

codex alimentarius commission

FOOD AND AGRICULTURE
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OF THE UNITED NATIONS

WORLD HEALTH
ORGANIZATION

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ALINORM 79/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Thirteenth Session, Rome, 3-14 December 1979

REPORT OF THE ELEVENTH SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Budapest, 2-6 July 1979

Introduction

1. The Codex Committee on Methods of Analysis and Sampling held its Eleventh Session from 2 to 6 July 1979 in Budapest by courtesy of the Government of Hungary. The session was opened by Dr. K. Süttö, President of the Hungarian National Codex Committee and Vice-President of the Hungarian Office for Standardization, who welcomed the participants and stressed the need for cooperation among international organizations in developing methods of analysis and sampling for Codex purposes. He introduced the Chairman of the Session, Professor R. Lásztity of the Department of Biochemistry and Food Technology of the Technical University of Budapest.
2. Professor Lásztity pointed out the particular importance of the present session in that examination of the work programme of the Committee at the 24th Session of the Executive Committee and at the 12th Session of the Commission had led to a revision of its terms of reference. These changes would be considered at the present session.
3. The session was attended by delegates from 23 countries and observers from five international organizations. The list of participants including officers from FAO is attached as Appendix I to this Report.

Adoption of the Agenda

4. The Committee agreed to the adoption of the Agenda without change. It also agreed that the points on sampling for discussion under items 6(a) and 6(b) should be dealt with by an Ad hoc Working Group from which, if necessary, a small sub-group could consider the more specific field of sampling plans under 6(b)(i) and 6(b)(ii). The delegations of Hungary, the Netherlands, Norway, Switzerland, the United Kingdom, the United States of America and observers of the EEC and IUPAC indicated their willingness to participate in the Working Group.

Appointment of Rapporteurs

5. The Committee appointed Mr. R. Sawyer of the delegation of the United Kingdom and Mme. J. Castang of the delegation of France as rapporteurs.

Matters arising from the Commission and Codex Committees

6. The Committee had before it document CX/MAS 79/2 and CX/MAS 79/2 - Add. 1 containing information of interest from the Reports of the Commission, from General Subject Commodity Committees and from the 2nd Joint FAO/WHO Expert Consultation on Methods of Sampling and Analysis of Contaminants in Food. It was noted that both the 24th Session of the Executive

Committee and the 12th Session of the Commission had considered the future work of the Committee and both bodies had decided that the Committee should continue but that its terms of reference should be modified. It was also strongly recommended by both bodies that the Working Group on Sampling should continue its work on the elaboration of acceptance sampling plans.

Classification of Codex Methods of Analysis

7. There had been considerable discussion at the Executive Committee (24th Session) on the proposed classification of Codex methods of analysis into four categories for which definitions and terminology were still to be formally agreed. The Executive Committee had suggested that more attention should be given to methods which were generally applicable to standards and to methods which could be used for the enforcement of regulations to meet the needs of the developing countries. It was also emphasized that the role of the Committee should be one of endorsement of methods proposed by Commodity Committees and coordination of activities with other specialized international organizations.
8. The Committee noted that the 2nd Joint FAO/WHO Expert Consultation (Rome, February - March 1978) had also considered and agreed with the proposed classification of Codex methods.
9. The Committee decided that a detailed review of the classification was necessary at the current session, since there had been little time at the 10th Session to examine the proposals of the Ad hoc Working Group (see ALINORM 78/23, Appendix II).
10. It was agreed that all future methods of analysis should be categorized into the four types first recommended by the 10th Session of the Codex Committee on Methods of Analysis and Sampling (ALINORM 78/23, Appendix II), which have been named:
 1. Defining (Type I) Method
 2. Reference (Type II) Method
 3. Approved Alternative (Type III) Method
 4. Tentative (Type IV) Method
11. The Committee then considered the definitions required to establish the four types of methods proposed by the Ad hoc Working Group. It was agreed that in the case of the Type I Method there was need to revise the definition. Detailed considerations of each type of method followed and led to these general observations.
12. Both Type I and Type II Methods are suitable for reference and calibration purposes. Type III Methods can be used as alternatives for regulatory and compliance purposes. The Committee also considered that there may be several Type III Methods in a single standard. As regards Type IV Methods, they require confirmation of criteria for Codex methods before they can be used as "official" methods in Codex standards. Moreover, when there are alternative methods to Type I, the "defining" method should serve for calibration purposes.
13. As described in Appendix II, the Committee also agreed to the criteria to be considered for all methods of analysis:
 1. Specificity
 2. Accuracy
 3. Precision
 4. Limit of detection
 5. Sensitivity
 6. Practicability
 7. Other criteria which may be selected as required.
14. There was also general agreement that the Ad hoc Working Group on Sampling (Appendix III) should prepare similar classification and similar criteria for sampling methods.

15. The basis of relationships with other Codex Committees as set out in paragraphs 12 and 13 of Appendix II to ALINORM 78/23 was also agreed to with the addition of the requirement to assess usefulness and application of methods submitted by the Commodity Committees.

Revised Terms of Reference of the Codex Committee on Methods of Analysis and Sampling and consequential changes in the Procedural Manual, including definition and classification of methods of analysis and sampling

16. The Committee had before it CX/MAS 79/3 on the above subject and two Conference Room Documents prepared by a small Ad hoc Working Group which had met during the session—Conference Room Document No. 1 containing an amended text proposed for para 13(c)(i) of the Procedural Manual which dealt with relations between Commodity Committees and General Committees, and Conference Room Document No. 2 containing proposed amendments to General Principles for Establishment of Codex Methods of Analysis (see also ALINORM 78/23, App.II).

Revised Terms of Reference of the Committee

17. The Committee recognized that the revised terms of reference (Appendix I to CX/MAS 79/3) proposed by the Secretariat had been agreed to by the Codex Alimentarius Commission at its 12th Session (ALINORM 78/41, para 284) and that consequential changes to the Procedural Manual would be considered at its 13th Session.

18. In examining the terms of reference, the Committee noted that there were some editorial amendments to be made and revised the text accordingly (see Appendix II to this Report).

Proposed Amendments to the Guidelines for Codex Committees (para 13(c)(i))

19. The Committee re-examined the revised text proposed by the Ad hoc Working Group. After discussion it was decided that modifications were necessary to make it clearer that methods of analysis and methods of sampling were complementary to one another.

20. The revised texts are attached as Appendix II to this Report.

"General Principles for the Establishment of Codex Methods of Analysis" [and Sampling]

21. In examining the present document which dealt with Methods of Analysis, there was some discussion as to whether, in the light of the work of the Ad hoc Working Group on Sampling, a similar text to the above dealing with the General Principles for the Establishment of Codex Methods of Sampling was necessary. It was decided that paragraph 1 of the present document which stated the purpose of Codex Methods of Analysis also applied to Sampling and that in the same document both the definition of types of methods of analysis and of methods of sampling and the general criteria for their selection should appear.

22. The Committee agreed that a section dealing with Sampling would need considerable work and discussion and agreed to consider the matter more in detail at its next session (see para 30) so that the new Working Group on Sampling would have the opportunity to prepare a suitable text.

23. The Committee noted that when there were alternative methods to Defining Methods (Type I), the defining method must also serve for calibration purposes and that in this respect they overlapped with Reference Methods (Type II). It was agreed to amend the definition of defining methods and reference methods accordingly to select appropriate terms for each type of method and to add further methods as examples of the types.

24. With regard to the General Criteria, the Committee noted that the distinction between repeatability and reproducibility as commonly defined (see e.g. 2nd Joint FAO/WHO Expert Consultation on Methods of Sampling and Analysis of Contaminants in Food, page 2) referred to intra-laboratory (within laboratory) and inter-laboratory (within laboratory, plus between laboratories) results respectively and agreed to make the distinction in the text.

25. It also noted that there was general consensus on the definitions of specificity, accuracy and precision but that such terms as 'sensitivity' were more open to interpretation and might need future examination by the Committee and it was agreed that a working document based on IUPAC/ISO recommendations amongst others would be a suitable basis for discussion. The amended document is attached as Appendix II.

Report of the Ad Hoc Working Group on Codex Methods of Sampling

26. The Committee had before it a report of the above Ad hoc Working Group (see Appendix III). In introducing the report the chairman of the Group (Mr. B. Levy, USA) drew the Committee's attention to several fundamental issues which the Group had discussed, i.e.:

- (a) There was a need to continue work on the harmonization of the approach to sampling and the establishment of practical sampling plans;
- (b) Codex recommendations concerning sampling should have an advisory character although it was noted that certain aspects of sampling plans such as the statistical criteria relating to 'Lot Acceptance' would have a mandatory character as regards the Codex Procedure for the Acceptance of Standards;
- (c) Provisions in Codex standards (e.g. minimum or maximum limits) required clarification in relation to the acceptability of the lot.

27. The Committee agreed that to ensure that the work on sampling, as envisaged in the new terms of reference of the Committee, could be realized, appropriate arrangements (facilities, secretarial assistance, etc.) would have to be made.

28. The Committee noted that the Group had also made recommendations for future work in the field of sampling and had dealt with the specific problems relating to sampling to net content determination and the amendment of the Sampling Plans for Prepackaged Foods (CAC/RM 42-1969) proposed by the Codex Committee on Processed Fruits and Vegetables.

29. The Committee agreed with the views of the Ad hoc Working Group concerning the purpose and status of Codex methods of sampling (see also para 21) and also agreed that it was essential that the criteria on the basis of which lots were rejected (e.g. interpretation of results of sampling and analysis in relation to provisions in Codex standards and in relation to lots) should be harmonized internationally. The recommendations of the Group for future work were also accepted.

30. In order that progress be made in the field of sampling, the Committee decided to set up a new Ad hoc Working Group on Sampling. The new Working Group's task would be to initiate work on the various aspects recommended by the present Working Group. The Committee requested Mr. R. Player (United Kingdom) to act as coordinator, assisted by the Hungarian and Codex Secretariats, of the activities of the Working Group. It was recognized that it might prove necessary to call a meeting of the Working Group between the present and the next session of the Committee. The Secretariat undertook to look into this matter.

31. The following countries and Organizations were designated as members of the Working Group, although it was understood that other countries and Organizations were welcome to participate as members of the Group: Australia, Egypt, Finland, Germany (Fed. Rep.), Hungary, the Netherlands, Norway, Switzerland, UK, USA and the EEC.

32. As regards the question of the moderate acceptance probability plan for the definition of net weight in relation to lots, the Working Group had the opinion that the above plan was of considerable technical merit.

33. The Committee agreed that a questionnaire should be prepared on the basis of which the views of governments could be sought on the general acceptability of the plan. The Secretariat undertook to issue such a questionnaire in consultation with the chairman of the present Working Group. It was also agreed that following reconsideration of the moderate probability plan on the basis of government comments, the question of net contents should be referred to interested Codex Committees for further consideration.

34. In considering the proposed amendment to the Sampling Plans for Prepackaged Foods (CAC/RM 42-1969) the Group had made specific comments in reply to the questions raised in document CX/MAS 77/2 - Add. 1. However, it was not in a position to assess the effect on lot acceptance probability as a result of the reduction of the sampling rate at the larger lot sizes proposed in the working document.

35. The Committee decided to refer the comments of the Working Group to the originating Commodity Committee so that these could be considered. The Secretariat was requested to look into the matter of how the amendment of the Sampling Plans should be pursued.

36. The Committee drew the attention of Commodity Committees to the Report of the Working Party on Sampling of Quick Frozen Foods (ALINORM 79/25, Appendix XIII) in which consideration had been given to the layout of the sampling and analysis sections of commodity standards to take into account the need for different sampling plans and lot acceptance procedures.

General Method for the Determination of Chlorides in Foods

37. The Committee had before it ALINORM 76/23, Appendix IV containing the above standard.

38. The Committee was informed that the use of SI units for measurements was now mandatory in some countries and international organizations and would come into force in the near future in others.

39. It was agreed that the above method should be edited to include SI units and that, as a general principle, their use should be instituted in future methods of analysis and sampling. SI units should be accompanied, where necessary, with the other conventional units in brackets to enable analysts to make the conversions easily during a transition period. Standards already published should, when revised, be treated in the same way.

40. The Committee noted that, at its 4th Session (ALINORM 69/23, paras 84-85) the Committee had considered the standard layout for a Codex Method of Food Analysis, based on ISO R78 as a highly useful document and decided to adopt it in toto and to recommend it for use by the Commodity Committees.

41. The document ISO R78 had now been revised and was accepted by the ISO Council as ISO Guide 18. Some delegations pointed out that the document at present under examination did not follow the ISO layout and suggested that it should be re-edited to this format for consideration by the Committee later in the session. It was noted, in particular, that data on reproducibility and repeatability were necessary to complete the text.

42. The Committee noted that, in general, only those methods of analysis and sampling which would be included in extenso in Codex Standards were required to be edited into standard format and that the present text, which had already been published elsewhere (JAOAC, 58, 399-400 (1975)) would only be given by reference. Nevertheless because this was a general method which had been authenticated by collaborative studies it was agreed to present it in standard layout as a model and to add to it references to the collaborative studies and for guidance to analysts to add the data indicating the characteristics of the method. The revised document is attached as Appendix IV to this Report. The Committee agreed to advance the method to Step 8 of the Procedure.

Endorsement of Methods of Analysis proposed by Codex Commodity Committees

43. The Committee had before it document CX/MAS 79/6 including methods of analysis requiring endorsement. A summary of the conclusions taken and endorsement status of the methods discussed is included in Appendix V. The question of introduction of SI units into existing methods was raised and it was agreed that this requirement should be drawn to the attention of Commodity Committees for action in future submissions.

NATURAL MINERAL WATERS

44. The Committee had before it document CX/EURO 79/5 containing proposed methods of analysis for Mineral Waters and CX/MAS 79/LIM.1 containing an extract from the Report of the Eleventh Session of the Coordinating Committee for Europe.

45. The Polish delegation stated that it had received the document CX/EURO 79/5 a few days before the session of the Codex Committee on Methods of Analysis and Sampling and that CX/MAS 79/LIM.1 had been received during the session. It considered that the document could not be discussed in a situation when the delegations had no time to read it. The delegation considered that documents of such great importance as those concerning the methods of analysis should be distributed at least two months before the session at which they were to be discussed as stated in the Procedural Manual, page 11, para 7. Distribution of the documents just before the session could not result in constructive discussion at this session.

46. The documents were introduced by the chairman of the Coordinating Committee for Europe, Dr. H. Woidich (Austria). The Committee noted para 48 of the Report of the Coordinating Committee for Europe in which it was recorded that recognition had been given to current developments in CCMAS in respect of classification of methods of analysis and the criteria necessary to establish their use. It also noted that the Coordinating Committee for Europe had discussed whether it should propose methods for the determination of substances not specifically limited or mentioned in the standard and had decided to refer such methods for consideration by the Codex Committee on Methods of Analysis and Sampling.

47. An attempt had been made by the Coordinating Committee for Europe to indicate the Type I (Defining Methods) in the list proposed for endorsement, CX/MAS 79/LIM 1, Appendix III. Those methods for which there were no provisions in the standard were also identified. Some delegations were of the opinion that methods, which sought to prove absence of particular undesirable substances, could not properly be classified as "defining methods". In some cases the measurement of specific substances or groups of substances was involved and in no case was information on limit of detection of the methods proposed available for consideration. The proposal to include methods in the standard where no specific provision is made in the appropriate compositional sections was discussed in detail. Several delegations expressed concern that an undesirable precedent was being set and that the manner in which the information was presented did not allow the Committee to make a proper examination of the propositions.

48. The Committee decided that the question of whether for a substance where there is no provision in the Standard a method of analysis should be required should be referred to the Codex Committee on General Principles for guidance.

49. The delegate of Norway stressed that due consideration be given to the irrelevance of methods referred to in paras 47 and 48 to the requirements of the standards especially where such requirements were apparently intended for substantiation of labelling matters. Attention was drawn particularly to the terms of the Codex Procedural Manual (p. 69, 2(d)). The Committee agreed that this matter required consideration by Governments.

50. The Committee recognized that there would need to be a period of transition created by the discussions on changes in terms of reference, the classification of methods and the emphasis being placed on criteria for acceptance of methods.

51. In view of this position the Committee agreed to temporarily endorse, pending collaborative studies, the two 'defining methods' (Type I) proposed by the Coordinating Committee for Europe, viz. 3.1.1 Total Dissolved Solids and 3.3.1 Total Organic Matter, but requested that these be developed in the appropriate ISO format and circulated for comment by governments. With regard to the remaining methods the Committee agreed that they be classified as Type IV methods and that they be circulated for comment at Step 6.

QUICK FROZEN FOODS

Quick Frozen Brussels Sprouts, Quick Frozen Green Beans and Wax Beans, Quick Frozen Carrots

52. The methods submitted were endorsed (see Appendix V to this Report).

Quick Frozen Spinach

53. The delegation of the United Kingdom commented that the sampling procedures submitted for endorsement were subject to future agreement on general principles on sampling and that a decision should be deferred until the Working Group on Sampling had reached workable conclusions. The remaining methods were endorsed with minor amendments shown in Appendix V.

PROCESSED FRUITS AND VEGETABLES

Pickled Cucumbers

54. The Committee agreed that the attention of the Codex Committee on Processed Fruits and Vegetables should be drawn to the Codex General Method for chloride and that the Secretariat should be given authority to update references to AOAC procedures as applicable. The Committee was concerned that three methods were proposed for the determination of fill of container and that one of these should be designated as the reference method.

Canned Carrots

55. The methods submitted were endorsed.

Dried Apricots

Determination of Moisture

56. There was a discussion as to whether the AOAC methods proposed by the Commodity Committee were defining or reference methods. Some delegations were of the opinion that both the oven and the electrical conductance methods were empirical methods and should therefore be regarded as "defining methods". Other delegations were of the opinion that the AOAC oven method, which measured loss of mass on drying, was a reference method which had been used for the calibration of the instrumental method. It was recognised by these delegations that the AOAC methods did have elements of defining methods and that this particular problem needed resolution before progress could be made.

57. The Committee accepted the suggestion of the delegate of Austria that the definition of a "defining method" should be changed by indicating that such methods also served for calibration purposes (see paras 12 and 23). Appendix II was revised accordingly. Such a clarification of "defining methods" was thought to overcome difficulties in deciding on occasions whether a method was a Type I or a Type II Method.

58. The methods proposed by the Commodity Committee for moisture determination were endorsed.

DATES

Determination of Moisture

59. The same conclusions were reached as under Dried Apricots above.

CANNED APRICOTS

Syrup Measurements

60. The delegation of Austria was of the opinion that the method for the determination of soluble solids in pickled cucumbers (see para 54) and the syrup measurement in canned apricots should be brought into line with each other. It was also questioned why in the former case no correction for temperature had been provided for. The Committee requested the Commodity Committee to clarify this matter.

PROCESSED MEAT AND POULTRY PRODUCTS

CANNED CORNED BEEF, LUNCHEON MEAT AND COOKED CURED HAM

61. The delegation of the USA wished to be informed whether collaborative testing had been carried out to establish the reliability of the ISO methods proposed by the Commodity Committee. The representative of ISO informed the Committee that the methods suggested by the Commodity Committee had been finalized by ISO and that the references quoted in the working paper required updating.

62. The Committee discussed whether, in endorsing proposed methods of analysis, it should insist at this session that evidence of collaborative studies be available. It was agreed that there was a need for a transition period until such information would be available.

Determination of Nitrite, Glutamic acid, Glucono-delta-lactone

63. In view of the discussion concerning the availability of results of collaborative testing (para 42) the Committee decided to temporarily endorse the above methods.

FATS AND OILS

Reduced Fat Margarine

64. The Committee was informed that the Commodity Committee would review this standard. It was more than likely that the standard would be substantially altered. Some delegations questioned whether bioassays were still appropriate as Codex methods. The delegation of the USA pointed out that the bioassays for Vitamin D measured biological activity and were therefore suitable as calibration methods and that many chemical methods were non-specific. The delegation of Norway informed the Committee that the alternative instrumental methods were being developed and could be subjected to collaborative study. In view of the fact that the standard is still subject to considerable alteration, the Committee agreed to draw the attention of the Committee on Fats and Oils to the problems likely to arise from the submission of older methods and to postpone further consideration of the methods of analysis suggested.

Edible Low Erucic Acid Rapeseed Oil

Determination of Fatty Acid Composition

65. A number of delegations were of the opinion that there was a need for an appropriate method of determination of erucic acid since the content of this fatty acid was the basis on which the above product was distinguished from the normal rapeseed oil.

66. The delegation of France was of the opinion that the IUPAC method quoted in the standard was adequate for this purpose. The representative of the EEC stated that the Community had developed a method of determination for erucic acid in fats and oils to be used for human consumption as well as for foods to which fats and oils have been added.

The principle of this method is based on the separation of methyl esters of fatty acids contained in the product, analyzed by low temperature thin-layer chromatography on silica gel loaded with silver nitrate and quantitative determination of the separated esters by gas chromatography. This method allows the separate determination of erucic acid in the presence of various isomers (e.g. fatty acids in fish oils as such or hydrogenated).

67. The delegate from Australia suggested that the title of the section dealing with the determination of fatty acid composition should be changed to reflect the intended purpose, i.e. the determination of erucic acid content.

68. The Committee, noting that the standard for low erucic acid rapeseed oil corresponded closely to rapeseed oil as regards the need for methods of analysis, agreed that the various methods proposed by the Commodity Committee should be temporarily endorsed. The reason for temporary endorsement was that the Commodity Committee was envisaging the review of the section on methods of analysis endorsed for fats and oils.

Edible Coconut, Palm, Palm Kernel, Rapeseed and Babassu Oils

69. The delegation of the United Kingdom indicated certain corrections to the various methods included in these standards. For similar reasons as those given in connection with low erucic acid rapeseed oil, the Committee agreed to temporarily endorse the proposed methods of analysis.

Bouillons and Consommés

70. The Committee agreed that it was not in a position to consider the proposed methods, in the absence of descriptions of the principles of methods proposed, and evidence that the methods proposed had been collaboratively tested and the methods corresponded to the criteria laid down by the Committee for the selection of Codex methods. The endorsement of the methods was postponed.

General Considerations

71. The Committee noted that the use of commas to denote three orders of magnitude (e.g. 1,000) was to be discouraged as this could lead to misunderstanding. Concern was expressed about the procedure followed by some Codex Committees in including provisions for methods of analysis at such a late step in the Procedure as to make it impossible for Governments to comment on the proposed methods of analysis. The Secretariat pointed out that it was not the intention that methods of analysis should not be subject to comments and expressed the opinion that, in such cases, the Committee should feel free to postpone endorsement pending the receipt of government comments. In reply to a question, the Secretariat informed the Committee that it was possible for the Committee to withdraw endorsement of a given method.

ISO/AOAC Method for the Determination of Nitrogen in Foods for Infants and Children

72. The representatives of ISO and AOAC informed the Committee that their Organizations would take into consideration the new classification of Codex methods of analysis (i.e. Types I, II, III and IV) in arriving at a jointly agreed method for the determination of nitrogen. The Committee looked forward to receiving such joint recommendations which would serve as a basis for the establishment of Codex methods.

73. It was noted that the use of mercury as a catalyst had been discontinued in a number of countries. The Committee agreed that the use of mercury as a catalyst for a Codex general reference method would not cause difficulties if used only as a defining method. It also noted that for specific purposes the use of potassium and copper sulphates as catalysts might be adequate.

1/ Note by the Secretariat: A joint IDF/ISO/AOAC method has already been developed as reference method for the determination of protein content in caseins and caseinates (Report of the 18th Session of the Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products (CX/70-18th, Appendix IX-D).

COOPERATION AND COLLABORATION BETWEEN INTERNATIONAL ORGANIZATIONS IN DEVELOPING METHODS FOR CODEX PURPOSES

74. The Committee had before it a report of an inter-agency secretariat meeting which had taken place 29-30 June 1979.

75. The delegate of ISO informed the Committee that representatives of several international organizations had participated in discussions on a table (Appendix II of the above report) showing a list of methods yet to be developed for inclusion in various Codex standards derived from CX/GEN 79/1. The table had been a compendium of methods of analysis contained in Codex standards and their state of endorsement. Those organizations specialized in developing methods for particular commodities had accepted the role of "lead organizations" in the coordination of the preparation of methods as shown in Appendix II of the above report. At the same time it was decided that assignment of priorities to various methods would not be practical because there was no direct relationship between Codex requirements and current laboratory work.

76. The Committee recognized that good progress had been made in establishing working relationships between the organizations concerned.

77. The delegation of Australia referred to document CX/GEN 79/1 and to a compendium of Codex methods prepared by Australia and proposed that it would be eventually necessary to examine these documents in order to see which methods required attention by the Committee and which methods required elaboration by the collaborating international organizations. The observer from NMKL (Nordic Committee on Food Analysis) noted with surprise that an invitation to this meeting had not been issued to their organization which represented five member countries having established official collaboration in the work of elaboration and harmonization of methods of analysis for food.

Other Business

78. The delegate of Czechoslovakia introduced a document distributed at the session concerning the standard layout for Codex methods of analysis. The Committee thanked the delegate of Czechoslovakia and agreed that the Codex standard layout should be updated at the 12th Session of the Committee in the light of the new revised ISO Guide 18 drawn up by ISO/TC 47 - Chemistry.

79. The representative of the EEC informed the Committee that the Community had promulgated a series of methods of analysis for sugars for human consumption, preserved milk and coffee extracts. These methods were considered as Community methods to be used in all cases; nevertheless analysts wishing, for reasons of convenience, to use simpler methods can do so. This option cannot prejudice results which would be obtained by the official method.

80. In answer to a question from the delegate of Australia as to the progression of Codex work on sampling, the Secretariat stated that the Codex Committee on Methods of Analysis and Sampling should establish without delay general principles on the basis of which Codex methods of sampling could be established. Plans could then be developed by the Codex Commodity Committees with the assistance of this Committee.

Future Work

81. The Committee noted that it had sufficient future work in the field of sampling, the review of methods of analysis in relation to the newly established classification and the endorsement of methods of analysis and sampling.

Date and Place of Next Session

82. The Committee was informed that the next session of the Committee would be held during the first half of 1981 in Budapest.

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APPENDIX I

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REVISED TERMS OF REFERENCE OF THE CODEX COMMITTEE ON METHODS
OF ANALYSIS AND SAMPLING AND CONSEQUENTIAL CHANGES IN THE
PROCEDURAL MANUAL INCLUDING DEFINITION AND CLASSIFICATION
OF METHODS OF ANALYSIS

Editorial Changes to the Terms of Reference

- (a) To define the criteria appropriate to Codex Methods of Analysis and Sampling;
- (b) To serve as a coordinating body for Codex with other international groups working on methods of analysis and sampling;
- (c) To specify, on the basis of final recommendations submitted to it by the other bodies referred to in (b) above, reference Methods of Analysis and Sampling appropriate to Codex Standards which are generally applicable to a number of foods;
- (d) To consider, amend, if necessary, and endorse, as appropriate, methods of analysis and sampling proposed by Codex (Commodity) Committees, except that methods of analysis and sampling for pesticide residues in food, the assessment of micro-biological quality and safety in food, the assessment of specifications for food additives, and those methods elaborated by the Joint FAO/WHO Committee of Government Experts on the Code of Principles concerning Milk and Milk Products, do not fall within the terms of reference of this Committee;
- (e) To elaborate sampling plans and procedures, as may be required;
- (f) To consider specific sampling and analysis problems submitted to it by the Commission or any of its Committees.

REVISION OF TEXT PROPOSED FOR PARAGRAPH 13(c)(i) OF THE PROCEDURAL MANUAL (pages 63-64)

(c)(i) Normal Procedure

Subject to the provisions of sub-paragraph (v) below, when Codex Committees have included provisions on methods of analysis and sampling in a Codex commodity standard, these should be referred to the Codex Committee on Methods of Analysis and Sampling at Step 4, to ensure Government comments at the earliest possible stage in the development of the standard.

A Codex Committee should, whenever possible, provide to the Codex Committee on Methods of Analysis and Sampling information, for each individual analytical method proposed, relating to specificity, accuracy, precision (repeatability, reproducibility), limit of detection, sensitivity, applicability and practicability, as appropriate.

Similarly a Codex Committee should, whenever possible, provide to the Codex Committee on Methods of Analysis and Sampling information for each sampling plan relating to the scope or field of application, the type of sampling (e.g. bulk or unit), sample sizes, decision rules, details of plans (e.g. "Operating characteristic" curves), inferences to be made to lots or processes, levels of risk to be accepted and pertinent supportive data.

Other criteria may be selected as required. Methods of analysis should be proposed by the Commodity Committees in consultation if necessary with an expert body.

At Step 4 Codex Commodity Committees should discuss and report to the Codex Committee on Methods of Analysis and Sampling on matters connected with:

- Provisions in Codex standards which require analytical or statistical procedure;
- Provisions for which elaboration of specific methods of analysis or sampling are required;
- Provisions which are defined by the use of Defining Methods (Type I);

1/ Main editorial change to the text agreed to by the 12th Session of the Codex Alimentarius Commission.

APPENDIX II

- All proposals to the extent possible should be supported by appropriate documentation; especially for Tentative Methods (Type IV);
- Any request for advice or assistance.

The Codex Committee on Methods of Analysis and Sampling should undertake a coordinating role in matters relating to the elaboration of Codex methods of analysis and sampling. The originating committee is, however, responsible for carrying out the Steps of the Procedure.

When it is necessary, the Codex Committee on Methods of Analysis and Sampling should try to ensure elaboration and collaborative testing of methods by other recognized bodies with expertise in the field of analysis.

Proposed Amendment to

GENERAL PRINCIPLES FOR ESTABLISHMENT OF CODEX METHODS OF ANALYSIS AND SAMPLING

1. Purpose of Codex Methods of Analysis and Sampling

The methods are primarily intended as international methods for the verification of provisions in Codex standards. They should be used for reference, in calibration of methods in use or introduced for routine examination and control purposes.

2. Methods of Analysis

(A) Definition of types of methods of analysis

(a) Defining Methods (Type I)

Definition: A method which determines a value that can only be arrived at in terms of the method per se and serves for calibration purposes.

Examples: Howard Mould Count, Reichert-Meissl value, loss on drying, salt in brine by density.

(b) Reference Methods (Type II)

Definition: A Type II method is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below). It should be recommended for use in cases of dispute and for calibration purposes.

Example: Potentiometric method for halides.

(c) Alternative Approval Methods (Type III)

Definition: A Type III Method is one which meets the criteria required by the Codex Committee on Methods of Analysis and Sampling for methods that may be used for control, inspection or regulatory purposes.

Example: Volhard Method or Mohr Method for chlorides.

(d) Tentative Method (Type IV)

Definition: A Type IV Method is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Codex Committee on Methods of Analysis and Sampling have not yet been determined.

Examples: chlorine by X-ray fluorescence, estimation of synthetic colours in foods.

(B) General Criteria for the Selection of Methods of Analysis

- (a) Official methods of analysis elaborated by international organizations occupying themselves with a food or group of foods should be preferred.
- (b) Preference should be given to methods of analysis the reliability of which have been established in respect of the following criteria, selected as appropriate:
 - (i) specificity
 - (ii) accuracy
 - (iii) precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories)
 - (iv) limit of detection
 - (v) sensitivity
 - (vi) practicability and applicability under normal laboratory conditions
 - (vii) other criteria which may be selected as required.
- (c) The method selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use.
- (d) All proposed methods of analysis must have direct pertinence to the Codex Standard to which they are directed.
- (e) Methods of analysis which are applicable uniformly to various groups of commodities should be given preference over methods which apply only to individual commodities.

(The above text is to replace paras 1 and 2 on pages 69-70 of the Procedural Manual, 4th Edition.)

3. Methods of Sampling

(To be elaborated)

REPORT OF THE AD HOC WORKING GROUP
ON CODEX METHODS OF SAMPLING

1. At the 11th Session of the Codex Committee on Methods of Analysis and Sampling (Budapest, 2-6 July 1979) an ad hoc Working Group was convened to discuss Agenda Item 6. The Working Group had before it documents CX/MAS 79/3, CX/MAS 79/4, CX/MAS 79/4 - Add. 1, CAC/RM 42-1969, ALINORM 78/23, App. III, II.3 and CX/MAS 77/2 - Add. 1.

2. Membership of the Working Group was as follows:

Hungary	L. Kőrmendy
Netherlands	M. Osse
Norway	A. Hougen
Switzerland	P. Koch
United Kingdom	R. Player (Rapporteur)
USA	B. Levy (Chairman)
	C. Brokaw
	J. Winbush
	W. Dubbert
EEC	F. Wolf
	Mlle O. Demine
IUPAC	K. Vas
FAO	L. Ladomery

3. The representative of FAO summarized the long history of sampling within the context of the Codex Alimentarius and the many issues which had yet to be resolved. After preliminary discussion the Working Group concluded that formulation of general principles for the selection of Codex procedures was not immediately possible until agreement on several fundamental issues had been reached. Amongst the foremost of these were:

- (a) Was there a real requirement to harmonize principles of sampling internationally via the Codex forum? The Working Group agreed that there was a need for such work to continue;
- (b) What status should Codex sampling procedures hold within a standard? The Working Group decided that in a similar way to decisions taken concerning methods of analysis, sampling plans should not be considered as "referee" but "reference" procedures, i.e. their application should be possible in routine day-to-day sampling situations and that any cases of dispute which arose would need separate consideration. The need was pointed out for a clear understanding of the term "sampling" which was considered by the Working Group to encompass all stages of the procedure from selection of items to evaluation of sample results and risks and including the discrete item termed the "sampling plan" which dealt solely with the statistically based numerical procedure. Although only this last-mentioned item was deemed to be strictly relevant to the discussions within the Working Group, it was realized that the many other stages of "sampling" in general would need consideration and guidelines or recommendations for other Committees formulated for these stages as well as the more limited area of sampling plans;
- (c) How should limit provisions in standards be interpreted with regard to the results obtained by reference sampling plans? The Working Group noted that this problem had been documented previously (CX/MAS 79/4, para 17 and CX/MAS 79/4 - Add. 1, App. I). Because of differing national legislation philosophies no agreement could be reached;

APPENDIX III

(d) Should the Codex Committee on Methods of Analysis and Sampling (CCMAS) be responsible for the formulation of sampling plans for other Committees? The Working Group considered that it was not possible for them to formulate sampling plans for individual commodities but that this required the expertise and knowledge of the particular commodity to be found in the relevant Codex Commodity Committee. The Working Group wished to emphasize the necessity of two-way communication between CCMAS and Commodity Committees so that, with suitable guidance in the form of required criteria for all sampling plans, the Commodity Committees themselves could discuss and select a plan which would be suitable for each individual case. CCMAS would then discuss such selected plans with a view to endorsement. This procedure, it was noted, was little different to that followed for methods of analysis.

4. In discussion of working papers CX/MAS 79/4 and CX/MAS 79/4 - Add.1, the Working Group wished to record the usefulness of the two documents. In the former paper, paragraphs 44-49 were generally supported as were all but one of the recommendations of the latter paper (page 5). The exception was recommendation 2 where no consensus could be reached as to the question of whether the final sample always should or should not relate to the lot from which it was drawn. However all conclusions and recommendations of the two working papers highlighted many aspects which will need consideration in the future discussions of Codex sampling procedures and the Working Group recommended that the information contained within these papers be drawn upon in future.

5. The Working Group noted that, in consequence of its decision that Codex Sampling Plans should be considered reference procedures (see para 3(b)), confusion may arise in the acceptance by Governments of standards in which are cited or referred to previously published Codex sampling plans. It was thought unclear which sections of sampling plans were presently considered mandatory: e.g. in the Sampling Plans for Prepackaged Foods (AQL 6.5) (CAC/RM 42-1969) are both the relationship between acceptance number (c) and sample size (n) and also between sample size (n) and lot size (N) mandatory and therefore subject to acceptance or otherwise by Governments? The Working Group considered that clarification from the Commission should be sought.

6. The Working Group discussed the terms of reference for CCMAS (CX/MAS 79/3) adopted by the Commission and found them satisfactory as regards sampling aspects. It was noted however that the terms of reference as written implied equal importance for both methods of analysis and sampling whereas arrangements for discussion of each topic were not equal. It was considered that, if future progress was to be made in the area of sampling, a more formalized approach than the present convening of ad hoc Working Groups may be necessary. The present Group was of the opinion that greater support from CCMAS and FAO/WHO Secretariats in terms of meeting facilities and prompt distribution of working papers would be necessary for sound progress to be made.

7. The Working Group concluded that, to be able to prepare complete and worthwhile general guidance for other Committees and governments it would be necessary to consider each stage of sampling in some depth and if necessary request responses from Governments and Commodity Committees to fundamental questions. This future work is detailed below.

Recommendations for further action

8. (a) Definition of terms used in sampling;
- (b) Guidelines on administrative aspects of sample taking;
- (c) Guidelines on methods of sample taking in order to ensure that the sample is representative of the lot;
- (d) Guidelines on resampling in cases of international disputes concerning compliance with Codex standards, etc.
- (e) General Principles for the establishment or selection of Codex Sampling Plans;

APPENDIX III

- (f) Elaboration of sampling plans for various purposes, e.g. bulk and unit sampling; sampling to check compliance with quality criteria, e.g. visual defects and analytical criteria (minima and maxima); health criteria, e.g. food additives, contaminants and nutritional provisions; metrological criteria, e.g. net weight, drained weight, minimum fill, etc.;
- (g) Revision of existing sampling plans in Codex standards in order to ensure consistency of approach;
- (h) Clarification of the meaning of provisions in Codex standards in relation to lots (i.e. recommendation on how to relate results of sampling and analysis to the acceptance or rejection of lots).

9. It was also agreed that work under (a) to (e) would be the responsibility of the CCMAS, while work under (f) to (h) would be initiated by the appropriate Codex Commodity Committees and carried out in consultation with the Codex Committee on Methods of Analysis and Sampling.

10. Concerning Agenda Item 6(b)(i) relating to a Moderate Acceptance Probability Plan, the Working Group believed that the proposed plan contained considerable technical and practical merit. During the interval between the 10th and 11th Sessions of the Codex Committee on Methods of Analysis and Sampling, a questionnaire was to have been prepared and circulated to all member countries to determine the general acceptability of the plan. This questionnaire, unfortunately, had not been circulated and hence the general acceptability of the plan was still unknown. Since this information is crucial to a decision the Working Group again recommended preparation and circulation of a questionnaire.

11. In consideration of Agenda Item 6 (b)(ii) (Revised Sampling Plans for Prepackaged Foods (AQL. 6.5), the Working Group had before it working paper CX/MAS 77/2, Add. 1. After discussion of the four points raised by the delegation of the USA in this paper, it was decided:

- (a) concerning point 1: - the diagram in Appendix III of CAC/RM 42-1969 was incorrect and should be replaced by a corrected curve;
- (b) concerning point 2: - the Working Group found some difficulty in making recommendations on the proposed amended sample sizes and acceptance numbers. The effect of the reduced sample sizes and associated changes in acceptance numbers could not be computed during the Session by the Working Group who undertook to do this at the earliest occasion. However it was thought that any such effects (e.g. confidence level) that arose by the application of the amended provisions should be left to the Commodity Committee to assess and decide whether the benefit of reducing sample size outweighed the consequent effect in confidence.
- (c) concerning point 3: - the Working Group wished to stress that caution should be exercised when resampling is considered as a result of a dispute over results from a primary sampling of a lot (see also paragraph 8(d)). In general it is not justified to disregard the results of the primary sample; otherwise distortion of statistical considerations would result. It was considered that re-inspection and the disregarding of primary results should only take place where there was a likelihood that a substantive error had occurred in the first sampling procedure. The Working Group concluded that this principle should be made clear in the published sampling plans (CAC/RM 42-1969).

APPENDIX III

- (d) concerning point 4: - the Working Group wished to stress that, as stated in the working paper, the field of application of the published plans (CAC/RM 42-1969) is restricted. However it appears that the Sampling Plans have on occasion been used more widely than, or in circumstances outside of, the restricted field of application intended. This should urgently be drawn to the attention of Commodity Committees so that this apparent abuse of the Plans could be halted.

12. The Working Group noted that ISO document ISO/TC 34/WG 1 had been referred to it in the working paper by New Zealand. No copies of this ISO document were available to the Group at the Session but discussion of the document would be included in the future.

APPENDIX IVGENERAL REFERENCE METHOD FOR THE DETERMINATION OF CHLORIDES
(CALCULATED AS SODIUM CHLORIDE) IN FOOD

1. SCOPE AND FIELD OF APPLICATION

The method determines total halides (including bromides iodides), expressed as sodium chloride, in foodstuffs.

2. DEFINITION

The chloride content, expressed as sodium chloride: the content of halides, expressed as sodium chloride, as determined by the method specified. (Significant quantities of bromides and iodides are ordinarily absent in foods.)

3. PRINCIPLE

The product is dispersed with H₂O and acidified; soluble halides are titrated potentiometrically with AgNO₃. Applicable to levels \geq 0.03% NaCl. For convenience in calculations, masses, volumes and solution concentrations are specified so that 1 ml AgNO₃ = 0.1% NaCl. If balance permitting rapid weighing of specified weight is not available, convenient sample mass and strength AgNO₃ solution may be used.

4. APPARATUS

4.1 Balance: Capacity, \geq 200g, taring range, \geq 100 g, and calibrated to 0.01g.

4.2 Electrodes: Ag billet combination electrode or separate indicating Ag and glass reference electrodes. Before initial use and before each day's use, if necessary, clean Ag billet electrode tip with scouring powder or other suitable material and rinse thoroughly with H₂O. (Hot H₂O may be

required with some kinds of samples.) Clean other electrodes as recommended by manufacturer. Reclean as frequently as necessary to prevent drifting of end point reading. With some samples, periodically rinse electrodes with H_2O and wipe with tissue to prevent accumulation of film. It is unnecessary to coat Ag billet electrodes with AgCl.

- 4.3 Magnetic stirrer: Operating through variable transformer to permit range of speed which, once set, is constant.
- 4.4 pH meter: Preferably direct reading, with scale divisions 10 mv or less; range at least \pm 700 mv, e.g., digital type.

5. REAGENTS

Use analytical reagent grade chemicals unless otherwise indicated.

- 5.1 Nitric acid, dilute: (1+49). Dilute 20 ml HNO_3 to 1 L with H_2O .
- 5.2 Silver nitrate standard solution: 0.0856 mol/l (0.0856N). Dissolve 14.541 g $AgNO_3$ in H_2O and dilute to 1 L in volume flask. Standardize as in 6.1 and adjust to exact concentration specified so that with indicated sample weight 1 ml = 0.1% NaCl. Store in Pyrex container out of direct sunlight. The solution is stable in room light.
- 5.3 Sodium chloride standard solution: 0.0856 mol/l (0.0856 N). Dissolve in H_2O 5.000 g NaCl (if assay is <100.0% NaCl, divide 5.000 g by % NaCl/100 to obtain correct weight), previously dried 2 hours at 110° , and dilute to 1 L in volumetric flask.

APPENDIX IV

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

6. PROCEDURE

6.1 Standardization

Pipette 25 ml NaCl standard solution (5.3) into 250 ml beaker, dilute to ca 50 ml with H_2O , and add 50 ml HNO_3 (5.1). Insert electrodes, start magnetic stirrer, and stir throughout titration at constant rate producing vigorous agitation without splashing. Titrate with AgNO_3 standard solution (5.2), adjusting increments with rate of voltage change so that accurate plot of mv against ml AgNO_3 solution can be prepared. Add total of 50 ml AgNO_3 solution to obtain complete curve.

Determine inflection point by drawing two straight lines with 45° slope with respect to axes and tangent to titration curve at the two points of greatest curvature. Inflection point is at intersection of titration curve with line drawn parallel to and midway between other two lines. From volume AgNO_3 solution used, calculate concentration and adjust to 0.0856 mol/l. Re-standardize occasionally. Use inflection point as end point in titrating samples. Re-check end point potential occasionally, and re-determine when either individual electrodes, combination electrode, or pH meter is replaced by preparing new titration curve.

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

6.2 Preparation of Sample

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

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

6.2.3 General method for heterogeneous (fish, meat, etc.), low moisture (cereal products, etc.) and hard-to-disperse, homogeneous (cheese, peanut butter, etc.) foods: Weigh 50.0 g sample into 1 L (quart) container of high-speed blender and add 450 g H₂O. Cover, start blender at low speed by use of variable transformer for initial dispersion, and blend thoroughly at high speed (1-2 minutes is usually adequate). Equivalent of 5 g sample is conveniently dispensed through 50 ml pipette with tip cut off. Thoroughly mix sample suspension immediately before pipetting aliquot for analysis so that solid material is uniformly suspended.

6.2.4 Other types of foods: Prepare sample by method 6.2.1, 6.2.2 or 6.2.3 or other suitable method.

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

6.3 Determination

6.3.1 For products containing less than 5 per cent salt: Place 5.00 g (or 5.00 ml if concentration is to be expressed on m/v basis) sample from 6.2.1 or 6.2.2 or 50.0 g from 6.2.3 into tared 250 ml beaker; add H₂O to ca 50 ml if 6.2.1 or 6.2.2 is used. (Use boiling H₂O with samples such as butter to melt fat). Add 50 ml HNO₃ (5.1). Titrate as in 6.1 using 10 ml burette if salt content is $\leq 1\%$. Let the number of mls of titrant used be V₀.

6.3.2 For products containing 5 or more per cent salt: Place 5.00 g (or 5.00 ml if concentration is to be expressed on m/v basis) sample from 6.2.1 or 6.2.2 into 100 ml volumetric flask and dilute to volume with H₂O. Mix, and transfer aliquot containing 50-250 mg NaCl to 250 ml beaker. If sample is prepared by 6.2.3, transfer weighed aliquot containing 50-250 mg NaCl to tared 250 ml beaker. Proceed as in 6.1, beginning ".... dilute to ca 50 ml with H₂O...". Let the number of mls of titrant used be V₁.

6.3.3 General case: Accurately weigh approximately sample weight stated. (If % NaCl $\geq 5\%$, weigh $< 5g$ sample rather than diluting to 100 ml, if more convenient). Use ca 0.1 mol/l AgNO₃ solution, accurately standardized as in 6.1, without adjusting to specific normality, and titrate as in 6.1. Let the number of mls of titrant used be V₂.

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

7. EXPRESSION OF RESULTS

7.1 Formula and method of calculation

The chloride content, expressed as g sodium chloride in 100 g of the sample is given by:

7.1.1 $V_0/10$ in the case of products containing less than 5% salt (6.3.1)

7.1.2 $(V_1 \times F)/10$ in the case of products containing 5 or more per cent salt (6.3.2)

7.1.3 $(V_2 \times n \times 0.05844 \times 100)/g$ in the general case (6.3.3)

where:

V_0 is the number of ml of 0.0856 mol/l $AgNO_3$ used in the determination 6.3.1;

V_1 is the number of ml of 0.0856 mol/l $AgNO_3$ used in the determination 6.3.2;

F is the dilution factor used in determination 6.3.2 and is equal to 100/ml aliquot titrated if sample is prepared by 6.2.1 or 6.2.2 or is 50g aliquot titrated if sample is prepared by 6.2.3;

V_2 is the number of ml of $AgNO_3$ used in determination 6.3.3;

n is the concentration in mol/l of $AgNO_3$ in determination 6.3.3;

g is the sample weight used in determination 6.3.3.

7.2 Repeatability

The difference (r) in g per 100 g of sample between the results of two determinations carried out simultaneously or in rapid succession on the same sample by the same analyst under the same conditions shall not exceed $9.37 \times 10^{-3} \overline{NaCl} + 1.78 \times 10^{-3}$ where \overline{NaCl} is the average of the two determinations expressed in g per 100 g of sample (see note 9.1).

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7.3 Reproducibility

The difference (R) in g per 100 g of sample between the results of two determinations carried out on the same sample in different laboratories by different analysts shall not exceed $1.68 \times 10^{-2} \overline{\text{NaCl}}$ + 1.48×10^{-2} where $\overline{\text{NaCl}}$ is the average of the two determinations expressed in g per 100 g of sample (see note 9.1).

8. REFERENCES

8.1 This method has been collaboratively tested; the results of the trial were published in JAOAC 1971 54, 471 and JAOAC 1974, 57, 1209.

9. NOTE

9.1 The repeatability and reproducibility criteria are calculated from the results of the collaborative trial (8.1) according to the appropriate procedure described in ISO 5725. These criteria are only to be used as guidelines; multiple analysis and continuous testing are required to ensure that laboratories are demonstrating repeatability and reproducibility at a minimum equal to that stated in Sections 7.2 and 7.3.

Table of Status of Endorsement of Methods of Analysis

Natural Mineral Water (See Annex I)

		Type	
3.1.1	Total Dissolved Solids	Temporarily endorsed	I
3.1.2	Dry residue	Temporarily endorsed	IV
3.1.3			
3.1.4	Sum of anions and	Temporarily endorsed but	
3.1.5	cations	see para (50)	IV
3.1.6			
3.2	Free Carbon Dioxide	Temporarily endorsed but	IV
		see para (47)	
3.3.1	Total organic Matter	Temporarily endorsed	I
3.3.2	Phenolic compounds	See para (47)	
3.3.3	Surface active agents	See para (47)	
3.3.4	Pesticides and PcBs	See para (47)	
3.3.5	Mineral Oil	See para (47)	
3.3.6	Polynuclear aromatic hydrocarbons	See para (47)	
3.3.7	Ra activity and Total beta activity	Temporarily endorsed	IV

Quick Frozen Foods

Draft Standard for Quick Frozen Brussels Sprouts

8.2	Thawing Procedure	Endorsed	
8.3	Cooking Procedure	Endorsed	
8.4	Determination of Net Weight	Endorsed	I

Draft Standard for Quick Frozen Green Beans and Quick Frozen Wax Beans

8.5	Determination of Tough Strings	Endorsed	I
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Type

Draft Standard for Quick Frozen Carrots

8.4.2 Mineral impurities Endorsed I

Recommended Standard for Quick Frozen Spinach

8.1 Sampling Endorsement postponed

8.2 Thawing Procedure Endorsed

8.3 Determination of Net Weight Endorsed I

8.4 Determination of Salt-Free Dry Matter Endorsed I

Dry Matter

Editorial amendment to 8.4.1

delete "over" insert "with"

Editorial amendment to 8.4.2

add reference: 32.048 A 12th ed

AOAC 1975.

8.5 Determination of Mineral Impurities Endorsed I

Editorial amendment:

SI units

Processed Fruits and Vegetables

Draft Standard for Pickled Cucumbers

9.2.1 Soluble solids Endorsed I

Editorial amendments: add

corrected to 20^oC. Update

references to AOAC

9.2.2 Salt (NaCl) Endorsed I

Committees attention drawn to

Codex General method.

9.2.3 Total Acidity Endorsed I

9.2.4 Mineral Impurities Endorsed I

9.2.5 Water Capacity of Containers Endorsed II

9.2.6 Water capacity of Containers Endorsed II

See para (54) on need for

three methods.

Draft Standard for Canned Carrots

Type

8.2	Determination of Drained Weight	Endorsed	I
8.3	Determination of Water Capacity of Containers	Endorsed	II

Draft Standard for Dried Apricots

8.2.1	Moisture	Ovendrying method endorsed (paras 56-58)	I
	Editorial amendment instrumental method to be added as footnote	Endorsed	
8.2.2	Sulphur dioxide	Endorsed	III
8.2.3	Broken Slabs, etc.	Endorsed	I

Draft Standard for Dates

8.1	Sampling	Endorsement postponed	
8.2.1.1	Moisture content	Endorsed	I
8.2.1.2	Moisture content (Instrumental method as footnote)	Endorsed	I
8.2.2	Internal defects	Endorsed	I

Draft Standard for Unshelled Pistachio Nuts

8.1	Sampling	Endorsement postponed	
8.2.1	Moisture	Endorsed	I
8.2.2	Specific Defects	Endorsed	I
8.2.3	Size Classification	Endorsed	I

Draft Standard for Canned Apricots

8.2	Determination of Drained Weight	Endorsed	I
8.3	Syrup measurements	Endorsed	I

Processed Meat and Poultry ProductsCanned Corned Beef

7.2	Nitrite	Temporarily endorsed see para (61)	IV
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Luncheon Meat

7.2	Nitrite	Temporarily endorsed see para (63)	IV
7.6	Glutamic acid	Temporarily Endorsed see para (63)	IV
7.8	Glucono-delta-lactone		

Cooked Cured Ham

7.8	Glutamic acid	Temporarily endorsed see para (63)	IV
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Fats and OilsDraft Standard for Reduced Fat Margarine

All methods	Endorsement postponed para (64)
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Draft Standard for Edible Low Erucic Acid Rapeseed Oil

All methods	Temporarily endorsed paras (65-68)
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Draft Standard for Edible Coconut Oil

8.1	Relative density	Temporarily endorsed
8.6	Reichert and Polenske value Other methods	Temporarily endorsed Temporarily endorsed

Draft Standard for Edible Palm Oil

8.1	Relative density at 50 ^o C	Temporarily endorsed
8.9	Determination of Carotenoids Other methods	Temporarily endorsed Temporarily endorsed

Draft Standards for Edible Palm Kernel,Grapeseed and Babassu Oils

All methods	Temporarily endorsed
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Draft Standard for Bouillons and Consommés

All methods	Endorsement postponed para (70)
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ANNEX I

3. PROPOSED METHODS OF SAMPLING AND ANALYSIS FOR MINERAL WATERS

D 3.1 Total dissolved solids(salts) (para 7.1.1 of the Standard)

3.1.1 Principle: During evaporation, the different constituents undergo considerable modification. This is especially the case with alkaline-earth hydrogencarbonates, which are partly or wholly converted into carbonates and free carbon dioxide (a deficiency of 20 to 30 percent may easily occur). However, it is fairly easy to compensate for this change by calculation. Even though this procedure does not guarantee absolute accuracy (as other substances also undergo alteration), it allows for a simplification.

3.1.2 The analytical procedure is relatively simple:

i) determination of dry residue (evaporation of 100 ml of water and drying at 240°C for calcic sulphated waters or at 180°C for the others);

ii) determination of alkalinity (HCO_3^-);

iii) on the basis of these values, calculation of decomposed hydrogencarbonates:

$$\text{Total dissolved solids} = \frac{\text{dry residue} + \text{HCO}_3^-(\text{mg/l})}{2}$$

(mg/l) (mg/l)

(Reference: HLCh)

3.1.5 A shortened analysis involving the determination of at least nine constituents (sodium, potassium, magnesium, calcium, chlorides, sulphates, hydrogencarbonates, nitrate, silicic acid) may be used optionally for comparison or as a first estimate of total dissolved solids. Depending on the nature of the mineral water, it may also be necessary to determine other constituents if present in quantities over 10 mg/kg.

3.1.6.1 Determination of Cations

A. Normal cations

* (a) Ammonium: Photometry using Nessler reagent or Indophenol directly or after distillation of NH_3 with Na_2CO_3 (Schweizerisches Lebensmittelbuch, DEV).
Sensitivity: Approx. 5 microgrammes per litre.

* (b) Lithium
AAS or emission spectrometry with acetylene/air flame. Large quantities of sodium (mostly as chloride) interfere with the determination. Most of the sodium chloride will be precipitated using HCl + alcohol. Lithium remains in solution.

(c) Sodium
AAS or emission spectrometry.

D = defining method

* = no provision in the standard

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- * (d) Potassium
 - (i) as for lithium and sodium
 - (ii) gravimetry as potassium tetraphenylborate
(P. Raff and W. Brotz, Zeitschrift für analytische Chemie, 133, 241, 1951).
 - * (e) Magnesium
AAS
 - * (f) Calcium
AAS
(Robinson J.W. - Atomic Absorption, London 1966).
 - * (g) Strontium
AAS
 - (h) Iron
Fe⁺⁺ ion using 2,2'-dipyridyl. Where both Fe⁺⁺ and Fe⁺⁺⁺ are present, the latter is brought into solution using acid and converted to Fe⁺⁺ with ascorbic acid
(Fresenius und Schneider, Z. Analyt. Chem. 209, 340, 1965, HLCh).
 - (i) Manganese
Photometric determination as permanganate (oxidation of Mn⁺⁺ to Mn₄⁺ by persulphate
(Gottschalk, Z. Analyt. Chem. 212, 303, 1965, HLCh, DEV)
 - * (j) Aluminium
Photometry with aluminon (Ammonium aurinthrincarboxylate)
(G. Giebler, Z. Analyt. Chem. 184, 401, 1961, HLCh)
- B. Heavy Metals for which a limit has been laid down in the Standard.
- (a) Lead
 - (i) AAS
 - (ii) By differential pulse stripping voltametry
(P. Klahre, P. Valenta u.W. Nürnberg "Vom Wasser" 51, 199-219, 1978)
 - (b) Copper
 - (i) AAS
 - (ii) Method as for Lead (P. Klahre)
 - (c) Manganese
(see 3.1.6.1.A.(i))
 - (d) Zinc
 - (i) AAS
 - (ii) Method as for Lead (P. Klahre)
 - (e) Cadmium
 - (i) Extraction of Cd-dithizonate with chloroform and determination by flameless AAS
(Handbuch der Spurenanalyse 1974)
 - (ii) Method as for Lead (P. Klahre)

(f) Barium

AAS

(g) Chromium

(3⁺ and 6⁺) (Provisional method), see 1)

Photometry with diphenylcarbazide
(ASTM, HLCh, DEV)

(h) Mercury

Cold vapor atomic absorption after enrichment.

NB. Atmospheric Hg interferes during analysis and should be
excluded.

3.1.6.2 Determination of anions

(a) Chloride

(i) Potentiometry with AgNO₃ (General Codex Method)
(App. IV, ALINORM 76/23)

(ii) Titrimetric method using AgNO₃ (DEV)

Note: Methods (i) and (ii) determine both bromide and iodide ions
and should be corrected for.

(b) Bromide

Iodometric, according to Höfer (determines both bromide and iodide
separately).

(P. Höfer, Gesundheitsingenieur, 74, 224, 1953)

(c) Iodide

(i) As (b) above

(ii) Iodometric after oxidation with bromine
(P. Höfer, Gesundheitsingenieur, 72, 277, 1951)

(d) Fluoride

(i) Ion selective electrode method
(Annual Book of ASTM Standards, 1973 and Camman K., Das Arbeiten
mit ionenselektiven Elektroden; Springer Verlag, 1973)

(ii) Photometry
(E. Quentin, A. Rosopulo: Lanthan Alizarin Komplexen,
Z.Analyt.Chemie 241 (1968), 241-250, HLCh)

(e) Sulphate

Gravimetric method using BaSO₄ (DEV, HLCh)

(f) Carbonate and hydrogencarbonate

titration with HCl to pH 8,3 followed by titration to pH 4,4
(AOAC, 1970) XI, 33.017)

* (g) Silicic Acid (SiO₂)

(AOAC, 1970, XI, 33.018)

1) Provision in the standard refers to Cr⁶⁺ only.

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(h) Nitrate

Photometry:

- (i) with 1,2,6-xylenol (DEV)
- (ii) with sodium salicylate (HLCh)

(i) Nitrite

Photometry:

- (i) with sulphanilamide and N-(1-Naphthyl)ethylenediamine (DEV)
- (ii) with alpha-naphtylamine (HLCh)

(j) Cyanide

Photometry with:

barbituric acid-pyridine (DEV)

(k) Sulphide

Photometry:

Ethylene - blue method (with p. aminodiethylaniline) (HLCh)

(l) Borate (boric acid)

Photometry with:

1,1'-dianthrimide
(Handbuch der Spurenanalyse, 1974, HLCh)

(m) Hydrogenphosphate

Photometry: extraction of phosphor molybdenblue with organic solvent and photometric determination (DEV)

(n) Hydrogenarsenate (Arsenic)

- (i) Flameless atomic absorption after reduction with sodium borohydrate. (M.Dujmovic, C-I-T. Fachzeitschrift f.d. Laboratorium, 4. April 1976, 336)
- (ii) Photometry after reduction to AsH₃ with silver diethyldithiocarbamate (Fresenius und Schneider, Zeitschrift Analyt.Chem. 203, 417, 1964)

(o) Selenium

Flameless atomic absorption (as for Arsenic)

(L. Henn: Analyt.Chem. 47, 1975, 428-432; D.Dujmovic: as for Arsenic)

3.2 Free carbon dioxide (Para 7.1.1. of the standard)

The free carbon dioxide can be determined by calculation; difference between the total carbon dioxide content minus carbon dioxide from carbonates and hydrogencarbonates:

- (i) Total carbon dioxide is determined by back titration with HCl to pH 8,3 after addition of excess sodium hydroxide.
- (ii) At low value, direct titration with sodium hydroxide to pH 8,3 is applicable.

3.3 Other Substances for which Limits are laid down in the Standard

Sensitivity of methods 3.3.1 to 3.3.6 should be mentioned so they can be applied to limits laid down in the standard for these substances.

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- D 3.3.1 (a) Organic Matter (Para 3.2 of the standard)
KMnO₄ method (DEV, HLCh)
- D 3.3.2 Phenolic compounds: (Method to be also considered by the Codex Committee on Food Additives)
Photometric determination after distillation and reaction with 4-amino antipyrine in presence of potassium ferricyanide (HLCh, ASTM).
- D 3.3.3 Surface active agents: (Method to be also considered by the Codex Committee on Food Additives)
(i) Anionic
Photometry with methylene blue (DEV, ASTM, HLCh)
(ii) Cationic
Photometry with bromophenol blue (DEV)
(iii) Non ionic
(R. Wickbold: Tenside, Detergents, 10 (1973), 1979-182; W. Leithe: Die Analyse der organischen Verunreinigungen in Trink-, Brauch- und Abwässern; Wissenschaftl. Verlags-GesmbH., Stuttgart (1972)).
- D 3.3.4 Pesticides and PCBs: (Methods to be also considered by the Codex Committee on Pesticide Residues)
(i) Individual organochlorine pesticides are determined quantitatively, after extraction with hexane, cleaning up, column chromatography, the GC analysis is carried out by electron capture detector (Weil L., Quentin K.E.; Zur Analytik der Pesticide im Wasser, VIII. Wasser- und Abwasserforschung 7, (1974) 147-152)
(ii) Polychlorbiphenyl (PCBs)
Gas chromatography (Schulte E., Acker L., Zeitschrift Analyt. Chemie 268 (1974), 260-267)
- D 3.3.5 Mineral oil: (Method to be also considered by the Codex Committee on Food Additives)
Method by gas chromatography (International Standards for Drinking Water, Third Ed., 1971, Table 3, page 37) which refers to L.R. Beynon, R. Kashnitz and G.W.A. Lijnden, 1968: Methods for the Analysis of Oil in water and soil; (The Hague, Stichting, Concawe).
- D 3.3.6 Polynuclear aromatic hydrocarbons (PAH): (Method to be also considered by the Codex Committee in Food Additives)
Extraction by organic solvent, separation by TLC and measuring fluorescence of individual PAH (DEV, preprint in "Vom Wasser", 47, 1976, Verlag Chemie (Weinheim). Woidich, H. et al. Chromatographia 10, 1977, No. 3, 140-146; Lebensmittel-Chemie und gerichtliche Chemie, 30, 1976, 8, 141-160).

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3.3.7 226 Ra activity and Total beta-activity: (Method to be also considered by the Codex Committee on Food Additives; and to be checked out with IAEA)

(i) 226 Ra; (a) WHO Method I, Methods of Radiochemical Analysis, WHO 1966, page 117). (WHO Method II is not specific to 226 Ra);

(b)(DEV, HLCh)

(ii) Total beta activity (except ^{40}K and ^3H);

a) Radiological examination, by N.T. Mitchell, Section 3.2, Gross beta activity, in Water Analysis for Water Pollution Control, edit. by M.J. Swess, WHO, Publisher: Pergamon Press, Oxford (1980);

b)(DEV, HLCh)

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 - 3) Handbuch der Lebensmittelchemie (Gesamtred: J. Schormüller), Band VIII, Teil 1 und 2, Wasser und Luft (S.W. Souci and K.E. Quentin), Springer-Verlag, 1969, (abb.HLCh).
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