORM 72/23. An extensive set of working papers was prepared by the United States to demonstrate their applicability.)

4. For Application of Specific Methods

(a) Specific methods for a commodity, with supporting data, are acceptable in the absence of a general method or where for various reasons the general method is not applicable or is not as good;

(b) In cases where a method is specific to a commodity, e.g. thawing and cooking procedures for fish (CAC/RM 40-1971), where there is little possibility of general applicability to other commodities, the Codex Commodity Committee may request a waiver of review of the method by the Committee on Methods of Analysis and Sampling;

(c) Recognized methods, preferably of international organizations, but without supporting data, should be endorsed only temporarily, pending supplying the necessary information;

(d) In most cases, when a general method is not proposed by a Codex Commodity Committee, evidence or reasons must be supplied to justify its absence.

5. Analysis of Documentation

(a) When several apparently equally valid methods are available, a synopsis of the available methods should be prepared outlining the principles of the methods, their scope and applicability, reliability, sensitivity and detectability, potential interferences, speed, and economy (of reagents and apparatus). (Examples are the Canadian document on metals CX/MAS 70/C/2; Federal Republic of Germany Synopsis on Methods for Fruit Juices, CODEX/ANALYS/67-2, OICC Synopsis on Methods for Cocoa and Chocolate Products, CODEX/ANALYS/67-3; and the U.S. review of the Determination of Solvent residues, Annex III to CX/MAS 70/03.)

(b) If such a comparison of methods still does not permit a choice, the Codex Committee might request a country or organization to conduct an international collaborative study to compare the candidate methods on a number of samples, preferably covering the scope of commodities, range of concentrations, and presence of interfering materials under consideration. The study should be sufficiently extensive to give a reasonable assurance that a decision can be rendered after a statistical analysis of the results.

(c) When different methods for the same constituent in different commodities are submitted by the same or different Codex Commodity Committees, the Commodity Committee(s) should be advised to try to recommend a single method for the various purposes. If the Committee(s) is unable to effect such a coordination, the Committee on Methods of Analysis and Sampling may assign the problem to a member government or to an international organization interested in the problem. The assignee may recommend a coordinated method on the basis of a synopsis or through performance of an international collaborative study.

(d) When it is known that a single general method will be required for numerous commodities (e.g. protein, sugars, calcium, preservatives, etc.) the Committee on Methods of Analysis and Sampling may request volunteer governments or organizations to prepare or develop the required general referee method. (See, for example, ISO and AOAC to develop joint text for crude fibre and crude protein, ALINORM 72/23, paras 29 and 30.) Interested individuals, organizations and governments may recommend methods for review and study by such reviewers. (See, for example, "General Method for the Determination of Chlorides in Foods" proposed by US in response to para 10.9, Appendix IV, ALINORM 74/26.) Volunteer methods must be accompanied by data which indicate a reasonable probability that they are suitable as referee methods for a number of commodities. Volunteer methods unaccompanied by interlaboratory comparative data regardless of sponsorship, should be rejected. Methods and documents prepared in a satisfactory manner will be sent for review to the Codex Commodity Committee(s).

(e) Methods submitted by Codex Commodity Committees should not be endorsed if they do not meet the primary criteria, if other methods are available which do meet the criteria. Codex Commodity Committees should submit not only the recommended method but the basis for the selection.

6. Revision of an Adopted Method

(a) Requests for a revision of an adopted method may be made by a Codex Commodity Committee, a member country or an International Organization or by the Codex Committee on Methods of Analysis and Sampling itself.

(b) When a request is made to the Committee, the same information must be furnished, as it is required for a general method, as previously outlined.

(c) The Committee may assign the comparison of the new method, or a revision, in the existing method to a member government, or to an international organization, which may be interested in the problem. The assignee may recommend the new method on the basis of a synopsis, or it may organize an international collaborative study.

(d) It is essential that any revised method does not alter the particular Codex commodity standard.

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APPENDIX III

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

INTRODUCTION

1. At the opening of the 8th session of the Codex Committee on Methods of Analysis and Sampling, held at Budapest, 3-7 September 1973, it was the Chairman's suggestion that the detailed study of suitable sampling plans should be referred to an ad hoc working group held during the session and comprised of members of countries expressing particular interest; namely, Australia, Canada, France, Hungary, the Netherlands, Switzerland, UK and the USA. The ad hoc Working Group was requested to prepare proposals for the determination of net contents which could be used as guidelines for the Secretariat. At the request of the Chairman of the Committee, Mr. G.E. Anderson of Canada agreed to act as Chairman of the ad hoc Working Group.

The representatives were as follows:

Australia	- Mr. R.F. Clement
France	- Mr. G. Janssens
Hungary	- Mr. E. Zukál and Dr. J. Kindler
The Netherlands	- Drs. A. Kruyse and Mr. M. Osse
Switzerland	- Dr. P. Koch and Dr. L. Lehner
UK	- Mr. A.W. Panario
USA	- Mr. P. Khan and Mr. R.P. Farrow, pending the arrival of Mr. R.S. Elder who continued as the US representative.

PREAMBLE

2. The Chairman of the ad hoc Working Group called upon the representatives of Switzerland, Dr. Koch and Dr. Lehner, to outline the general principles of their sampling plans as most members of the Committee did not have an opportunity prior to the opening of the Plenary Session to study the papers included in document CX/MAS 73/13 and 73/14. In essence it might be said that the Swiss plan involved a sequential sampling scheme based upon the method of variables combined with an attributes plan. One of the interesting features of the Swiss plan was that it required a maximum sample size of $n = 25$. Because sequential sampling plans are inherently more efficient than single sampling plans, the average sample number, ASN, would, therefore, be approximately 10. This, of course, could result in considerable economies in inspection, particularly where destructive testing of samples might prove necessary. One of the key points of the plan was that for an average net content of the lot equal to the declared net content, the probability of acceptance by the plan would be equal to or greater than 95%.

3. Mr. Anderson submitted a brief paper with tables which served as corrections and additions to the paper CX/MAS 73/13. He pointed out that Table XI appearing in his original paper was in error in some respects and should be replaced by Table XI* which gives the probabilities of acceptance of lots of varying composition, so that for a lot containing 50% good items, 49% marginal items and 1% defective items, as defined in his paper, the probability of acceptance would be equal to or greater than 99%.

Mr. Anderson went on to emphasize that since the writing of the original paper in November 1971, the Canadian position had changed to the extent that the probability of acceptance of a lot such as that specified above would be reduced from 99% to approximately 50%. The decision to make this change was dictated in part by a study of the possibility of processing equipment capable of extremely tight control over packing processes being used in such a way that a packer could "target" at a lot average below the declared net contents with a limited risk of rejection.

4. Mr. R.S. Elder from the USA briefly outlined the US scheme which was based upon a variables plan. Under this plan, for acceptance the average of a sample must not be less than the declared net contents, subject to the further restriction that no single item in the sample should be below a limiting value which is equal to the declared net contents minus a certain tolerance. Mr. Elder noted that the plan currently being considered in the USA differs from the plan outlined in his paper contained in document CX/MAS 73/13 and 73/14 and also from the sampling plans published for comment in the US Federal Register, December 18, 1971.

5. After some discussion it appeared that Mr. Elder and Mr. Anderson agreed that, although their sampling plans differed considerably in principle, yet in practice they would result in very similar operating characteristics.

6. The UK position was described as embodied in the Weights and Measures Act of 1963 which provided that no items should fall below the declared weight. However, in practice, this had been interpreted by the UK official representatives after consultation with UK industry as implying that not more than 2.5% of all the items should fall below the declared weight, with the additional constraint that not more than one "considerably" deficient item in 1000 should be produced. The term "considerably" was deliberately left vague since it was considered that different views might be upheld by the courts in relation to different commodities. However, as a broad guide, 2% deficiency was suggested. It has been found in practice, as tested in the courts, that this essentially fulfilled the intentions of the legislation.

7. The Netherlands representative, Drs. A. Krusysse, indicated that the position in his country was almost the same as in the UK. Thus, the Netherlands Food Act requires that each item should conform with the regulations in that the net weight of each item should not be less than the declared weight. However, it appeared as a result of actual practice that a small portion of a lot could be permitted to contain less than the declared net contents. As a result of this requirement, the average net contents of a lot would be above the official value.

8. The Australian delegate, Mr. Clement, stated that although there is no uniform legislation in operation throughout Australia at present, a system very similar to the one proposed by the USA was under consideration. In fact, the only difference between the US and the Australian proposed plans appeared to be in the sample size.

Thus, there appear to be essentially three types of plans:

- (a) those which aim to give maximum protection to the consumer;
- (b) those which stress the need to prove beyond reasonable doubt that a lot is in violation before enforcement action is taken; and
- (c) those plans which propose to adopt an intermediate position by equating the producer's and consumer's risks at 50% for a lot whose lot average was equal to the declared weight.

POINTS OF AGREEMENT

9. A consensus was reached on the following points:

- (1) For all plans the lot average must be equal to or greater than the declared weight.

(2) There should be at least one lower limit, i.e. a net content " L_a " equal to the declared content minus "e", where "e" is a tolerance.

(3) The tolerance "e" should in general decrease as a percentage of the declared content "D" as "D" increases in value.

(4) Some consideration must be given to the ease or difficulty of packing when seeking to establish values of "e".

(5) Such values of "e" should be established by the appropriate Codex Commodity Committees, taking into account the possibility that different commodities may not differ essentially in ease or difficulty of filling.

(6) It would be desirable to group commodities so that there would be at most two or three sets of tolerances "e" for all commodities.

(7) The values selected for "e" should be recognized as being dependent upon the value chosen for "p" as discussed in Point 9 below.

(8) The values of "e" should be shown in tabular or graphical form as absolute values or as percentages of the declared net content for each set of tolerances and for each value or range of value of declared net contents.

(9) The value of "p", i.e. the maximum permissible percentage of items in the lot below the limit " L_a " should be a single value for all commodities and for all values of "e". Thus, "p" might be 5% as in Switzerland, 2.5% as in the EEC proposal or 1% as in Canada.

(10) In addition to the limit " L_a ", there could also be specified an absolute lower limit " L_b " below which the net content of no item shall lie. " L_b " will, of course, always be lower than " L_a ". " L_b " may be expressed as $L_b = D - h$. e. The value of "h" should be related to the value of "p" but it is not expected to exceed 2.5.

(11) It is agreed that the mathematical statistics of all plans should be based on the assumption that the lot size is infinite.

UNRESOLVED ISSUES

10. It appears that approach to agreement on a plan must be dependent upon the specification of an Operating Characteristic Curve acceptable to all parties. If such a curve could be agreed upon, then it was thought that the method of sampling to be used, i.e. by attributes, by variables, or by some combination of these, would not be of critical importance from a purely statistical point of view provided the method was such as to result in conformity to the agreed curve. Other factors, such as economic considerations, type of personnel employed, simplicity of inspection methods, whether or not destructive testing would be required, etc. should influence the details of the method chosen.

11. However, no agreement could be reached on a suitable Operating Characteristic Curve. Essentially there appeared to be two divergent points of view. The first was held by the Swiss delegation who wished to specify an Operating Characteristic Curve which, with the lot average at the declared weight, would provide a 95% probability of acceptance. It should be noted that the Swiss point of view is shared by the Federal Republic of Germany and has been proposed for adoption by the Commission of the EEC. The second point of view held by Canada, the USA and Australia, specified an Operating Characteristic Curve which, for a lot average equal to the declared net contents, would have a probability of acceptance equal to 50%; i.e. the declared value could be considered to be the Indifference Quality or IQ.

12. Thus, the USA and Canadian plans would require packers to fill to a greater extent than would the Swiss plan. Again, the legislative requirements both in the Netherlands and the UK would require an even greater degree of over-filling on the part of packers.

13. The question of variations in sample size in relation to lot size was discussed at considerable length but no consensus could be reached. It was felt that this question could be discussed further at a future meeting.

RECOMMENDATIONS

14. The ad hoc Working Group noted the decision on the interpretation of "net contents" given at the meeting of the Codex Committee on Food Labelling in Ottawa, May-June 1973; that the declaration was intended to be on the basis of "average contents". Having considered this definition, the ad hoc Working Group recommended that:

(1) the documents CX/MAS 73/13 and 73/14, as amended, together with this report be circulated to all member countries of the Commission. In particular, governments should be asked to comment on the following two points:

(a) which of the two philosophies on sampling for average net weight set out as points (b) and (c) in paragraph 8 of the section of this report titled "PREAMBLE" is preferred;

(b) whether the eleven points set out in paragraph 9, the section titled "POINTS OF AGREEMENT" are acceptable.

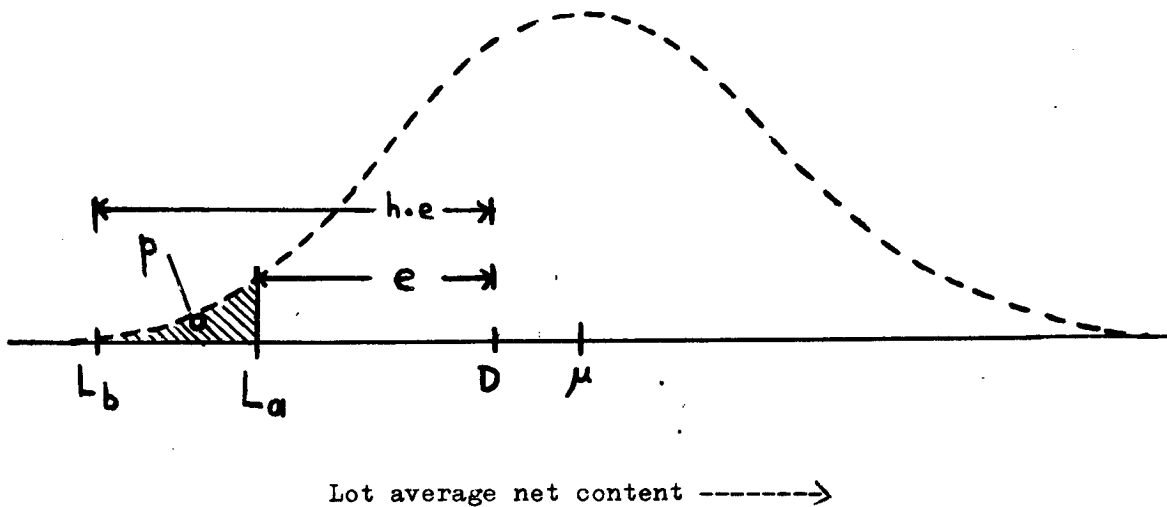
(2) in order to show in an objective fashion the full implications of these general observations it would be desirable to develop a series of Operating Characteristic Curves for each plan and for a selected set of tolerances, and range of net contents and such critical points as may be thought desirable. It was, therefore, recommended that the ad hoc Working Group should continue its work until the next meeting at which time such curves could be presented for distribution and comment to the members of the whole committee;

(3) the Chairman of the Committee should be respectfully requested to suggest suitable working arrangements in the interim period so that these calculations could be carried out.

ALINORM 74/23
APPENDIX III
Annex

The following Diagram may prove useful in defining certain terms as used in the foregoing report.

Distribution of net content in the lot, assumed normal or nearly normal.



D = declared net content of the lot

μ = lot average net content: it has been shown in the diagram above to the right of D but it could lie to the left or on D itself.

L_a = first lower limit. $L_a + e = D$

L_b = second and absolute lower limit below which no item may lie
 $L_b + h \cdot e = D$

e = permissible tolerance

p = the percentage of units from a lot specified as allowable below the point L_a

DETERMINATION OF SODIUM AND POTASSIUM IN FOODS

1. SCOPE: Generally applicable to the determination of sodium and potassium in foods, including low sodium foods.
2. PRINCIPLE: The food is evaporated and dried, if necessary, and ashed. The residue is dissolved in acid and diluted, if necessary, to a concentration of sodium and/or potassium which can be determined in a flame photometer. In special cases, the determination may be performed directly on liquid foods or aqueous extracts. If sufficient potassium is present to interfere with the sodium determination, the measured amount of potassium is added to the sodium standards to provide a potassium-corrected standard curve.
3. REAGENTS:
 - 3.1 Sodium stock standard solution.--1.000 g Na/l. Dry reagent grade NaCl at 100°C overnight. Weigh 2.5422 g, dissolve in H₂O, and dilute to 1 l with H₂O.
 - 3.2 Sodium intermediate standard solution.-- 100 mg Na/l. Dilute 10 ml stock solution to 100 ml with H₂O.
 - 3.3 Sodium working standard solutions.--Dilute 1, 2, 4, 6, 8, and 10 ml intermediate standard solution to 100 ml to prepare solutions containing 1, 2, 4, 6, 8, and 10 parts per million Na. Store in polyethylene bottles. Other working standard solutions as appropriate to the particular instrument may be prepared.
 - 3.4 Potassium stock standard solution.--1.000g K/l. Dry reagent grade KCl at 100°C overnight. Weigh 1.9068 g, dissolve in H₂O, and dilute to 1 l with H₂O.
 - 3.5 Potassium intermediate standard solution.--100 mg K/l. Dilute 10 ml stock solution to 100 ml with H₂O.
 - 3.6 Potassium working standard solutions.--Dilute 1, 2, 4, 6, 8, and 10 ml intermediate standard solution to 100 ml to prepare solutions containing 1, 2, 4, 6, 8, and 10 parts per million K. Store in polyethylene bottles. Other working standard solutions as appropriate to the particular instrument may be prepared.
 - 3.7 Lithium standard solution.--1.000 g Li/l (1000 ppm). Dissolve 6.108 g LiCl in H₂O and dilute to 1 l. For use only with instruments requiring use of an internal standard.
4. APPARATUS
 - 4.1 Flame photometer.--Instrument which can measure 1-10 parts per million sodium conveniently at 589 nm and 1-10 parts per million potassium at 768 nm. (Beckman DU with oxyhydrogen flame and photomultiplier tube is satisfactory.) Instrument using LiCl internal standard is also satisfactory. Operate instrument in accordance with instructions of manufacturer.
5. PREPARATION OF SAMPLE
 - 5.1 Direct determination (for wines, mineral water, etc.)--Dilute sample with H₂O, if necessary, to reduce Na and/or K concentration to range covered by flame photometer.

- 5.2 Aqueous filtrate (for fruits and fruit products).--Weigh into 1.5 or 2 l beaker 300 g fresh fruit or equivalent of dried fruit, canned fruit, fruit juice, preserves, marmalades, etc. which have been pulped or mixed in blender or mechanical grinder. Add about 800 ml H₂O and boil 1 hr, replacing at intervals H₂O lost by evaporation. Transfer to 2 l volumetric flask, cool, dilute to volume and filter. Use filtrate for determination. (Proportionately smaller quantities may be used.)
- 5.3 Acid solution of ash (for other foods)—Ash indicated quality of sample (Table 5.5) in Pt, Vycor, or other inert Na- and K-free dish at 525-550°C until ash is carbon-free. Dissolve in 0.1 N HNO₃ or HCl, filter into 200 ml volumetric flask, and rinse and wash dish and filter to final volume.
- 5.4 Alternatively, ash indicated sample at 525-550°C for 1-2 hr, dissolve in 50 ml 0.1 N HNO₃ or HCl, filter, and wash with the 0.1 N acid to about 100 ml. Return paper and unburned carbon to dish and re-ash until carbon-free. Dissolve remaining ash in about 50 ml of the 0.1 N acid and filter into retained filtrate, washing with the acid to final volume of 200 ml.

5.5 <u>FOOD</u>	<u>SAMPLE SIZE</u>
Baking powder	2.5 g
Beer	50 ml
Beverages and concentrates	5-10 g
Bread	2 g
Butter, cheese, ice cream	3-5 g
Cacao products (water-, sugar-, fat-free material)	1 g
Cereal products, flour	3-5 g
Coffee, tea, wort	5-10 g
Eggs and egg products	5 g
Fish and marine products	4 g
Flavours	10 ml
Gelatin and dessert preparations	2.5 g
Grains	5-10 g
Low sodium foods	10 g
Meat and meat products	2-3 g
Milk and liquid products (cream, evaporated milk, sweetened condensed milk)	5 g
Milks, dried	1 g
Nuts and nut products	2-3 g
Oils and fats	5 g
Plants	5-10 g
Syrups, sugars	5-10 g
Spices	2 g
Vegetable products, processed	2-3 g

6. DETERMINATION

- 6.1 Sodium in absence of interfering amounts of potassium.--Dilute sample solution, if necessary, to reduce Na concentration to range covered by flame photometer (preferably 4-10 ppm). Aspirate solution directly into flame and record reading. Similarly aspirate working standard solutions into flame, record reading, and prepare standard curve. When analyzing series of samples, include at least one working standard solution at convenient intervals to check stability of instrument.

Calculate and report as mg Na/100 g sample or as ppm Na.

If internal standard type instrument is used, add appropriate amount of LiCl standard solution indicated in instructions to all sample and working standard solutions before diluting to final volume.

6.2 Potassium.--Dilute sample solution, if necessary, to reduce K concentration to range covered by flame photometer. Proceed as in 6.1 using K working standards and appropriate wavelength.

6.3 Sodium in presence of interfering amounts of potassium.--Determine Na and K as in 6.1 and 6.2. Repeat the Na determination using a new series of sodium working standard solutions containing 1-10 ppm NA and the concentration of K indicated by 6.2. Determine the Na concentration using the Na-K standard solutions.

7. SPECIAL ASHING TREATMENTS

7.1 Dry products.--Wet down with few drops H₂O and distribute solids evenly over bottom of dish. Dry on steam bath, and place dish at entrance to muffle so that sample smoulders without catching fire. Then ash as in 5.3.

7.2 Oils and high fat products.--Use low temperature first to smoke off fat without burning; then raise to specified temperature.

7.3 High sugar products.--Heat sample on steam bath or under infrared lamp until H₂O is expelled and material begins to char. Add few drops pure olive oil to low purity samples to avoid spattering. Continue heating under infrared lamp until swelling stops. Then place in heated muffle.

8. SPECIAL CASE WHERE SUFFICIENT CALCIUM IS PRESENT TO INTERFERE IN DETERMINATION

In those cases where the calcium concentration is sufficient to affect the determination of sodium and/or potassium, determine the amount of calcium present as directed for potassium, 6.2, using a wavelength of 423 nm and a calcium standard solution (1.000 g Ca/l, appropriately diluted, prepared from 2.500 g CaCO₃ dissolved in a slight excess of diluted HCl and diluted to 1 l). Repeat the sodium (or potassium) determination as in 6.3, except to add the equivalent amount of calcium standard solution to the Na (or K) standard solution.