

codex alimentarius commission

FOOD AND AGRICULTURE
ORGANIZATION
OF THE UNITED NATIONS

WORLD HEALTH
ORGANIZATION

JOINT OFFICE: Via delle Terme di Caracalla 00100 ROME Tel.: 57971 Telex: 625852-625853 FAO I Cables: Foodagri Rome Facsimile: (6) 57973152-5782610

ALINORM 93/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Twentieth Session
Geneva, 28 June - 7 July 1993

**REPORT OF THE EIGHTEENTH SESSION OF THE
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Budapest, 9-13 November 1992**

Note: This report incorporates Codex Circular Letter CL 1992/34-MAS

W/Z 9740

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CX 4/50.2

CL 1992/34-MAS
November 1992

TO: - Codex Contact Points
- Participants at the Eighteenth Session of the Codex Committee
on Methods of Analysis and Sampling
- Interested International Organizations

FROM: Chief, Joint FAO/WHO Food Standards Programme, FAO,
Via delle Terme di Caracalla, 00100 Rome, Italy

SUBJECT: Distribution of the Report of the Eighteenth Session of the Codex
Committee on Methods of Analysis and Sampling (CCMAS)

The report of the Eighteenth Session of the above Committee (ALINORM 93/23) will be considered by the Twentieth Session of the Codex Alimentarius Commission (Geneva, 28 June - 7 July 1993).

PART A: MATTERS FOR ADOPTION BY THE COMMISSION

The following matters will be brought to the attention of the 20th Session of the Codex Alimentarius Commission for adoption:

- (i) The new terms of reference as proposed by the Codex Committee on General Principles and as amended by the 18th Session of the CCMAS (para. 30, ALINORM 93/23);
- (ii) The recommendation for a checklist of information required to evaluate methods of analysis submitted to the CCMAS for endorsement (paras. 39, 94 and Appendix II, ALINORM 93/23);
- (iii) Precision criteria on methods of analysis submitted for endorsement and guidelines for use by the CCMAS (paras. 39, 94 and Appendix III, ALINORM 93/23);
- (iv) Endorsement of Methods of Analysis

The Committee agreed to endorse the provisions concerning methods of analysis in a large number of Codex standards (para. 69 and Appendix V, ALINORM 93/23).

Governments wishing to propose amendments or to submit comments regarding the implications which the above matters may have for their economic interest should do so in writing in conformity with the Codex Procedure, to the Chief, Joint FAO/WHO Food Standards Programme, FAO, Via delle Terme di Caracalla, 00100 Rome, Italy, no later than 31 May 1993.

PART B: REQUEST FOR COMMENTS AND INFORMATION

- (i) Review of General Methods for Contaminants (para. 73, ALINORM 93/23)

The Delegation of Canada agreed to prepare a paper for the next session of the CCMAS relating the government comments to the specific methods so that a decision could be made on the acceptance of the proposed test methods.

SUMMARY AND CONCLUSIONS

The Eighteenth Session of the Codex Committee on Methods of Analysis and Sampling reached the following conclusions:

MATTERS FOR CONSIDERATION BY THE COMMISSION:

- Recommended the adoption of its terms of reference as follows:
 - (a) to serve as a coordinating body for Codex with other international groups working in methods of analysis and sampling and quality assurance systems for laboratories;
 - (b) to (f) unchanged as reported in the Procedural Manual, 7th Edition;
 - (g) to define procedures, protocols, guidelines or related texts for the assessment of food laboratory proficiency, as well as quality assurance systems for laboratories (para. 30, ALINORM 93/23).

OTHER MATTERS OF INTEREST TO THE COMMISSION:

- Agreed that the Codex Commodity Committees should refer to the CCMAS all questions and needs in the sampling area, and that the Codex Secretariat should contract a consultant to prepare draft Codex General Guidelines on Sampling. The document elaborated by the consultant should be circulated for government comments and discussion at the next session of the CCMAS (paras. 26-27);
- Identified an urgent need for improved communication between itself and other Codex Committees involved in the development of analytical methods and sampling plans. The CCMAS considers that consistency in the treatment of these activities is an essential prerequisite to the development of the horizontal approach;
- Considered procedures for approving methods of analysis between sessions, including the use of electronic mail and facsimile services (para. 94);
- Approved the Checklist and the Guidelines for Use by the Committee on Methods of Analysis and Sampling and agreed to attach them to the report as Appendix II and III for information to governments and interested international organizations (para. 39);
- Approved the amplified Protocol for the Design, Conduct and Interpretation of Method Performance (Collaborative) Studies and agreed that the document CX/MAS 92/7 be forwarded to IUPAC for consideration (para. 39);
- Recognized that laboratory proficiency testing area should be considered in the priority of future work by Codex and that the concerned international organizations should be adequately informed of CCMAS's comments concerning proficiency testing harmonized protocol for laboratory analysis in order to review the future development in this area at the next session of the CCMAS (para. 47);

SUMMARY AND CONCLUSIONS (Cont.d)

- Discussed the definition of Limit of Determination for Codex purposes and agreed that this question should be referred to IUPAC, taking into consideration that a IUPAC project on Limit of Determination was being considered (para. 57);
- Discussed new criteria for evaluating acceptable methods for Codex purposes without reaching agreement on this question and agreed that this issue be reconsidered following consideration by the Commission of the proposed new terms of reference (paras. 58-63);
- Endorsed the provisions concerning methods of analysis in a large number of Codex standards (para. 69, ALINORM 93/23);
- Agreed to clarify the area of Codex procedures related to the endorsement of methods and their classification in four types, taking into consideration that an exhaustive discussion document should be prepared on this topic for the next CCMAS session (para. 68);
- Revised the general methods of analysis for contaminants in Codex standards and requested Canada to prepare a paper for the next session of the CCMAS so that a decision could be made on the acceptance of the proposed test methods (para. 73);
- Discussed the general method of ashing for the determination of heavy metals and agreed that the general method of ashing was in fact only a segment of a method and that methods of analysis for heavy metals should be developed through the normal procedure (para. 81);
- Considered several methods of analysis required for Codex standards and which need to be developed and noted several inputs received by international organizations concerning proposed methods (paras. 82-84); and
- Was informed on criteria to limit the number of false positive and false negative results for analytes near the limit of determination (paras. 95-89).

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INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Eighteenth Session from 9-13 November 1992 in Budapest, by courtesy of the Government of Hungary. The Session was attended by 80 delegates and observers from 22 countries and 11 international organizations. A complete list of participants, including the Secretariat is provided in Appendix I to this report.

2. Mr. A. Salamon, President of the Hungarian National Codex Committee, welcomed the participants and emphasized the importance of the international harmonization of sampling and analytical procedures for food. Mr. Salamon introduced Dr. Gy. Raskó, Secretary of State for Agriculture. Dr. Raskó gave a review of the actual situation in the Hungarian agriculture and food industry concerning the process carried out by Government for the privatization and the establishment of a market economy in the agricultural production and food processing in Hungary, as well as the low subsidy of the Hungarian export products. Dr. Raskó underlined that agriculture and food processing industry played an important role in the Hungarian economy, accounting for 30% of the value of Hungarian export products, with about 2.5 billion U.S. dollars in 1992. The Secretary of State pointed out that the food quality control and monitoring system for small and medium companies would be most important for the time being and that therefore Hungary's participation in the Codex Alimentarius activities would become essential in the next years.

ADOPTION OF THE AGENDA (Agenda Item 2)

3. The Committee adopted the Provisional Agenda (CX/MAS 92/1) as proposed. In order to facilitate discussions concerning sampling and endorsement of methods of analysis, the Committee agreed to discuss Agenda Items 5-6 and Agenda Items 7-12 respectively at the same time.

APPOINTMENT OF RAPPORTEUR (Agenda Item 3)

4. The Committee agreed with the proposal of the Chairman to appoint Ms. J. A. Springer (USA) as rapporteur.

MATTERS OF INTEREST TO THE COMMITTEE (Agenda Item 4)

5. The Secretariat introduced documents CX/MAS 92/2 and CX/MAS 92/2-Addendum 1 (Conference Room Document 8), presenting the following matters of interest:

a) Matters arising from the 19th Session of the Commission

6. The Committee was informed of the recommendations of the FAO/WHO Conference on Food Standards, Chemicals in Food and Food Trade, as endorsed by the Commission, regarding the horizontal approach to food standardization, the review of Codex standards, and the establishment of a Committee to deal with import/export control. The decision of the Commission regarding the distribution of competence between the Codex Committee on Methods of Analysis and Sampling, the Codex Committee on Nutrition and Special Foods for Dietary Uses and the Codex Committee on Food Labelling was also recalled.

7. The Committee was further informed that an expert consultation on sampling plans for aflatoxins would be held at the beginning of 1993 and that its objectives would be to consider sampling plans in relation to the maximum levels for aflatoxins in peanuts proposed by the Committee on Cereals, Pulses and Legumes, and possibly with lower levels as requested by several delegations attending the last session of the CCCPL.

8. The Committee noted the recommendation of the Committee on Food Additives Contaminants to lower the level of lead in sugar to 0.5 mg/kg, and that the method for determination of lead at this level would be considered under Agenda Item 12.

b) Matters arising from other Codex Committees

9. The Committee was informed that the last session of the Committee on Pesticide Residues had proposed a "Recommended Method of Sampling for the Determination of Pesticide Residues in Milk and Fish for Control Purposes", and that a harmonization of work in this area with the Committee on Residues of Veterinary Drugs in Foods was recommended. The Delegation of the Netherlands expressed the view that sampling plans for pesticides as well as other sampling plans should be referred to the CCMAS; the Secretariat reminded the Committee that sampling plans for pesticides and veterinary drugs were specifically excluded from the terms of reference of the CCMAS as they were the responsibility of the relevant Committees. However, it was for this session to decide if an amendment of its terms of reference in this respect was needed. The Committee agreed that this issue would be discussed under Agenda Items 5 and 6 dealing specifically with sampling.

10. The conclusions of the Committee on General Principles regarding the modification of acceptance and elaboration procedures were presented. The Observer of the IDF inquired about the implications of the proposed alignment of procedures for milk products on Codex procedures with respect to methods of analysis. It was pointed out that the Codex Committee on Milk and Milk Products would normally operate as other Codex Committees. However, the Committee on General Principles had agreed that the Committee on Milk and Milk Products should examine its terms of reference, as well as the acceptance procedures and the general implications of this transformation, and that it also applied to the definition of methods of analysis.

11. The Committee was informed of the amendments proposed to its terms of reference by the CCGP, following the decision of the Commission to widen its scope in the field of laboratory proficiency testing, at the request of the last session of the Committee. It was agreed that this question would be considered in detail under Agenda Item 8.

12. The main conclusions of the Committee on Food Import and Export Inspection and Certification Systems were presented, and it was pointed out that all matters related to food laboratory practices were excluded from its terms of reference, as they were under the responsibility of the CCMAS.

13. The Committee was informed that the last session of the Committee on Nutrition and Foods for Special Dietary Uses had reviewed the methods of analysis for use in standards for special foods and agreed on a list of methods for consideration by the CCMAS.

c) Other bodies

- International Federation of Glucose Industries (IFG)

14. The Observer of the IFG informed the Committee that this organization developed methods as part of ISO TC 93 on starches and related products and that many of the methods for starch hydrolysis products involved methods which had been in use for many years. Collaborative studies of such methods did not seem warranted and the observer was of the opinion that they should be endorsed as valid methods.

- International Organization for Standardization (ISO)

15. The Observer of the ISO referred to the list of methods presented in document CX/MAS 92/9 "Progress Report on Review of Standard Methods by International Organizations on Methods of Analysis and Sampling", whereby all recent ISO methods were presented.

- Nordic Committee on Food Analysis (NMKL)

16. The Observer from the NMKL presented its objectives as regarded methods of analysis for foods, especially for official control, and its activities in the area of quality assurance of laboratories. She also stressed the importance for all food laboratories to develop their quality systems.

- European Committee for Standardization (CEN)

17. The Observer of the CEN presented the objectives and work of CEN, which included the 12 EEC countries and 6 EFTA countries, through its technical committees operating in several areas. He indicated that they took into account the work carried out by other organizations and that the first standards would be published in 1993.

- AOAC International (AOAC)

18. The Observer of the AOAC informed the Committee of the change of its name to reflect its expanding activity, and of its work on programmes of method validation as well as harmonization of methods and quality systems protocols; it was also pointed out that a Nutrient Labelling Task Force had been established to carry out an extensive review of methods for nutrient analysis. The Committee was further informed that a new European sub-session had been created and that the next meeting of the AOAC would take place from 25-29 July 1993 in Washington.

- Canners International Permanent Committee (CIPC)

19. The Observer of CIPC informed the Committee of its wide international membership and its activities regarding analysis and sampling. He pointed out the wide use made by the canning industry of sampling plans developed by Codex, as well as commodity standards for processed fruit and vegetables, and stressed the necessity for continuous updating of these texts, especially as international trade in canned foods was very important.

REVIEW OF CODEX STANDARDS WITH REGARD TO SAMPLING (Agenda Item 6)
AND DISCUSSION PAPER ON CODEX SAMPLING (Agenda Item 5)

20. In view of the complementary nature of Items 5 and 6 of the Agenda, the Committee decided to discuss them together. The papers available for discussion were: CX/MAS 92/3 prepared by the Delegation of the United Kingdom and document CX/MAS 91/4 prepared by the Codex Secretariat.

21. The Committee was reminded that the Commission had agreed that a single advisory Codex document on sampling should be developed as recommended by the Sixteenth Session of CCMAS, rather than including sampling provisions in individual Codex Standards (paras. 339-341, ALINORM 89/40).

22. Comprehensive information on a review of sampling plans in Codex Standards had been prepared by the Secretariat and presented in the document cited above. The document provided information on the status of sampling plans in Codex, summarizing that about 24% of the Codex Standards made specific reference to the FAO/WHO Codex Sampling Plans for Prepackaged Food (CAC/RM 42-1969), 47% of Codex Standards did not have any reference to sampling and only 2% of Codex Standards made reference to sampling plans elaborated following the Codex Sampling Instructions.

23. The Delegation of the United Kingdom introduced document CX/MAS 92/3 which was prepared as an outline of a possible Volume of the Codex Alimentarius on Sampling to cover all aspects on Sampling. The document was considered a first draft of the work developed in the sampling area during many years for sampling to covering specific areas for commodity defects, compositional criteria, net contents and health related properties. The Committee was requested to indicate clearly in

which direction the General Guidelines should be developed to be a useful advisory document to control commodities moving in international trade.

24. Several delegations expressed appreciation to the Delegation of the United Kingdom for the excellent work produced in preparing the Codex Sampling Document and considered that, in view of the horizontal approach recommended by the Commission, all the Codex Sampling Plans including residues of pesticides, residues of veterinary drugs, contaminants heavy metals and aflatoxin should be incorporated in the General Guidelines to avoid discrepancies and duplication of effort in the area of Codex sampling. The Delegation of Hungary offered its contribution in completing the definitions following Section 5.5 on the heterogeneous nature of the distribution of quality characteristics in a lot. The Committee also discussed the need to combine the sampling procedure for Commodity Defects and Compositional Criteria in one section or to add another section dealing with Sampling for Control of Nutrients declared in labelling.

25. Other delegations supported the principle that the General Guidelines on Sampling should include a part on administrative aspects of sampling and a part dealing with sampling plans. The Guidelines on sampling plans should include simple sampling plans for routine control of commodities in trade, specifying criteria of acceptance of the lot similar to the sampling plans elaborated for residues of pesticides in food and Guidelines on Sampling elaborated on the basis of statistical plans to be used in case of dispute for commodities moving in international trade.

26. The Committee also considered that the Codex Commodity Committees should refer all questions and needs in the sampling area to the CCMAS. It was recommended that the Glossary on Sampling elaborated by IUPAC in collaboration with ISO should be used in the further elaboration of the General Guidelines on Sampling and that particular attention should be given to sampling concerning health related matters and consumer protection.

27. The Committee agreed to recommend that the Secretariat should contract a consultant to prepare draft Codex General Guidelines on Sampling, taking into consideration the document elaborated by the Delegation of the United Kingdom, the recommendation expressed by several delegations and documents elaborated by international organizations and FAO on Sampling. The document elaborated by the consultant would be circulated for government comments and discussion at the next session of the CCMAS.

NEW TERMS OF REFERENCE OF THE COMMITTEE (Agenda Item 8)

28. The Committee had before it document CX/MAS 92/6 recalling that, in the perspective of the horizontal approach, the CCMAS had expressed its interest in work on laboratory certification and quality assurance. The 19th Session of the Commission had agreed that the Codex Committee on General Principles should consider new terms of reference for the CCMAS, so as to widen its scope in this new area.

29. The Committee was further informed that the last session of the CCGP had considered the proposed new terms of reference and noted that the use of the expression "laboratory certification" might be understood as referring to laboratory accreditation, which was outside the mandate of Codex. In order to avoid any confusion, it had been proposed to replace it with "quality systems for laboratories", as indicated in document CX/MAS 92/2-Addendum 1 (Conference Room Document 8) where the new terms of reference were presented, as amended by the CCGP.

30. The Committee was of the opinion that this wording did not adequately reflect the activities the Committee intended to develop in this area, as it should be clear that CCMAS was responsible for all matters relating to proficiency testing of laboratories and quality assurance. The Committee therefore agreed that the

terms of reference should refer explicitly to "quality assurance systems for laboratories" and consequently agreed to recommend to the Commission to amend its terms of reference as follows:

- (a) to serve as a coordinating body for Codex with other international groups working in methods of analysis and sampling and quality assurance systems for laboratories;
- (b) to (f) unchanged;
- (g) to define procedures, protocols, guidelines or related texts for the assessment of food laboratory proficiency, as well as quality assurance systems for laboratories.

31. It was recalled that the Committee on General Principles had noted the possible overlap of competence between the Committee on Food Additives and Contaminants and the CCMAS regarding methods of analysis for food additives in food. It was pointed out that a Working Group of the CCFAC had been convened to consider these methods some years ago and a considerable amount of information was available to allow continuation of this work. It was suggested as a practical solution, to start with a small number of additives as this task was a very large one; the CCFAC could establish a priority list of additives requiring action by the CCMAS.

32. The Committee was of the opinion that consideration should first be given to the aspects related to procedure and how the CCMAS should work with the CCFAC, but also other Committees such as the CCRVDF and the CCPR. The Committee agreed to establish a Working Group composed of Australia (Chair), Finland and United States to examine relations between the CCMAS and other Committees with a specific activity in the field of analysis and sampling and to consider how effective cooperation could be established so as to prevent any overlap. The conclusions of the Working Group would be presented under Agenda Item 17: Other Business.

33. The Committee noted that these questions would be brought to the attention of the CCFAC, the CCPR and other Committees for information and to the Commission for consideration at its next session, so as to clarify the respective responsibilities of the Committees involved.

RECOMMENDATION FOR A CHECKLIST OF INFORMATION REQUIRED TO EVALUATE METHODS OF ANALYSIS SUBMITTED TO THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING FOR ENDORSEMENT (Agenda Item 9)

34. At the request of the Chairman, the Delegation of the United States introduced document CX/MAS 92/7 (Conference Room Document 5) and recalled that the last session of the Committee had decided on a review of the information required to evaluate methods. This revision, carried out by the United States with the assistance of Finland, had taken into account the comments received in reply to CL 1991/14-MAS on the circulated checklist. The Delegation pointed out that some important changes had been proposed to the IUPAC/ISO/AOAC Protocol for collaborative studies, such as the deletion of the double split level as unnecessary and perhaps statistically unsound and stressed the importance of reproducibility. The Delegation of the Netherlands mentioned the comments of the Dutch Standards Institute and forwarded these to ISO.

35. The Observer from the AOAC, while expressing appreciation of the revised checklist, stressed the necessity for a harmonized protocol and indicated that the changes should be discussed by the harmonized group IUPAC/ISO/AOAC.

36. The Delegation of the United States agreed with the proposal of the AOAC and indicated that the checklist as such was presented in the first part of the document and was for the use of the Committee, while the second part, with the proposed amendments would be transmitted by the Committee to IUPAC with a

recommendation to include the limit of inter-laboratory determination. In reply to a question, the Delegation further indicated that the table on pages 6-7 were examples of parameters for valid results, one including outliers, one excluding outliers, and that invalid results should not be confused with outliers.

37. The Delegation of the United Kingdom noted that the checklist was most useful as a working document for the activities of the Committee, and that it would also be valuable to Commodity Committees.

38. It was noted that the Draft Guidelines for Use by the Committee on Methods of Analysis and Sampling were part of the checklist and after an exchange of views it was agreed to retain them in their present form.

39. The Committee subsequently approved the Checklist (para. 96) and the Guidelines and agreed to attach them to the report as Appendix II and III. The Committee also agreed that the proposed revised Protocol would be transmitted to IUPAC for consideration.

REPORT OF THE NINTH MEETING OF INTERNATIONAL ORGANIZATIONS WORKING IN THE FIELD OF ANALYSIS AND SAMPLING (IAM) (Agenda Item 10)

40. The Committee was provided with a report of the Ninth Inter-Agency Meeting (IAM) on Methods of Analysis and Sampling, held at the premises of the Hungarian Office for Standardization (MSZH) on Friday, 6 November 1992. The Report (Conference Room Document 1) was introduced by Dr. K. Lingner (ISO). The meeting had been attended by representatives of ten organizations active in the field of food analysis and control, including CEN which for the first time attended this Meeting. The IAM was chaired by Mr. Castan (ISO/AFNOR). The report of the meeting is attached to the present report as Appendix IV. The IAM had considered matters specifically of interest to CCMAS such as:

- exchange of information on collaborative studies;
- status of joint work by AOAC, IUPAC and ISO towards a harmonized protocol for collaborative studies;
- methods of analysis and sampling required by the CAC in general subject areas and on commodity-related subjects;
- progress achieved with respect to harmonized terminology in the field of methods of analysis and sampling;
- exchange of views on proprietary laboratory techniques versus traditional methodology;
- proposal for reorganization of the IAM;
- laboratory proficiency testing.

41. The Delegation of the United Kingdom, in view of the new role assumed by the CCMAS, pointed out that there was a need to review the terms of reference of the Inter-Agency Meeting. The Delegation of the United Kingdom, supported by other delegations, also urged the international organizations to clarify their procedures to avoid duplication of work and infringement of each other's copyrights.

42. The Committee expressed its appreciation to the organizations represented on the IAM for all support provided to the CAC in the area of methods of analysis and sampling.

PROGRESS REPORT ON THE DEVELOPMENT OF A PROFICIENCY TESTING HARMONIZED PROTOCOL FOR LABORATORY ANALYSIS (Agenda Item 10 (a))

43. The Committee had before it document CX/MAS 92/8 which was introduced by the Delegation of the United Kingdom which had prepared the paper summarizing the draft IUPAC/ISO/AOAC harmonized protocol on proficiency testing.

44. The Delegation of the United Kingdom pointed out the need for laboratories to become involved with laboratory proficiency testing and the importance for Codex to operate in this area which is related and complementary to the selection and endorsement of validated method of analysis to control provisions in Codex Standards. The revised draft of this protocol has been prepared in the light of the comments made at an open Geneva meeting (May 1991), and finalized at a Workshop held in Delft in May 1992.

45. The Delegation of Hungary noted that reference to a statistical procedure should be included in Section 3.3.5 dealing with the "true" result. The Delegation of Sweden suggested that in the operation of a proficiency testing scheme consideration should also be given to improving performance of participating laboratories. The Delegation of France informed the Committee that different procedures were used in that country for proficiency testing and that the comments of CCMAS should be sent to the ISO TC 69, the ISO Committee which will be directly involved in the development of a more general protocol not strictly related to food analysis. The Delegation of the Russian Federation discussed the limits for acceptable z-scores, considering it not essential to fix the acceptable limit as referred to in Section 9 of the document, which considered unacceptable values of the z-scores exceeding an absolute value of 3.0. He also questioned the limit of inter-laboratory determination being included in the document.

46. The Delegation of the United Kingdom indicated that the document presented was a summary of the draft protocol elaborated in Delft and that in the final published version it would include the statistical procedure for "true" result and calculation of z-score, and that results for the z-scores of less than 2 were to be regarded as satisfactory. The Delegation pointed out that proficiency testing referred to procedures for assessing laboratories and not methods of analysis.

47. The Committee recognized that this area should be considered in the priority of future work by Codex and that the concerned international organizations should be adequately informed of the CCMAS's comments concerning the harmonized protocol in order to review the future development in this area at the next session of the CCMAS.

PROGRESS REPORT ON REVIEW OF STANDARD METHODS BY INTERNATIONAL ORGANIZATIONS (Agenda Item 10 (b))

48. The Committee had before it document CX/MAS 92/9 concerning the progress report on review of standard methods by international organizations prepared by the Delegation of the United Kingdom as requested by the Committee at its previous session.

49. The Delegation of the United Kingdom noted that there had been many activities in the various international and national standardization organizations which were of interest to delegates to the CCMAS and that such information had been summarized in the above paper.

50. It was noted that the information contained in the document was not complete; but had been collected over two years (1991 and 1992) and so only a selection had been made.

51. The Committee was also informed that the AOAC would collect information for Codex from the international organizations which were members of the IAM. Other international organizations provided oral information not included in the progress

report. The Delegation of the Netherlands referred to Guidelines elaborated for validation of methods of analysis at the level of one laboratory prior to their review on collaborative studies, which could be submitted to IUPAC and AOAC for consideration and review.

52. The Committee expressed its appreciation to the Delegation of the United Kingdom and looked forward to being kept informed of future progress.

LIMIT OF DETERMINATION (Agenda Item 10 (c))

53. The Committee had before it document CX/MAS 92/10 containing the comments of the Russian Federation on the limit of inter-laboratory determination, and document CX/MAS 92/10-Add.1 (Conference Room Document 9) presenting a response to comments from the 17th Session of CCMAS prepared by IUPAC and the comments of the United States. The Secretariat recalled that the Committee had considered this issue at its last session, and that its comments on a paper prepared by IUPAC had been forwarded to this organization.

54. The Delegation of the Russian Federation recalled that this question had been examined for several years and outlined the major elements of its proposal, especially the introduction of the MQS, minimum quantity of an analyte (ISO/IUPAC terms) that may be determined in the test sample by any method for safety purposes. The Delegation was of the opinion that this question could be taken up by IUPAC.

55. The Delegation of the United States stressed the necessity for the Committee to find an acceptable definition in this matter. It pointed out that an IUPAC project on Limit of Determination was being considered, and drew the attention of the Committee to the work of the ISO TC on Statistics in this area. The Delegation indicated that there were already several definitions of the limit of determination but it appeared difficult to find a satisfactory one. The Delegation of Austria shared this view and was of the opinion that the limit of determination could be estimated at the level of the laboratories through a practical rather than a statistical approach and international organizations could be helpful by giving rules for estimation rather than definition.

56. The Delegation of the Netherlands indicated that it was not in favour of the addition of new criteria for the limit of determination. The Delegation of the United Kingdom recalled that, as indicated under Agenda Item 9, IUPAC could be requested to include a definition of Limit of Determination in the harmonized protocol in quality control which it was developing.

57. The Committee agreed that this question should be referred to IUPAC.

METHODS OF ANALYSIS FOR AFLATOXINS - CRITERIA FOR EVALUATING ACCEPTABLE METHODS FOR DETERMINING AFLATOXINS (Agenda Item 11)

58. The Committee had for its consideration document CX/MAS 92/11, prepared by the Delegation of the United Kingdom and document CX/MAS 92/11-Add.1 prepared by the Delegation of the United States on the Reliability of Mycotoxin Assays, CX/MAS 92/11-Add. 2 prepared by AOAC (List of Methods) and Conference Room Document 11 presenting the comments of Hungary.

59. The Delegation of the United Kingdom, while introducing the document, recalled that the Committee had agreed at its last session to prepare a list of criteria for evaluating acceptable methods. The Delegation used the analysis of aflatoxins as an example of this approach. This approach represented a new development as it was not intended to prescribe methods as such. Its purpose was to establish criteria to ensure the acceptability of the methods, so as to offer the analyst a wider choice and allow more flexibility in the whole process.

60. The Delegation of the United States, referring to the document on Reliability of Mycotoxin Assays, emphasized the relevance of the present procedure

so as to limit the number of methods available to those methods which had been fully validated. The Delegation was of the opinion that in some cases, current methods were not applicable to the determination of maximum levels set by the standards, as in the case of aflatoxin M₁ in milk. Very large variations arose from low levels such as 5 micrograms/kg and the incidence of analytical errors, false positives or negatives, was very high. On the whole, it appeared essential to apply the procedure for accepting methods which had been perfected during many years and should not now be by-passed.

61. The Delegations of the Netherlands, Hungary and Sweden expressed their support of the approach presented by the United Kingdom, pointing out that the criteria would not replace existing methods and would be strict enough to limit the number of methods. Moreover, they would be of great practical use for analytical purposes.

62. The Delegation of the United Kingdom suggested including this issue along with other issues in the problems of interaction with other Codex Committees to be discussed by the Working Group (Australia, Finland, U.S.A.).

63. The Committee did not reach an agreement on this question and it was suggested that this issue be reconsidered after consideration by the Commission of the proposed new terms of reference. The Chairman indicated that this question would be discussed in more detail at the next session of the Committee.

ENDORSEMENT OF METHODS OF ANALYSIS IN CODEX STANDARDS (Agenda Item 7)
REPORT OF THE WORKING GROUP ON ANALYSIS (Agenda Item 12)

64. The Committee had before it document CX/MAS 92/5 which contained a comprehensive list of methods of analysis proposed for endorsement, as prepared by the Secretariat and the Conference Room Document 2 containing a Report of the Working Group on Endorsement which met on 7 November 1992. The following member countries and international organizations were represented: Canada, Finland, France, Germany, Hungary, Netherlands, United Kingdom, United States of America, AOAC International, IFG, ISO and OIV.

65. The Working Group, under the Chairmanship of Dr. W. Horwitz (U.S.A.), had the following tasks to perform:

- (a) to consider endorsement of methods of analysis in Codex standards, listed in document CX/MAS 92/5;
- (b) to consider the document CX/MAS 92/14 concerning methods of analysis required for Codex Standards in order that the participants to the Working Group could present proposals for newly developed methods.

66. The Working Group recommended that:

- (a) when the principle of methods of analysis under review was changed, the concerned international organizations responsible for the method should completely change the related reference, not only the year;
- (b) in reference to the endorsed IUPAC method for the determination of milk fat in Specified Vegetable Fat Products (Codex Standard 157-1987), the Working Group requested at its previous session to be informed on the conversion factor used for converting from butyric acid to milk fat, because it was not specified in the endorsed IUPAC method. The Working Group was informed by the Commodity Committee that there was a natural range for the content of this acid in 100% milk fat but a compromise "average" conversion factor of 3.6% had been adopted by a number of enforcement authorities. The Working Group recommended inserting an appropriate clear reference to the Codex Standards,

- (c) a discussion in plenary on how the Committee should deal with the procedure of endorsement of methods which were routinely used in the control of commodities, but were not collaboratively studied as required for their full endorsement. If this issue could not be resolved in the plenary, it is recommended that a discussion document be prepared on this topic for review at the 19th session of CCMAS.

67. The Working Group noted that several inputs and information were provided to the Secretariat on document CX/MAS 92/14, scheduled for discussion under Agenda Item 14.

68. The Committee discussed the working papers and the report of the Working Group and noted that the Working Group had endorsed several methods not provided with collaborative study data as Type IV, taking into account their long use for many years in the control of commodities. This procedure was considered a solution by the Working Group to a recurring problem for many methods. Several delegations expressed the opinion that it was urgent to clarify this area of the Codex procedures related to the endorsement of methods and their classification in four types. The Delegation of the Netherlands expressed its availability to provide its contribution in this work, considering that an exhaustive discussion document should be prepared on this topic for the next Session. The Delegations of Egypt, Hungary, the United Kingdom, the United States and the representative of IFG expressed their intention to collaborate on this topic. It was agreed that the Secretariat should provide guidance on particular aspect of concern to the CAC.

69. The Committee adopted the report of the Working Group, and decided to attach the list of methods together with their status of endorsement as Appendix V to this report. The Committee expressed its appreciation to Dr. Horwitz, and to the Members of the Working Group.

REVIEW OF GENERAL METHODS OF ANALYSIS FOR CONTAMINANTS IN CODEX STANDARDS
(Agenda Item 13 (a))

70. The Committee had before it documents CX/MAS 92/12 and CX/MAS 92/13 (Conference Room Document 6), CX/MAS 92/12 - Add. 1 (Conference Room Document 4), CX/MAS 92/12-13 - Add. 1 (Conference Room Document 10) and CX/MAS 92/12-13 - Add. 2 (Conference Room Document 12), which contained comments on an updated list of ten collaboratively studied general methods prepared by the Canadian Delegation and circulated to governments (CL-1992/20-MAS) for comments. The Committee received comments from the Czech and Slovak Federal Republic, Denmark, Egypt, Finland, Hungary, the Netherlands, Poland, the Russian Federation, Sweden, Switzerland and the United States.

71. The Committee was also provided with document CX/MAS 92/12 - Add. 1 (Conference Room Document 4), containing a list of 69 collaboratively studied methods and procedures, as well as a list of 674 literature references relating to multi-element and/or multi-food methods which have not been collaboratively studied. The delegations were invited to provide comments on these methods as well as their suitability as general methods for Codex purposes.

72. The Committee noted that the comments generally supported the acceptance of the 10 collaboratively studied method. The Delegation of Switzerland commented on the benefits of microwave digestion and the high pressure asher method, the risks of using perchloric acid and of contamination (air, glassware, etc.). The Delegation of Hungary pointed out that the microwave digestion method was adequate for the preparation of typically homogeneous samples for the determination of elements in relatively high concentrations.

73. The Committee requested the Canadian Delegation to prepare a paper for the next session of the CCMAS relating the government comments to the specific methods so that a decision could be made on the acceptance of the proposed test methods.

GENERAL METHOD OF ASHING FOR THE DETERMINATION OF HEAVY METALS (Agenda Item 13 (b))

74. In reply to Circular Letter CL 1992/20-MAS requesting comments on this method, the Committee had before it the following government comments: Canada and Finland in document CX/MAS 92/12 & 92/13 (Conference Room Document 6) (Part b); Egypt, Poland, Sweden and U.S.A. in CX/MAS 92/12 and 92/13 - Add. 1 (b) (Conference Room Document 10); Czechoslovakia and Hungary in Conference Room Document 11, the Russian Federation in Conference Room Document 12.

75. The Delegation of the Russian Federation, referring to its comments, recalled the studies which had been undertaken in its laboratories to define this general method, and suggested that it be adopted as a Type I method by the Committee.

76. The Delegation of the United Kingdom expressed its appreciation of the work carried out in Russia, but raised a general issue of procedure with respect to the endorsement of methods. The General Method of Ashing was in fact only a segment of a method, and the Committee could only consider the endorsement of complete methods. The Delegation of the Netherlands supported this view.

77. The Delegation of Poland pointed out that the determination of mercury required a special procedure and that this should be mentioned in the proposed method, especially in view of the serious problems posed by the contamination of fish products by mercury.

78. The Delegation of the Russian Federation, while recognizing the importance of this contamination, was of the opinion that mercury needed a special procedure. The Delegation further indicated that this was a preparatory method and that though no collaborative studies had been carried out, it was generally accepted in many countries.

79. The Secretariat recalled that the definition of a general method would be consistent with the horizontal approach; however, many methods were currently in use and it might prove necessary to apply different methods for different foods according to the levels specified for heavy metals.

80. The Delegation of the United States indicated that it was for the Committee to define whether general or specific methods were needed and, while noting the extensive work carried out in the Russian Federation, pointed out that this method was a classical one and that more modern techniques were now applied in several countries. The Delegation stressed that it was not intended to define methods to ensure compliance with high levels of heavy metals, but with low levels, especially where selenium or mercury were considered.

81. The Committee agreed that work on methods of analysis for heavy metals should continue and that these methods would have to be developed through the normal procedure.

METHODS OF ANALYSIS REQUIRED FOR CODEX STANDARDS AND WHICH NEED TO BE DEVELOPED AND/OR VALIDATED (Agenda Item 14)

82. The Committee had before it document CX/MAS 92/14, presenting a list of provisions included in Codex Standards and Guidelines whereby methods of analysis needed to be defined.

83. The Observer from ICC indicated that a method had been developed for the particle size of durum wheat semolina and durum wheat flour and proposed the cooperation of ICC in this matter. The Observer from IFG informed the Committee that his organization would undertake work to provide a suitable method for the determination of gliadin in gluten-free foods. It was also noted that a method for the determination of honey in fruit juices had been defined by CEN. The Delegation

of the Netherlands suggested that the CEN should be asked for develop a method to estimate fruit content in fruit juices.

84. In reply to a question on the status of work as to guideline levels for total mercury in fish the Secretariat recalled that the levels indicated in CX/MAS 92/14 had been adopted by the Commission and that the definition of predatory fish was currently under review; in this matter, the CCMAS, according to its terms of reference, was competent only as far as the definition of a method was concerned.

CRITERIA TO LIMIT THE NUMBER OF FALSE POSITIVE AND FALSE NEGATIVE RESULTS FOR ANALYTES NEAR THE LIMIT OF DETERMINATION (Agenda Item 15)

85. The Committee had before it document CX/MAS 92/15 - Conference Room Document 7 which was prepared by the Delegation of the Netherlands.

86. Dr. W.G. de Ruig introduced the document and explained that due to errors the samples assessed could be classified in four categories including true positive, true negative and false positive and false negative. The aim of the paper was to present an approach which gave an insight into the risk of false results.

87. Dr. de Ruig illustrated that through an analysis of the statistical distribution of false positive and false negative results it was possible to establish a relation between precision and inspection decision, making clear the importance of the decision making.

88. The Delegation of the Russian Federation, supported by several other countries, emphasized the importance of this paper for inter laboratory inspection and control. The Delegation of the United States underlined the importance of this paper for collaborative studies and proposed that the paper should be sent to IUPAC for consideration.

89. The Committee expressed its appreciation to Dr. de Ruig for the excellent paper useful for the quality of inspection procedures and for collaborative studies.

FUTURE WORK (Agenda Item 16)

90. The Committee noted that the agenda for its next session would include the following matters:

- Consideration of the procedures for the endorsement of methods of analysis;
- Consideration of working relationships between CCMAS and other Codex Committees;
- Sampling plans;
- Endorsement of Methods of Analysis in Codex standards - report of the Working Group on Methods of Analysis;
- Recommendations for a checklist of information required to evaluate methods of analysis;
- Report of the 10th Inter-Agency Meeting;
- Methods of Analysis for Aflatoxins;
- Review of methods of analysis for contaminants;

- Methods of Analysis and procedures required for Codex standards and which need to be developed and/or validated;
- Alternative criteria for evaluating acceptable methods of analysis for Codex purposes; and
- Proficiency testing harmonized protocol for laboratory analysis.

OTHER BUSINESS (Agenda Item 17)

91. During the discussions of Agenda Items 8 and 9, the Committee had agreed to establish an *Ad hoc* Working Group comprising Australia (Chair), Finland and the USA to consider how to define and develop working relationships between the CCMAS and other Codex Committees with respect to methods of analysis and sampling. Dr. Dahl, Chairman of the Working Group, presented its report and recommendations to the Committee.

92. The Group had identified a number of areas where action should be taken so as to ensure that the horizontal approach was applied to methods of analysis and sampling:

1. Criteria used for the endorsement of methods
2. Sampling plans
3. Classification/Typing of methods
4. Format of methods

93. The Working Group noted that the Checklist of Information approved by the Committee under Agenda Item 9 (CX/MAS 92/7) provided the acceptance criteria required. It agreed that, as a rule, methods of analysis and sampling plans relating to Codex standards should be considered by the CCMAS so as to ensure consistency in the work of all Committees in this area, without prejudice to the specific responsibility of other Committees on technical matters. As regards the format of methods, the Group agreed that the ISO format (ISO Standard 78-2:1982 Layout for Standards - Part 2: Standard for Chemical Analysis) should generally apply.

94. In this perspective, the Working Group made the following recommendations:

Recommendation 1

To inform the Commission that the CCMAS has identified an urgent need for improved communications between itself and other Codex Committees involved in the development of analytical methods and sampling plans. The Codex Committee on Methods of Analysis and Sampling considers that consistency in the treatment of these activities is an essential prerequisite to the development of the horizontal approach.

Recommendation 2

To recommend that the Commission advise all subsidiary bodies operating under Rule IX 1 (i):

- (a) that the CCMAS will provide them with a checklist of information required for the evaluation of methods of analysis;
- (b) that the CCMAS offers its advice and counsel in regard to the application of the acceptance criteria including the selection of methods;
- (c) that in response, Codex subsidiary bodies are to:

- (i) advise the CCMAS of all methods of analysis and sampling plans under development, and
- (ii) submit for endorsement all new methods of analysis and sampling plans.

Recommendation 3

That the CCMAS consider procedures for approving methods of analysis between sessions, including the use of electronic mail and facsimile services.

Recommendation 4

- (a) That the Protocol for the Design, Conduct and Interpretation of Method Performance (Collaborative) Studies as amplified in Annex II of CX/MAS 92/7 be forwarded to IUPAC for review of the amendments now incorporated;
- (b) That the checklist at the beginning of document CX/MAS 92/7 as well as the Draft Acceptance Criteria and Draft Guidelines for Use by the Codex Committee on Methods of Analysis and Sampling (CX/MAS 92/7, pp. 22-23) be attached to the report of the present session (ALINORM 93/23).

95. The Observer from AOAC pointed out that the Committee had agreed to the provisions of Recommendation 4 under Agenda Item 9.

96. Delegations expressed their support for the proposals of the Working Group and the Committee approved the Recommendations. The Committee noted that these Recommendations would be brought to the attention of the 20th Session of the Commission (July 1993).

97. The Delegation of the United States suggested that the amended Protocol on Collaborative Studies be sent to IUPAC before the General Assembly, so that approval could be given to IUPAC to work on the Protocol. This matter would also be examined by the Tenth Inter-Agency Meeting preceding the next session of the CCMAS.

DATE AND PLACE OF NEXT SESSION (Agenda Item 18)

98. The Committee was informed that its Nineteenth Session was tentatively scheduled to be held in Budapest in the beginning of November 1994.

ALINORM 93/23

APPENDIX I

LIST OF PARTICIPANTS ¹
LISTE DES PARTICIPANTS
LISTA DE PARTICIPANTES

Chairman: Dr. P. Biacs
Président: Professor, General Director
Presidente: Central Food Research Institute
Herman Ottó ut 15
H-1022 Budapest, Hungary

Secretary: Dr. P. Molnár
Secrétaire: Professor, Head of Food Quality Centre
Secretaria: Central Food Research Institute
Herman Ottó ut 15
H-1022 Budapest, Hungary

¹ The Heads of Delegations are listed first; alternates, advisers and consultants are listed in alphabetical order.
Les chefs de délégation figurent en tête et les suppléants, conseillers et consultants sont énumérés par ordre alphabétique.
Figuran en primer lugar los Jefes de las delegaciones, los Suplentes, Asesores y Consultores aparecen por orden alfabético.

MEMBER COUNTRIES

PAYS MEMBRES

PAISES MIEMBROS

AUSTRALIE
AUSTRALIE

Dr. C. Dahl
Government Analyst
Australian Government Analytical
Laboratories
P.O. Box 65,
Belconnen, A.C.T.2616
Australia

Mrs. B. Lee
Chief
Accreditation and Contaminants
Laboratory Services Division
Central Experimental Farm
Building No. 22
Ottawa; Ontario K1A 0C6
Canada
Tel. 613.995.4907
Fax. 613.992.5819

AUSTRIA
AUTRICHE

Dr. R. Kapeller
BA für Lebensmitteluntersuchung
Bürgerstrasse 47
A-4020, Linz
Austria

CZECH-SLOVAK FEDERAL REPUBLIC
REP. FED. TSCHEQUE ET SLOVAQUE
REP. FED. CHECA Y ESLOVACA

J. Barvir
Dipl. Chemist
Czech Agriculture and Food
Inspection
J. Plachty 16
151 18 Prague 5
Czech-Slovak F.R.

CANADA

Mr. J. Lawrence
Food Research Division
Health Protection Branch
Health and Welfare Canada
Sir Frederick Banting Building
Ottawa, Ontario KIA 0L2
Canada
Tel. 613.957.0946
Fax. 613.957.4775

Dr. J. Kalas
Head of Lab Dept.
Slovak Agricultural and Food
Inspection
Podjavorinskej 4
81549 Bratislava
Czech-Slovak F.R.

SUMMARY STATUS OF WORK

Subject	Action to be taken by:	Document Reference (ALINORM 93/23)
Guidelines on Sampling	Codex Secretariat	paras. 20-27
New Terms of Reference of the Committee	CAC	paras. 28-30
Methods of analysis for food additives in food	Codex Secretariat CCFAC	paras. 31-32
Checklist of information required to evaluate methods of analysis and Guidelines for use by the CCMAS	CAC	paras. 34-39
Proficiency Testing Harmonized Protocol for laboratory analysis	Concerned international organizations CCMAS	paras. 40-47
Limit of Determination	IUPAC Codex Secretariat	paras. 53-57
Criteria to ensure the acceptability of the methods of analysis	Codex Secretariat CAC	paras. 58-63
Codex procedures related to the endorsement of methods and their classification	Codex Secretariat Working Group	para. 68
Review of General Methods of Analysis for Contaminants	Canada Codex Secretariat Governments	paras. 70-73
Methods of analysis required for Codex standards	Codex Secretariat International organizations Governments	paras. 82-84

DENMARK
DANEMARK
DINAMARCA

K. Orbaek
Senior Scientific Officer
National Food Agency of Denmark
Morkhøj Bygade 19
DK-2860 Soborg
Denmark

EGYPT
EGYPTE
EGIPTO

Dr. A. Abdelkader
Sector of Quality Control
S.S.D.E.
12 Gawad Hasni st.
Cairo
Egypt

Dr. Salwa Dogheim
Chief Researcher
Central Agricultural Pesticides
Laboratory
Cairo
Egypt
Tel. 360.1395
Fax. 360.2209

FINLAND
FINLANDE
FINLANDIA

Mrs. H. Wallin
Senior Research Scientist
VTT/Food Research Laboratory
P.O. Box 203
SF-02151 Espoo
Finland
Tel. 358.0.4561
Fax. 358.0.455.2103

Mrs. P.L. Penttilä
Senior Research Scientist
National Food Administration
P.O. Box 5
SF-00531 Helsinki
Finland

FRANCE
FRANCIA

J.B. Bourguignon
Président
Commission Générale d'Unification
des Méthodes d'Analyse
Ministère des Finances
D.G.C.C.R.F.
Carré Diderot
3-5 Boulevard Diderot
75572 Paris Cedex 12
France

Ms. N. Normand
Association Française de
Normalisation
Tour Europe - Cédex 7
92049 Paris La Défense
France

GERMANY
ALLEMAGNE
ALEMANIA

Dr. W. Sanitz
Lebensmittel-Technologe
Bundesgesundheitsamt
Thielallee 88-92
D-1000 Berlin 33
Germany

Dr. J. Hild
Ltd. Chem. Direktor
Chem. Research Institute
Pappelstrasse 1
D-W-5800 Hagen
Germany

HUNGARY
HONGRIE
HUNGRIA

Dr. M. Váradi
Scientific Deputy Director
Central Food Research Institute
Herman Ottó út 15
H-1022 Budapest
Hungary

Ms. I. Boros
Chemical Eng.
National Food Investigation
Institute
Mester u. 81
H-1095 Budapest
Hungary

Dr. J. Domoki
Head of Department
National Institute of Food Hygiene
and Nutrition
Gyáli út 3/A
H-1097 Budapest
Hungary

Dr. A. Gergely
Head of Trace Element Department
National Institute of Food Hygiene
and Nutrition
Gyáli út 3/A
H-1097 Budapest
Hungary

Dr. M. Harkay-Vinkler
Ass. Professor
Universite Horticulture
Somlói út 14/16
H-1118 Budapest
Hungary

Dr. K. Matyasovszky
Head of Department
National Institute of Food Hygiene
and Nutrition
Gyáli út 3/A
H-1097 Budapest
Hungary

Dr. F. Örsi
Professor
P.O. Box 91
H-1502 Budapest
Hungary

Dr. K. Soós
Consultant
National Institute of Food Hygiene
and Nutrition
Gyáli út 3/A
H-1097 Budapest
Hungary

Dr. É. Szalánczy
Ass. Head of Laboratory
Malomkutató Kft.
Kisrökus u. 15/B
H-1024 Budapest
Hungary

T. Szerdahelyi
Senior Officer
Ministry of Agriculture
Department of Quality Control
Kossuth tér 11
H-1860 Budapest
Hungary

Dr. M. Tóth-Markus
Chemist
Ujvidék tér 7
H-1145 Budapest
Hungary

JAPAN
JAPON

H. Watanabe
Chief
Research Planning Section
Technical Research Division
Center for Quality Control
and Consumer Service
4-4-7, Konan, Minato-ku
Tokyo
Japan

LEBANON
LIBAN
LIBANO

Dr. C. Hilan
Director of FAO Project "Food Hygiene"
Agricultural Research Institute
Beirut
Lebanon

NETHERLANDS
PAYS-BAS
PAISES BAJOS

Mr. W.J. de Koe
Ministry of Welfare, Health and
Cultural Affairs
Inspectorate for Health Protection
P.O. Box 5406
2280 H.K. Rijswijk
Netherlands

Dr. W.G. de Ruig
State Institute for Quality Control
of Agricultural Products
Postbus 230
6700 AE Wageningen
Netherlands
Tel. 31.70.340.6960
Fax. 31.70.340.5435

Mr. H.A. van der Schee
Ministry of Welfare, Health and
Cultural Affairs
Regional Inspectorate for Health
Protection
Hoogte Kadijk 401
1018 BK Amsterdam
Netherlands
Tel. 020.23.75.25
Fax. 020.20.82.99

Dr. F. Smeets
Adviser
Commission for the Dutch Food
and Agricultural Industry
Unilever Research Laboratory
P.O. Box 114
3130 AC Vlaardingen
Netherlands

NORWAY
NORVEGE
NORUEGA

Mr. P.A. Rosness
Assistant Director General
Norwegian Food Control Authority
P.O.Box 8187
N-0034 Oslo
Norway

Mr. A. Vidnes
Norwegian Food Control Authority
P.O.Box 8187 Dep.,
N-0034 Oslo
Norway

POLAND
POLOGNE
POLONIA

Dr. K. Cwiek-Ludwicka
Food Analyst
National Institute of Hygiene
24 Chocimska Str.
00-791 Warsaw
Poland

Dr. E. Dunajski
Head of Laboratory
Quality Inspection Office
Ministry of Foreign Economic
Relations
8/12 Al. Pitsudskiego
81-378 Gdynia
Poland

Dr. R. Jedrzejczak
Food Analyst
Institute of Biotechnology of the
Agriculture and Food Industry
Rakowiecka 36
02-532 Warsaw
Poland

Dr. S. Tyszkiewicz
Professor, Head of Dept.
Meat and Fat Research Institute
Rakowiecka 36
02 532 Warsaw
Poland

RUSSIAN FEDERATION
FEDERATION RUSSE
FEDERACION RUSA

I. Ikurikhin
Head of Laboratory
of Institute of Nutrition
Russian Academy of Medical Sciences
Ustinsky proesa 2/14
109240 Moscow
Russian Federation

Dr. I.M. Skurikhin
Professor
Head of Laboratory of Food Chemistry
Institute of Nutrition
Academy of Medical Sciences of
the Russian Federation
2/14 Ustinsky Proezd
109240 Moscow
Russian Federation

SWEDEN
SUEDE
SUECIA

Dr. U. Edberg
Head of Laboratory
National Food Administration
Statens Livsmedelsverk Kem 2
P.O. Box 622
S-751 26 Uppsala
Sweden

Mrs. E. Lönberg
Codex Coordinator
National Food Administration
P.O. Box 622
S-751 26 Uppsala
Sweden

SWITZERLAND
SUISSE
SUIZA

Dr. R. Gerber
Section Head
Federal Office of Public Health
Haslerstrasse 16
CH-3000 Bern 14
Switzerland

Mr. P. Rossier
Head of Delegation
Federal Office of Public Health
Haslerstrasse 16
CH-3000 Bern 14
Switzerland

Mr. P. Venetz
NESTEC S.A.
Quality Management Department
55 Av Nestlé
CH-1800 Vevey
Switzerland

THAILAND
THAILANDE
TAILANDIA

P. Boriboon
Medical Scientist
Food Analysis Division
Dept. of Medical Science,
Yod-Se
Bangkok 10100
Thailand
Tel. 223.1444

S. Liamrangsi
Director
Food Hygiene Division
Health Department
10200 Bangkok
Thailand

T. Somsakul
Scientist
Standard Analysis Division
Department of Foreign Trade,
Ministry of Commerce
10200 Bangkok
Thailand

S. Srikongsri
Scientist
Biological Science Division
Ministry of Science,
Technology and Environment
Rama 6 Rd
10400 Bangkok
Thailand

N. Thongtan
Director
Agricultural Chemistry Division
Ministry of Agriculture and
Cooperatives
10900 Bangkok
Thailand

P. Traipuech
Scientist
Ministry of Commerce
Standard Analysis Division
10200 Bangkok
Thailand

UNITED KINGDOM
ROYAUME-UNI
REINO UNIDO

Mr. E.B. Reynolds
Public Analyst
83, Heavitree Road
Exeter, EX1 2ND
United Kingdom

Dr. R. Wood
Head of Department
Ministry of Agriculture,
Fisheries and Food
Food Science Laboratory
Colney Lane
Norwich NR4 7U
United Kingdom

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

Dr. G.W. Diachenko
Chief, Food Formulation Branch
Center for Food Safety and Applied
Nutrition (HFF-413)
Food and Drug Administration
200 C Street, S.W.
Washington, D.C. 20204, U.S.A.
Tel. 202.205.4319
Fax. 202.205.5025

Mr. E. Elkins Jr.
Principal Research Scientist
National Food Processors Association
1401 New York Avenue, N.W.
Suite 400
Washington, D.C. 20005, U.S.A.

Mr. W. Franks
Deputy Director
Science Division
Agricultural Marketing Service
U.S. Department of Agriculture
Room 3522, South Building
Washington, D.C. 20250, U.S.A.
Tel. 202.720.3075
Fax. 202.720.6496

Dr. W. Horwitz
Scientific Advisor
Center for Food Safety and Applied
Nutrition (HFF-7)
Food and Drug Administration
200 C Street S.W.
Washington, D.C. 20204, U.S.A.
Tel. 202.205.4307
Fax. 202.205.5205

Mrs. I. Kamishlian
Manager, Conc. Quality Programs
The Coca-Cola Company
P.O.Box 1734
Atlanta, GA 30301, U.S.A.

Dr. A.P. Rainosek
Professor of Statistics
National Marine Fisheries Service
University of South Alabama
Mobile, AL 36688
U.S.A.

Mrs. J. Springer
Director, Division of Mathematics
Center For Food Safety and Applied
Nutrition (HFF-100)
Food and Drug Administration
200 C Street, S.W.
Washington, D.C. 20204, U.S.A.
Tel. 202.205.5051
Fax. 202.205.5354

OBSERVER COUNTRY
PAYS OBSERVATEUR
PAIS OBSERVADOR

SLOVENIA
SLOVENIE
ESLOVANIA

Dr. I. Koren
Food Technology
Fructal Tovarniska
65270 Ajdovscina
Slovenia

D. Zavrnik
Specialist in Sanitary Chemistry
University Institute of Public Health
and Social Welfare
Trubarjeva 2
61000 Ljubljana
Slovenia

INTERNATIONAL ORGANIZATIONS
ORGANISATIONS INTERNATIONALES
ORGANIZACIONES INTERNACIONALES

AOAC INTERNATIONAL

Mr. R.R. Christensen
Executive Director
AOAC International
2200 Wilson Blvd., Suite 400
Arlington, VA 22201-3301, U.S.A.

Mrs. M. Lauwaars
European Representative
AOAC International
P.O. Box 153
6720 AD Bennekom
Netherlands

EUROPEAN COMMITTEE FOR STANDARDIZATION

Mr. M. Jeanson
Technical Officer
Rue de Stassart 36
B-1050 Brussels
Belgium

**COMITE INTERNATIONAL PERMANENT
CONSERVE CIPC**

Mr. Y. Michelon
Secretary-General
44 rue d'Alésia
75014 Paris
France

EUROPEAN COMMUNITIES (EC)

Dr. G. Verardi
Principal Administrator
Directorate General
for Internal Market and
Industrial Affairs
200 Rue de la Loi
1049 Bruxelles
Belgium

**INTERNATIONAL ASSOCIATION FOR
CEREAL SCIENCE AND TECHNOLOGY**

Dr. Á. Mosonyi
Scientific Expert
Malomipari Kutató és Fejlesztő Kft.
Kis Rókus u. 15b
H-1024 Budapest
Hungary

INTERNATIONAL DAIRY FEDERATION (IDF)

Mr. E. Hopkin
Secretary General
41 Square Vergote
1040 Brussels
Belgium

INTERNATIONAL FEDERATION OF GLUCOSE
MANUFACTURERS (IFG)

Dr. B. Whitehouse
Quality Assurance Manager
Cerestar SA/NV
Havenstraat 84
B-1800 Bruxelles
Belgium
Tel. 322.254.2605
Fax. 322.254.2604

INTERNATIONAL ORGANIZATION FOR
STANDARDIZATION (ISO)

Mr. G. Castan
Expert
8 rue de Varize
75016 Paris
France

Mr. K.G. Lingner
Technical Group Manager
Planning and Technical Coordination
ISO Central Secretariat
1, Rue de Varembe
CH-1211 Geneva 20
Switzerland

Mrs. E. Nagy
Secretary of ISO/TC 34
Hungarian Office for Standardization
Secretariat of ISO/TC 34
"Agricultural Food products"
Üllői út 25
H-1095 Budapest
Hungary

MARINALG INTERNATIONAL

Mr. J.J. Piot
Adviser
Association Mondiale des Producteurs
d'Extraits d'Algues Marines
World Association of Seaweed Processors
85, Bd Haussmann
75008 Paris
France

NORDIC COMMITTEE ON FOOD ANALYSIS
(NMKL)

Mrs. H. Wallin
Secretary General
VTT/Food Research Laboratory
P.O. Box 203
SF-02151 Espoo
Finland

INTERNATIONAL VINE AND WINE OFFICE
(OIV)

Dr. B. Mandrou
Professor
Faculté de Pharmacie
34060 Montpellier Cedex 1
France

JOINT FAO/WHO SECRETARIAT
SECRETARIAT CONJOINT FAO/OMS
SECRETARIA CONJUNTO FAO/OMS

Dr. Enrico Casadei
Food Standards Officer
Joint FAO/WHO Food Standards
Programme
FAO
Via delle Terme di Caracalla
00100 Rome
Italy

Ms. Selma Doyran
Food Standards Officer
Joint FAO/WHO Food Standards
Programme
FAO
Via delle Terme di Caracalla
00100 Rome
Italy

ALINORM 93/23
APPENDIX II

RECOMMENDATIONS FOR A CHECKLIST OF INFORMATION
REQUIRED TO EVALUATE METHODS OF ANALYSIS
SUBMITTED TO THE CODEX COMMITTEE
ON METHODS OF ANALYSIS AND SAMPLING FOR ENDORSEMENT

(Prepared by the Delegation of the United States)

The Report of the Seventeenth Session of the Codex Committee on Methods of Analysis and Sampling indicated that the United States would review the comments of governments on the first two protocols from the International Union of Pure and Applied Chemistry (IUPAC) and prepare a revised draft checklist for consideration at this Eighteenth Session. Replies to this request were received from Cuba, Finland, Mexico, Poland and Thailand. The comments were primarily editorial and translational in nature and their excellent suggestions have been considered in the preparation of the revised versions which are attached. Ms. Harriet Wallin of the Delegation of Finland assisted in the preparation and revision of these documents.

The submitted revisions consist of the following documents and their appendices for consideration as working guides for submission of methods of analysis and their documentation by Codex Committees for review and endorsement by CCMAS:

RECOMMENDATIONS FOR A CHECKLIST OF INFORMATION REQUIRED TO EVALUATE METHODS OF ANALYSIS SUBMITTED TO THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING FOR ENDORSEMENT
(Revision of ALINORM 91/23, Appendix III).
Appendix 1: Example of a completed table

ANNEX 1: PROTOCOL FOR THE DESIGN, CONDUCT AND INTERPRETATION OF METHOD-PERFORMANCE (COLLABORATIVE) STUDIES - THE IUPAC 1987 HARMONIZED PROTOCOLS

Appendix 1: Symbols
Appendix 2: Definitions
Appendix 3: Critical values for the Cochran and the Grubbs tests;
Calculation of Cochran outlier and Grubbs test values.
Appendix 4: Flowchart for outlier removal

ANNEX 2: ACCEPTANCE CRITERIA

In view of their length, the CCMAS may wish to utilize references to the protocols and/or their amplified versions published by standards-setting organizations rather than reprinting them.

[Editorial note: Underline and bold face in text (heads in original are already underlined) indicate additions made to the previous draft. Some sections (indicated with *) have been revised to conform to the IUPAC-1987 Protocol, Annex 1.]

RECOMMENDATIONS FOR A CHECKLIST OF INFORMATION REQUIRED TO EVALUATE METHODS OF ANALYSIS SUBMITTED TO THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING FOR ENDORSEMENT

TYPE OF INFORMATION REQUIRED FOR SUBMISSION TO THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING FOR CONSIDERATION

1. REPORT FORMAT

1.1 IDENTIFICATION INFORMATION

1.1.1 Responsible Codex Committee

The Codex Committee requesting the endorsement for reference and referral.

1.1.2 Codex Standard and Status

A reference to the specific commodity item under consideration, its endorsement status, and a citation to its appearance in the Codex documentation.

1.1.3 Analyte or Property

The specific component, constituent, or property (chemical, physical, microbiological) which is to be measured and for which there exists a requirement for a limit or specification in the applicable standard.

1.1.4 Codex Specification or Limit

The specific specification, limit, tolerance, or guideline which is given in the standard and which provides the boundary between acceptable and unacceptable material.

1.1.5 Method of Analysis

(a) Title and Principle. -- A statement of the method of analysis which incorporates a summary of the principles of isolation and/or measurement.

(b) Limit of Detection, Limit of Determination, or Applicable range (as needed).

(c) Classification (Type). -- The method classification as defined in the Codex Alimentarius Commission Procedural Manual, pp. 143-144 as amended by the Commission in 1991 (Ref. ALINORM 91/21 or ALINORM 91/23, para. 43:

Defining Methods	(Type I)
Reference Methods	(Type II)
Alternative Approved Methods	(Type III)
Tentative Methods	(Type IV)

(d) Reference to Source of Method of Analysis. Bibliographic citation to the scientific or technical publication, to the Codex document, or to the internal reference number of the national or international organization, as applicable. The reference given should permit tracing back to original source documents discussing the application of the method to the analyte and commodity involved.

1.2 DESIGN, CONDUCT AND REPORTING OF RESULTS OF COLLABORATIVE STUDY SUPPORTING THE ENDORSEMENT OF THE METHOD

The design and conduct of the collaborative study of the method must follow the principles outlined in the 1987-IUPAC Harmonized Protocol for the Design, Conduct and Interpretation of Collaborative Studies (see Annex 1; reproduced from reference 1).

1.2.1 Bibliographic Reference to Collaborative Study

Include citation to the published collaborative study as a literature reference, Codex document number, or to the national or international organization internal reference number, as applicable. Give sufficient documentation so that a librarian can obtain the referenced document directly from the journal, by interlibrary loan, or by request to the organization responsible for its production.

1.2.2 Design

State the number of materials, laboratories, determinations, replicates and tests used. If these vary from material to material, a separate line may have to be introduced in the table for the variable information.

1.2.3 Material identification and composition

Identify the materials in the column heads for a table of data, including information from 1.2.4 to 1.2.8.

1.2.4 * Outliers Removed *

Report number of laboratories remaining after removal of outliers and/or percent of outliers which had to be rejected in order to obtain the precision parameters reported in 1.2.8. If no outliers were rejected, report that fact as 0. Indicate identification number(s) of laboratories removed in order to note a consistent systematic bias on the part of any of them, if present.

1.2.5 Concentration of Analyte or Value of a Property

Report if known or assumed. If it is same throughout, it may be incorporated into the material identification, 1.2.3.

1.2.6 Average Found and Units

Give the average value found for each material, indicating the units in the row heading. If the number of replicates reported by each laboratory was not the same, use the average of each laboratory for averaging to avoid weighting of results.

1.2.7 Recovery

Report percent recovery, if amount of analyte present is known or assumed.

1.2.8 Precision Parameters

Report the following precision parameters

- (a) Repeatability (within laboratory) standard deviation s_r , in the same units as the average.
Repeatability relative standard deviation ($s_r \times 100/\text{average}$).
Repeatability limit ($2.8 \times s_r$).
- (b) Reproducibility (among laboratories, including within -) standard deviation, s_R , in the same units as the average.
Reproducibility relative standard deviation ($s_R \times 100/\text{average}$)
Reproducibility limit ($2.8 \times s_R$)

Report precision parameters (1) with all valid results and (2) with results from which outliers have been removed. Do not include data from laboratories that did not follow the method, that reported problems with instruments or columns, whose system suitability parameters did not conform to specifications, or who experienced similar aberrant behaviour.

The standard deviations must be obtained material by material. The relative standard deviations are usually the most informative precision parameters in food analysis because it is often constant over a wide range of concentrations. It is important to recognize the relative standard deviation among laboratories is not obtained by calculating the standard deviation of all the data from a material (except when only single determinations are performed); it must be obtained by a "one-way analysis of variance", as demonstrated in the Steiner portion of the AOAC Statistical Manual, or ISO 5725 Section 7.2 (b).

The repeatability limit and the reproducibility limit indicate how close two analysts in the same laboratory (repeatability) or in different laboratories (reproducibility) should agree in 19 of 20 analyses of the same test sample.

See example in Appendix 1 of a completed Table containing information under 1.2.1 - 1.2.8.

2. NOTES

(Additional information, exceptions, and reasons for not following the recommendations.)

2.1 References to same method endorsed for other Codex Standards.

2.2 If a Codex method is available for this analyte or property for a different commodity and this method is not recommended for the commodity standard under consideration, give the reasons for not using the previously used method and for using a different method for this commodity or concentration level.

2.3 If a general Codex method is available for this analyte or property and it is not used in this standard, give the reasons for not using the general method.

2.4 Give reasons for any modifications of the previously used or endorsed method for other commodities or of the general method.

3. REFERENCES

Horwitz, W. (1988) Protocol for the Design, Conduct and Interpretation of Collaborative Studies, Pure and Appl. Chem. 60, 855-864.

Youden W.J. & Steiner. E.H. (1975) Statistical Manual of the AOAC. AOAC International, Washington, D.C., U.S.A.

International Organization for Standardization (ISO). International Standard 5725:1986. Precision of Test Methods - Determination of Repeatability and Reproducibility for a Standard Test Method by Interlaboratory Tests. Available from ISO, Geneva, Switzerland and National Standardization Organizations. Currently being revised.

EXAMPLE OF A COMPLETED TABLE REPORTING PRECISION PARAMETERS. ADOPTED FROM POCKLINGTON, W.D. (1990) HARMONIZED PROTOCOL FOR THE ADOPTION OF STANDARDIZED ANALYTICAL METHODS AND FOR THE PRESENTATION OF THEIR PERFORMANCE CHARACTERISTICS (1).

Two interlaboratory method-performance tests carried out at the international level in 1987 and 1988 (2) by the IUPAC Commission on Oils, Fats and Derivatives, in which 7 and 13 laboratories participated, each performing 2 replicates, gave the following statistical results (evaluated in accordance with 1987-IUPAC Harmonized Protocol (3) and ISO 5725):

TABLE OF COLLABORATIVE STUDY PARAMETERS

Butyric acid (g/100 g)

Identification No.	A	B	C	D
No. of laboratories retained after eliminating outliers	11	7	11	11
No. of outlying laboratories	2	-	-	2
Code of outlying laboratories	d, f			d, g
No. of accepted results	22	14	22	22

Mean and precision parameters for all valid results (as an example)

Mean (g/100g)	0.20	0.35	1.79	3.46
True or accepted value, if known	-	-	1.82	3.45
Repeatability std. deviation (s_r)	0.12	0.015	0.044	0.22
Repeatability relative std. deviation (RSD_r , %)	6.0	4.3	2.5	6.4
Repeatability limit, r ($2.8 \times s_r$)	0.34	0.04	0.12	0.62
Reproducibility std. deviation, (S_R)	0.40	0.056	0.16	0.36
Reproducibility relative std. deviation (RSD_R , %)	20.0	16.0	8.7	10.4
Reproducibility limit, R ($2.8 \times S_R$)	0.11	0.16	0.43	1.0

Mean and precision parameters for accepted results with outliers removed

Mean (g/100g)	0.190	-	-	3.46
True or accepted value, if known	-	-	-	3.45
Repeatability std. deviation (s_r)	0.0080	-	-	0.10
Repeatability relative std. deviation ($RSD_r, \%$)	4.2	-	-	2.9
Repeatability limit, r ($2.8 \times s_r$)	0.02	-	-	0.29
Reproducibility std. deviation, (S_R)	0.024	-	-	0.24
Reproducibility relative std. deviation ($RSD_R, \%$)	12.6	-	-	7.0
Reproducibility limit, R ($2.8 \times s_R$)	0.11	0.16	0.43	0.68

REFERENCES

- (1) Pure and Appl. Chem. (1988) 62, 149-162.
- (2) Pure and Appl. Chem. (1986) 58, 1419-1428.
- (3) Pure and Appl. Chem. (1988) 60, 855-864.

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APPENDIX III

**METHODS OF ANALYSIS SUBMITTED FOR ENDORSEMENT BY THE
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING:
PRECISION CRITERIA**

(Interpretative summary of Protocol appearing in
Pure & Applied Chemistry (1990) 62, 149-162)

BACKGROUND OF THE ACCEPTANCE CRITERIA

It has been shown that when the precision of a method is expressed as the reproducibility (among-laboratories) relative standard deviation, RSD_R , the magnitude of this value is strongly dependent on the analyte concentration. Essentially similar RSD_R values are obtained for similar analyte levels irrespective of the nature of the analyte, matrix, or analytical method. For this reason the magnitude of the RSD_R value is frequently used as the basis for the evaluation of the acceptability of the precision of a method of analysis.

In a survey of a large number of method performance studies involving a wide range of analytes, matrices, and separation and measurement techniques, it was found that the relative standard deviation for reproducibility generally can be predicted from the so-called Horwitz equation (1):

$$(1) \quad RSD_R \text{ (predicted)} = 2^{(1-0.5 \log C)} \approx 2C^{-0.1505}; \text{ and}$$

$$(2) \quad \text{HORRAT} = RSD_R \text{ (observed)} / RSD_R \text{ (predicted)},$$

where C is the concentration expressed as a decimal fraction and \log is to the base 10. The ratio between observed RSD_R values and the RSD_R values predicted by this equation, designated as HORRAT, can, according to Horwitz et al. (2), be regarded as an indication of the acceptability of a method with respect to its precision. In an interlaboratory performance study, a series of HORRAT ratios close to 1.0 or consistently smaller, indicates acceptable precision of a method; HORRAT values unconsistently near or above 2 probably indicate an acceptable method with respect to precision. The Horwitz equation, based on almost 7,000 interlaboratory data sets available over the past century, is a convenient, succinct summary of the relative precision encountered in actual practice.

The maximum acceptability criterion "An RSD_R value no higher than twice the predicted value indicates an acceptable method" has been adopted by IUPAC and is contained in the harmonized protocol for the adoption of standardized analytical methods (3). The protocol lists three criteria to be considered when adopting analytical methods for publication as a standard method, and these are listed below as the suggested acceptance criteria of the Codex Committee on Methods of Analysis and Sampling.

DRAFT GUIDELINES FOR USE BY THE CODEX COMMITTEE
ON METHODS OF ANALYSIS AND SAMPLING

The Codex Committee on Methods of Analysis and Sampling follows the criteria described below (3) when considering the endorsement of methods of analysis submitted to the Committee:

1. The organization of the method performance study and the statistical analysis of the results obtained were carried out according to the principles outlined in the IUPAC 1987 harmonized protocol. (4)
2. Each set of data (obtained from the analysis of test samples with different matrices or analyte concentration must not be found to contain more than 2/9 ($\approx 22.2\%$) of statistically inexplicable outlying laboratories (maximum 2 laboratories out of 9).
3. The calculated expected values for relative standard deviation for reproducibility (RSD_R) at the analyte concentration in question, expressed as concentration ratios are comparable to the following values:

Concn	10^{-9}	10^{-8}	10^{-7}	10^{-6}	10^{-5}	10^{-4}	10^{-3}	10^{-2}	10^{-1}	1
RSD_R , %	45	32	23	16	11	8	5.6	4	2.8	2

Note 1: A "Concn" (Concentration) of 1 is 100% in conventional units; 10^{-1} is 10%; 10^{-6} is mg/kg (ppm); 10^{-8} is 10 μ g/kg (ppb); etc.

Note 2: The RSD_R values cited above have been calculated from the Horwitz equation (1) above, e.g., for $C = 10^{-6}$,
 $RSD_R = 2^{(1-0.5 \log C)} = 2^{(1-0.5(-6))} = 2^{(1+3)} = 2^4 = 16\%$.

Note 3: In the absence of overriding information, values within 2 times the RSD_R (calculated from the concentrations found) are considered as acceptable precision of method-performance among-laboratories. Within-laboratory method performance (RSD_r), frequently is about one half to two thirds of these values.

References

- (1) Peeler, J.T., Horwitz, W. & Albert, R. (1989). J. Assoc. Off. Anal. Chem. 72, 784-806.
- (2) Horwitz, W., Albert, R., Deutsch, M.J. & Thompson, J.N. (1990), J. Assoc. Off. Anal. Chem. 73, 661-680; (1992), 75, 227-229.
- (3) Pocklington, W.D. (1990) Pure and Appl. Chem. 62, 149-162.
- (4) Horwitz, W. (1988) Pure and Appl. Chem. 60, 855-864.

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APPENDIX IV

REPORT OF THE NINTH INTER-AGENCY MEETING (IAM)
Budapest, 6 November 1992

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OPENING OF THE MEETING

1. The Ninth Inter-Agency Meeting was opened by Mr. Sándor Vass, Director of the Department of International Relations at the Hungarian Office for Standardization (MSZH). Mr. Vass welcomed the Representatives of the various international organizations (see Annex), extending a special welcome to Mr. Jeanson, who attended the IAM as Representative of the European Committee for Standardization (CEN). He then stressed the importance of international standardization work in the area of food products to the foreign trade of his country. Referring to the recent changes of the national system, Mr. Vass informed the IAM that Hungary was planning to establish a new standardization system which, as in most of the Western countries, will be based on voluntary standards.

ELECTION OF CHAIRMAN

2. Upon the proposal of Mr. Vass, Mr. G. Castan (Special Advisor to AFNOR), who had chaired three previous Inter-Agency Meetings, was elected Chairman. Mr. Castan, after thanking the meeting for the renewed confidence was pleased to note the current positive developments in Hungary. He then wished the participants a successful meeting and a pleasant stay in Budapest.

ADOPTION OF THE AGENDA

3. The agenda was adopted without amendment. Following a proposal by the Representative of AOAC, it was agreed to consider questions of copyright, exchange of information and mutual recognition of methods of analysis under the item "Other activities of interest to the IAM".

REVIEW OF MEMBERSHIP OF THE IAM

4. Bearing in mind the Terms of Reference of the IAM, participants agreed that its current membership was adequate and responded to the needs of CAC. In reply to a query by the representative of ICUMSA, the representative of the Codex Secretariat confirmed that the topic of laboratory accreditation should not be considered by the IAM for the time being. In this context, the Representative of IDF drew attention to the International Laboratory Accreditation Conference (ILAC) which had prepared useful guidance documents on related subjects.

ACTION TAKEN BY THE CODEX SECRETARIAT IN RESPONSE TO THE RESULTS OF THE EIGHTH IAM

5. The Representative of the Codex Secretariat informed the IAM that the following action had been taken since the Eighth IAM:

- The report of the Eighth IAM had been circulated as Appendix IV to the report of the Seventeenth Session of CCMAS (document ALINORM 91/23);
- A computerized compilation of 529 Codex methods of analysis had been prepared, of which 387 had been endorsed by CCMAS; 100 were under discussion at different stages, i.e. temporarily endorsed, submitted for endorsement or not endorsed. Another 40 methods would have to be developed for the purpose of CAC. In view of the results of the International Conference on Food Standards and Trade and several recommendations by this Conference which had been adopted by CAC, as well as the need to revise some of the old standards, the volume of Codex methods had not yet been published;
- The proposal to change the Terms of Reference of CCMAS as agreed by the Codex Committee on General Principles, would be considered at the Eighteenth Session of CCMAS;
- The CCMAS Working Group on the Endorsement of Methods would be meeting subsequent to the IAM.

6. As suggested by the Secretary, the Representative of the Codex Secretariat also agreed to circulate a computerized Codex list of references to methods developed by the various organizations to the organizations concerned for updating.

EXCHANGE OF INFORMATION ON COLLABORATIVE STUDIES

7. The Representative of AOAC referred to a compilation of Methods of Analysis and Sampling prepared by her organization. This compilation included references to methods of AOAC, IDF, IFG, ISO, NMKL and others. Information on collaborative studies planned and carried out by different organizations was regularly collected and updated as a result of a circular letter by AOAC to all interest organizations.

8. The Representative of CEN informed the IAM that his organization would follow the criteria of the Annex to the EEC Directive 85/591 and therefore would preferably choose collaboratively tested methods following ISO 5725. This would be the case if the procedure of mandates from the EEC was extended to the food sector.

STATUS OF JOINT WORK BY AOAC, IUPAC AND ISO TOWARDS A HARMONIZED PROTOCOL FOR COLLABORATIVE STUDIES

9. The IAM noted that the Harmonized Protocol had already been adopted by several organizations and that large parts of the protocol had also been taken into account in the current revision work of ISO 5725. It recognized, however, that ISO had been unable to adopt the Protocol so far, since divergent views still existed in this organization with respect to the treatment of outliers.

10. The Chairman felt it would be ideal to arrive at an identical publication of the Harmonized Protocol by all organizations concerned. If this should not be possible, reference to the Protocol should at least be included in the forward to the relevant standards in order to give proper credit to the results of common effort by the collaborating organizations.

11. The Representative of AOAC then informed the IAM that AOAC, IUPAC and ISO were currently developing a Harmonized Protocol on the presentation and organization of proficiency testing schemes. A draft of this Protocol was ready for submission to the secretariats of three organizations. It was agreed to include this subject in the agenda of the next IAM.

METHODS OF ANALYSIS AND SAMPLING REQUIRED BY THE CAC

12. Following the suggestions by the Representatives of IDF and AOAC, it was agreed to limit the discussion to matters of direct interest to the IAM rather than consider the full activity reports submitted by the various organizations. In this context, the IAM was pleased to note that most of the organizations attending had submitted written statements or other information material on their relevant activities.

Methods of Sampling

13. The Representative of the Codex Secretariat informed the IAM that CCMAS was currently developing general Codex Guidelines on Sampling which covered all matters relating to sampling, including sampling plans. The aim of these general Guidelines was to avoid duplication of effort between the various Codex Committees and to cover all existing needs. The IAM noted that, pending the publication of these Codex Guidelines no action was required for the time being.

Pesticide Residues

14. The Representative of the Codex Secretariat, referring to a list of 177 compounds which had been established by a Working Group of the Codex Committee on Pesticide Residues, informed the IAM that the limits specified were frequently

above the limits of determination of the respective methods. He added that, while there was no specific request to the IAM, the Codex Secretariat would appreciate receiving information from the specialized organizations.

Residues of Veterinary Drugs

15. The Representative of the Codex Secretariat informed the IAM that a Working Group of the Codex Committee on Residues of Veterinary Drugs in Foods was currently identifying suitable methods. So far, 13 standards had been prepared, 5 methods recommended and 8 methods provisionally recommended. He invited the Organizations participating in the IAM to provide input to this Working Group.

16. The Representative of AOAC offered to provide information to those interested in a symposium on Veterinary Drug Residues to be held in Eindhoven (The Netherlands) in early 1993.

Methods of Analysis for Nutritional Food Labelling

17. The IAM was informed that the CAC had given responsibility for the endorsement of relevant methods to the Codex Committee on Nutrition and Foods for Special Dietary Uses. It also noted the activity of AOAC in this area, as mentioned in the report submitted by this organization as well as AOAC's work relating to fat analysis which had been subject of an article in a recent edition of "The Referee". The Representative of the Codex Secretariat informed the IAM that there were no specific requests to the IAM regarding methods required.

Food Additives and Contaminants

18. The Representative of the Codex Secretariat drew attention to the new General Standard for Food Additives and the difficulties in using a similar approach for all commodities, including those not covered by Codex standards. He added that, while the Terms of Reference of the Codex Committee on Food Additives and Contaminants included the selection of methods, the Committee had not yet defined any methods. On the subject of contaminants, the Representative of the Codex Secretariat informed the IAM that only a few methods were required, such as the determination of vinyl chloride and acrylonitrile in packaging materials.

Microbiology

19. While there were no specific requests by the CAC, the IAM noted that the respective Codex standards frequently included references to the methods developed by ISO/TC 34/SC 9 "Microbiology" and ISO/TC 147/SC 4 "Water Quality - Microbiological Methods".

Nutrition and Foods for Special Dietary Uses

20. The IAM noted that several methods were required by the CAC, such as the determination in infant formula and follow-up formula of biotin vitamin H, choline, dietary fibre, carbohydrates, iodine, pantothenic acid, Vitamin A, D, E, K, calcium, ammonium, magnesium, phosphorus and gluten.

21. The Representative of AOAC informed the IAM of a recent meeting in Bonn (Germany) of a Working Group on Methods of Analysis for Special Dietary Foods. She pointed out that various methods suitable for milk and milk-based foods exist, however, that other products, such as cereal-based foods presented a problem.

Methods of Analysis for Irradiated Foods

22. The IAM had before it the status report on methods for the identification of irradiated foods which had been submitted by the Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture. Participants noted that support of collaborative studies using the detection methods for irradiated foods by

interested international organizations of the IAM would promote the development as official, validated methods. Such methods are needed for the following purposes:

- (a) Detection methods (i.e. qualitative yes/no methods) for the verification of label declaration or for the monitoring of food in trade;
- (b) Quantitative methods for the determination of "absorbed radiation dose" to check compliance with legal limits (N.B. these methods cannot determine whether good irradiation practices have been followed just as determination of pesticide residues cannot determine whether "good agricultural practices" have been followed).

23. The IAM was also informed that CAC would be invited, in due course, to endorse or temporarily endorse any official methods developed through cooperation with interested international organizations, for irradiated foods.

Methods for the Detection and Determination of Radionuclide Contamination of Foods

24. The Chairman recalled that this topic had been included in the agenda as a result of the discussions at the previous IAM. The IAM noted that AOAC was developing methods in this field and that some of the methods prepared by ISO/TC 147/SC 3 "Water Quality Radiological Methods" were also applicable to foods. Although for the time being there were no special requests by the CAC, it was agreed to pursue discussion of methods for the detection and determination of radionuclide contamination at the next IAM.

Cocoa Products and Chocolate

25. The Representative of the Codex Secretariat informed the IAM that the respective Codex Committee had adjourned *sine die*. Referring to the meeting of the Working Group on Endorsement to be held on 7 November 1992, he said that several methods would have to be developed, for example methods for cocoa powders and dry cocoa-sugar mixtures, fat reduced cocoa powders composite and filled chocolate and cocoa butter confectionery. The Chairman regretted that no Representative of IOCCC was present at the IAM who could provide information on the current status of work within this Organization.

Milk and Milk Products and Edible Ices

26. The Representative of IDF informed the IAM that IDF, ISO and AOAC, having cooperated for over 20 years, had recently welcomed CEN in a quadripartite arrangement for the new Technical Committee CEN/TC 302 which was expected to start work in 1993. Details remain to be elaborated. He also said that the structure of groups of experts was being reorganized for greater efficiency and better use of expertise. While the need for methods was apparently increasing, the availability of experts to develop them was obviously declining. Proprietary laboratory techniques had continued under discussion. IDF and AOAC had developed mechanisms to validate and publish standards based on proprietary methods. Laboratory Quality Assurance and Good Laboratory Practice were receiving special attention. A Seminar was held in Sonthofen (Germany) in May 1992 under the joint auspices of AOAC, CEC, IDF and its German National Committee on this topic. Authors of new and revised standards would be required to include QA and GLP provisions in the text. Proficiency testing was being considered in the context of a proposed international network of dairy laboratories.

27. The Representative of CEN confirmed that the new CEN Technical Committee on Milk and Milk Products would use and endorse the standards resulting from the joint work of IDF, AOAC and ISO in accordance with the provisions of the Vienna Agreement between ISO and CEN. The main purpose of these new provisions was to facilitate the transposition of international standards into European standards whenever possible so that duplication of effort at the international and regional levels was avoided.

Fats and Oils

28. The IAM noted the existing good cooperation between IUPAC, AOAC and ISO on projects of common interest. It was also informed that CEN had recently decided to establish a Technical Committee in this field which was expected to use, as far as possible, the existing ISO standards in this field.

29. In response to a query by the Chairman, the Representative of the Codex Secretariat confirmed that the question of multiple references in Codex standards to technically equivalent methods developed by different organizations was still under review.

Fruit Juices

30. The IAM regretted that FIJU could not attend and provide information on relevant activities. It noted the work of CEN in this area which would contribute to the publication of the first European standards on methods of analysis for fruit juices in 1993.

Processed Fruits and Vegetables

31. The Representative of the Codex Secretariat informed the IAM that the respective Codex Committee had adjourned *sine die* and that it was not possible to formulate specific requests for methods at this stage.

Processed Meat and Poultry Products

32. The Representative of ISO gave a brief account of the relevant activities, including the development of methods for the determination of starch, colouring agents, ascorbic acid, nitrate and histamines. There were no specific requests by CAC.

Fish and Fishery Products

33. The Representative of the Codex Secretariat mentioned that the Committee on Fish and Fishery Products had established a Working Group for the revision of existing standards and methods. One of the main problems encountered was that many methods had been submitted without sufficient data from collaborative studies which were required for the endorsement of these methods.

Sugars

34. The IAM noted that the CAC had approached ICUMSA in order to obtain the missing data for the methods required and that the existing methods for powdered sugar and icing sugar were currently being examined with a view to their applicability to other commodities. The Representative of IFG offered to examine the methods for fructose.

Starch Hydrolysis Products

35. The IAM was informed on the relevant work of ISO/TC 93 "Starch, including derivatives and by-products". CAC and CEN had expressed interest in the standards developed by this Committee which will hold its next meeting on 20 November 1992. The Representative of IFG mentioned in this context that the ISO methods for modified starches were based on those of the Corn Refiners Association (CRA) of the United States.

Cereals, Pulses and Legumes

36. The Representative of ICC introduced the activity report submitted to the IAM and said that some of the methods published by this Organization were currently being reviewed with respect to the statistical evaluation.

37. The Representative of ISO, after drawing attention to the activity report of ISO/TC 34/SC 4 "Cereals and Pulses" reported that comparative results on the applicability of ISO and AOAC methods for the determination of fat acidity had been provided by the French member of SC 4. These results supported the endorsement of ISO 7305 as a Codex method. ISO 7305: 1986 would be revised to express results as mg KOH units.

38. The Representative of AOAC noted that differences between ISO 7305: 1986 and the relevant AOAC method were not caused by the methodology but rather by the provisions in the Codex standard which was based on an AOAC/AACC method.

39. The Representative of NMKL informed the IAM that a new method for low contents of Ochratoxin A (at the level of 10 ng/g) had been jointly developed by AOAC, IUPAC and NMKL. This method will be proposed to CEN TC 275 Working Group on Mycotoxins. The Representative of NMKL further informed the IAM that the method was currently under further testing within NMKL to investigate its applicability to the determination of Ochratoxin A in cereals and baby food at a level lower than 10 ng/g. The Representative of ICC offered the cooperation of this organization on the further development of this method.

40. The IAM also noted that the Codex Committee on Cereals, Pulses and Legumes had expressed interest in a method for the determination of organic and inorganic extraneous matter in cereal products, for which AOAC offered its support.

Mineral Waters

41. The Representative of ISO was pleased to note that references to the methods developed by ISO/TC 147 "Water Quality" had been included in the Regional Codex Standard on Natural Mineral Waters, which would eventually be transformed into a worldwide Codex standard.

Wines and Spirits

42. The Representative of OIV gave an account of the work of the OIV Sub-Commission on Methods of Analysis. She emphasized the importance of the existing good cooperation between OIV, AOAC and ISO with a view to achieving mutual acceptance of methods after validation. The OIV has already developed a programme of collaborative studies which is being coordinated by Mr. Wittkowsky of the Federal Health Institute in Berlin (Germany). Matters of particular interest to the IAM were:

- Residues of antifoams in wines (results of the collaborative study to be published shortly);
- Sorbitol in wines (a collaborative study is ongoing);
- Determination of water addition in wines by NMR (a collaborative study is planned);
- Collection of methods for spirits and wine derived alcoholic beverages;
- Revision of the Oenological Code, i.e. the Code concerning the products used in wine making;
- Biogenic amines in wines (e.g. histamine);
- Determination of ethyl carbamate traces and reduction of ethyl carbamate concentration by means of urease addition;
- Recommendations for the reduction of lead content by technological means to achieve a maximum limit of 0.25 mg/l;

- Establishment of a data base for the reduction of other metallic elements in wines.

43. The Representative of OIV also informed the IAM that her organization recommended the limits for pesticide residues as specified for grapes by the CAC. A list of pesticide residues present in wines would be established by OIV with a view to specifying limits. She added that the English text of the OIV methods would be available by the end of 1993.

OTHER ACTIVITIES OF INTEREST TO THE IAM

44. The Representative of AOAC raised the issue of ownership rights for methods and copyright. He offered to prepare a draft paper on this subject for consideration at the next IAM. The Representative of IDF added that recognition of authorship of standards for the scientists who had contributed their expertise was under discussion in his organization and could be examined at the same time. The Representatives of IDF, NMKL, ICUMSA and ISO agreed to cooperate and to participate in a Working Group which would be established to consider this important subject.

HARMONIZED TERMINOLOGY IN THE FIELD OF METHODS OF ANALYSIS AND SAMPLING

45. The Representative of AOAC informed the IAM of the present status of IUPAC work of terminology and nomenclature. Work had also been started on terminology related to collaborative studies. He invited interested organizations to collaborate with IUPAC and AOAC on this subject.

EXCHANGE OF VIEW ON PROPRIETARY LABORATORY TECHNIQUES VERSUS TRADITIONAL METHODOLOGY

46. The Representative of AOAC introduced a first draft paper on this subject which had been prepared by her organization. She added that AOAC was currently working on a kind of certification mark for test kits. Manufacturers may submit their products to the AOAC Research Institute for testing with a view to obtaining such a certificate mark.

47. Several organizations expressed interest in this topic and offered to cooperate. Accordingly, all organizations participating in the IAM were invited to send their comments on the first draft paper to AOAC, with a copy to the European Representative of AOAC, by the end of February 1993. This will enable AOAC to consider, together with IDF, ISO and other international organizations, the comments received and prepare a new draft for discussion at the next IAM. The Chairman welcomed this initiative by AOAC which would eventually result in a harmonized document that could be used among the interested organizations.

PROPOSALS FOR REORGANIZATION OF THE IAM

48. The Representative of AOAC introduced a paper she had prepared which contained some proposals for the future organization of the Inter-Agency Meetings. Whilst recognizing that the IAM in its present form served a useful purpose as an Advisory Group to the CCMAS and provided a forum for discussion and exchange of information between interested organizations, she felt that the present structure of meetings should be changed, taking into account the following proposals:

- Retain the IAM for all agenda items except methods reports;
- Each organization should prepare one or two posters for the agenda item "Methods of Analysis required by CAC";
- Arrange the IAM so as to enable representatives to stay on the first and second day of the CCMAS session.

49. During the ensuing discussion, different views were presented. Some of the organizations, including IDF, supported the proposals for the reorganization of IAM. Others, including ICC, IFG, ISO, NMKL and the Representative of the Codex Secretariat expressed some reservations. Finally it was agreed to organize the next IAM as in the past, e.g. as a meeting of one full day, prior to the session of CCMAS. However, in response to the suggestions made by the Representative of AOAC, it was agreed that all participating organizations should submit written activity reports to the Secretariat for circulation in advance of the IAM. This would in future avoid the rather lengthy presentation of reports under the respective agenda item so that more time could be made available for discussion and exchange of views on matters of common interest to all organizations.

ANY OTHER BUSINESS

50. There were no suggestions for discussion under this agenda item.

DATE OF THE NEXT IAM

51. It was agreed to hold the next IAM in association with the Nineteenth Session of CCMAS.

52. Before closing the Ninth IAM, the Chairman thanked the participants for their work on the common objectives, the Hungarian hosts for the excellent arrangements made, the interpreter and the secretary.

53. On behalf of all participants, Dr. Horwitz (AOAC) thanked the Chairman for his able conduct of the meeting.

ATTENDANCE LIST OF DELEGATES FOR THE NINTH INTER-AGENCY MEETING
Budapest, 6 November 1992

Chairman: Mr. G. Castan
Président: Association française de normalisation
Presidente: Tour Europe
Cedex 7
92049 Paris La Défense
France

Secretary: Mr. K.G. Lingner
Secrétaire: ISO Central Secretariat
Secretaria: 1, Rue de Varembe
CH-1211 Geneva 20
Switzerland

ASSOCIATION OF OFFICIAL ANALYTICAL
CHEMISTS (AOAC)

Mr. R.R. Christensen
Executive Director
AOAC International
2200 Wilson Blvd., Suite 400
Arlington, VA 22201-3301
U.S.A.

Mr. J. Lawrence
Health and Welfare Canada
Banting Research Center
Ottawa, Ontario KIA 0L2
Canada

Dr. W. Horwitz
Center for Food Safety and Applied
Nutrition (HFF-7)
Food and Drug Administration
200 C Street S.W.
Washington, D.C. 20204
U.S.A.

Mr. G.W. Diachenko
Chief, Food Formulation Branch
Food and Drug Administration
HFF-413
200 C Street, S.W.
Washington, DC 20204
U.S.A.

Mrs. M. Lauwaars
European Representative
AOAC International
P.O. Box 153
6720 AD Bennekom
Netherlands

Mr. R. Wood
Ministry of Agriculture,
Fisheries and Food
Food Science Laboratory
Colney Lane
Norwich NR4 7U, U.K.

Mr. E. Elkins Jr.
Director
Chemistry Division
National Food Processors Association
1401 New York Avenue, N.W.
Suite 400
Washington, D.C. 20005, U.S.A.

Mr. W.J. de Koe
Food Standard Officer
Ministry of Welfare, Health and
Cultural Affairs
Sir Winston Churchillaan 362
P.O. Box 5406
2280 H.K. Rijswijk
Netherlands

CODEX COMMITTEE ON METHODS OF
ANALYSIS AND SAMPLING

Prof. P. Biacs
Central Food Research Institute
Herman Ottó út 15
Budapest
Hungary 1022

EOQ

Mr. P. Molnár
Central Food Research Institute
Herman Ottó út 15
Budapest
Hungary 1022

EUROPEAN COMMITTEE FOR STANDARDIZATION
(CEN)

Mr. M. Jeanson
Rue de Stassart 36
B-1050 Brussels
Belgium

INTERNATIONAL CEREAL CHEMISTRY (ICC)

Mr. H. Glattes
Secretary General
P.O. Box 77
Wienerstrasse 22a
A-2320 Schwechat
Austria

Mr. G. Schögl
Deputy Secretary General
P.O. Box 77
Wienerstrasse 22a
A-2320 Schwechat
Austria

INTERNATIONAL DAIRY FEDERATION (IDF)

Mr. E. Hopkin
Secretary General
41 Square Vergote
1040 Brussels
Belgium

INTERNATIONAL FEDERATION OF GLUCOSE
MANUFACTURERS (IFG)

Dr. B. Whitehouse
Quality Assurance Manager
Cerestar SA/NV
Havenstraat 84
B-1800 Bruxelles
Belgium

JOINT FAO/WHO FOOD STANDARDS
PROGRAMME

Dr. E. Casadei
Food Standards Officer
Joint FAO/WHO Food Standards
Programme
FAO
Via delle Terme di Caracalla
00100 Rome
Italy

NORDIC COMMITTEE ON FOOD ANALYSIS
(NMKL)

Mrs. H. Wallin
Secretary General
VTT/Food Research Laboratory
P.O. Box 203
SF-02151 Espoo
Finland

INTERNATIONAL VINE AND WINE OFFICE
(OIV)

Mrs. B. Mandrou
Faculté de Pharmacie
34060 Montpellier Cedex 1
France

INTERNATIONAL ORGANIZATION OF
STANDARDIZATION (ISO)

Mr. R. Zwart
Nederlands Normalisatie-instituut
Kalfjeslaan 2
P.O. Box 5059
2600 GB Delft
The Netherlands

Ms. N. Normand
Association Française de
Normalisation
Tour Europe - Cédex 7
92049 Paris La Défense
France

Mr. G. Castan
Expert
8 rue de Varize
75016 Paris
France

Mrs. E. Nagy
Secretary of ISO/TC 34
Hungarian Office for Standardization
Secretariat of ISO/TC 34
"Agricultural Food Products"
Üllői út 25
H-1095 Budapest
Hungary

OBSERVER

Dr. W.G. de Ruig
State Institute for Quality Control
of Agricultural Products
Postbus 230
6700 AE Wageningen
Netherlands

ALINORM 93/23
APPENDIX V

**LIST OF METHODS OF ANALYSIS
CONSIDERED FOR ENDORSEMENT
BY THE EIGHTEENTH SESSION
OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING**

Note: * Endorsed by the Eighteenth Session of the CCMAS.

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
Canned crab meat ((516)) (090-1981-V)	Water capacity of container	Method described in standard	Weighing	I	E*
Canned crab meat ((524)) (090-1981-V)	Net contents	Method described in standard 94-1981	Weighing	I	E*
Canned mackerel and jack mackerel ((520)) (119-1981-V)	Net contents	Method described in standard	Weighing	I	E*
Canned salmon ((521)) (003-1981-V)	Net contents	Method described in standard 94-1981	Weighing	I	E*
Canned sardines and sardine-type products ((519)) (94-1981-V)	Net contents	Method described in standard	Weighing	I	E*
Canned shrimps or prawns ((518)) (037-1981-V)	Water capacity of container	Method described in standard 090-1981	Weighing	I	E*
Canned shrimps or prawns ((522)) (037-1981-V)	Net contents	Method described in standard 94-1981	Weighing	I	E*
Canned tuna and bonito ((517)) (070-1981-V)	Water capacity of container	Method described in standard 090-1981	Weighing	I	E*
Canned tuna and bonito ((523)) (070-1981-V)	Net contents	Method described in standard 94-1981	Weighing	I	E*
Cocoa butters ((168)) (086-1981-VII)	Copper < 0.4 mg/kg	IUPAC Method Pure and Appl. Chem., 60, No 6 p. 893 (1988) ISO 8294 (in press) AOAC (1990) 990.05	Graphite furnace	II	E*
Cocoa butters ((169)) (086-1981-VII)	Lead < 0.5 mg/kg	IUPAC Method Pure and Appl. Chem. Vol 63, No. 8, p.1183, 1991	Graphite Furnace	II	E*
Cocoa butters ((170)) (086-1981-VII)	Iron < 2 mg/kg	IUPAC Method Pure and Appl. Chem., 60, No 6 p. 893 (1988) ISO 8294 (in press) AOAC (1990) 990.05	Graphite Furnace	II	E*
Dextrose anhydrous ((92)) (007-1981-III)	Sulphur dioxide < 20 mg/kg	ISO 5379:1983	Acidimetric and nephelometric	IV	E*
Dextrose monohydrate ((96)) (008-1981-III)	Sulphur dioxide < 20 mg/kg	ISO 5379:1983	Acidimetric and nephelometric	IV	E*

((note reference)) ; (standard-year-volume) ;

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
Dried glucose syrup ((104)) (010-1981-III)	Sulphur dioxide < 40 mg/kg, < 150 mg/kg	ISO 5379:1983	Acidimetric and nephelometric	IV	E*
Edible low erucic acid rapeseed oil ((500)) (123-1981-XI)	Erucic acid content < 5 % (m/m) of the component fatty acids	IUPAC 7th Ed., 1984 2.311 ISO 8209:1986	Gas chromatographic	II	E*
Fats and oils ((297)) (1000-XI)	Copper < 0.1 - 0.4 mg/kg	IUPAC Method Pure and Appl. Chem., 60, No 6 p. 893 (1988) ISO 8294 (in press) AOAC (1990) 990.05	Graphite Furnace	II	E*
Fats and oils ((298)) (1000-XI)	Lead < 0.1 mg/kg	IUPAC Method Pure and Appl. Chem., Vol. 63 No. 8, p.1183, 1991	Graphite Furnace	II	E*
Fructose ((125)) (102-1981-III)	Conductivity ash < 0.1 %	ICUMSA (1979) 85-86	Conductimetric	I	E*
Fructose ((126)) (102-1981-III)	Colour < 30 ICUMSA units	ICUMSA (20th), General Subject 2 Report, Appendix 1 and Recommendation No 2	Colorimetric	I	E*
Fructose ((127)) (102-1981-III)	pH 4.5 - 7.0	ICUMSA (1979) 131	Potentiometric	IV	E*
Glucose syrup ((100)) (009-1981-III)	Sulphur dioxide >40 mg/kg, < 400 mg/kg	ISO 5379:1983	Acidimetric and nephelometric	IV	E*
Grated desiccated coconut ((510)) (177-1991-II)	Total acidity of extracted oil < 0.3 (m/m) as lauric acid	Described in the standard	Titration of extracted oil	IV	E*
Grated desiccated coconut ((511)) (177-1991-II)	Moisture < 3 % (m/m)	AOAC (1990) 925.40	Loss on drying	I	E*
Grated desiccated coconut ((512)) (177-1991-II)	Oil content > 55 % (m/m)	AOAC (1990) 948.22		I	E*

((note reference)) ; (standard-year-volume) ;

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
Grated desiccated coconut ((513)) (177-1991-II)	Ash < 2.5 % (m/m)	AOAC (1990) 950.49		I	E*
Grated desiccated coconut ((514)) (177-1991-II)	Extraneous vegetable matter < 15 fragments per 100 g	method described in the standard	counting extraneous material with the naked eye	IV	E*
Guidelines for Acrylonitrile ((527)) (2016-GI-AI/91)	Acrylonitrile 0.02 mg/kg in food	AOAC (1990) 985.13	Gas chromatographic	II	E*
Guidelines for Vinyl Chloride Monomer ((528)) (2026-GI-AI/91)	Vinyl Chloride Monomer 1.0 mg/kg (packaging)	ISO 6401:1985	Gas chromatographic	II	E*
Guidelines for Vinyl Chloride Monomer ((530)) (2017-GI-AI/91)	Vinyl Chloride Monomer 0.01 mg/kg in food	Commission Directive 81/432/EEC O.J. No. L.167, p. 6, 24.6.81	Gas chromatography "head-space"	II	E*
Infant Formula and Follow-up Formula ((531)) (72 and 156-IX)	Total Dietary Fiber	AOAC (1990) 991.43	Enzymatic gravimetric	I	E*
Lactose ((105)) (011-1981-III)	Anhydrous lactose > 99.0 %	ICUMSA (17th)S.14 Rept., Appendix 1 amended ICUMSA (20th) S.15 Rept., Table 6 & Rec. 4	Titrimetric	IV	E*
Lactose ((106)) (011-1981-III)	Sulphated ash < 0.3 % (m/m)	ICUMSA (1979) 83-84	Gravimetric	IV	E*
Lactose ((108)) (011-1981-III)	pH 4.5 - 7.0	ICUMSA (1979) 131	Potentiometric	IV	E*
Pearl millet flour (African regional standard) ((418)) (170-1989-XVIII)	Colour 18 - 30 units	Modern Cereal Chemistry, 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetric using specific colour grader	IV	E*
Powdered dextrose (Icing dextrose) ((121)) (054-1981-III)	Sulphur dioxide < 20 mg/kg	ISO 5379:1983	Acidimetric and nephelometric	IV	E*
Processed tomato concentrates ((27)) (057-1981-II)	Mineral impurities < 60 mg/kg	AOAC (1990) 971.33	Ashing	IV	E*

((note reference)) ; (standard-year-volume) ;

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
Soft sugars (81) (006-1981-III)	Sucrose plus invert sugar > 88 % , > 97 %	ICUMSA (17th) S. 14 Rept. Appendix 1 amended ICUMSA (20th) S. 15 Rept Table 6 and Rec. 4 and ICUMSA (18th) 120-122	Titrimetric	I	E*
Soft sugars (83) (006-1981-III)	Invert sugar in sugars with content >10%	ICUMSA (17th) S. 14 Rept. Appendix 1 amended ICUMSA (20th) S. 15 Rept. Table 6 and Rec. 4	Titrimetric	I	E*
Soft sugars (84) (006-1981-III)	Sulphated ash < 3.5 % (m/m)	ICUMSA (1979) 83-84	Gravimetric	I	E*
Soft sugars (85) (006-1981-III)	Conductivity ash < 0.2 % (m/m)	ICUMSA (1979) 85-86	Conductimetric	I	E*
Soft sugars (87) (006-1981-III)	Colour white to dark brown	ICUMSA (20th), General Subject 2 Report, Appendix 1 and Recommendation No 2	Colorimetric	I	E*
Sorghum flour (432) (173-1989-XVIII)	Colour > 18, < 30 units	Modern Cereal Chemistry, 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetric using specific colour grader	IV	E*
Special Foods (216) (980-IX)	Total Dietary Fiber	AOAC (1990) 985.29	Enzymatic gravimetric	I	E*
Special Foods (218) (980-IX)	Loss on drying	AOAC (1990) 925.23	Drying in vacuo	I	E*
Special Foods (223) (980-IX)	Linoleate (in the form of glycerides) > 300 mg/100 kjoul	AOAC (1990) 922.06; 979.19	Acid hydrolysis, spectrophotometric	II	E*
Table olives (35) (066-1987-II)	Acidity of brine > 0.4 % (m/m)	Method described in standard	Titrimetric	IV	E*
Table olives (36) (066-1987-II)	pH of brine < 4.0, < 4.5	Method described in standard	Potentiometric	IV	E*
White sugar (64) (004-1981-III)	Polarisation in sugars requiring no clarification > 99.5 degrees S	ICUMSA (18th) 341-344, ICUMSA (19th) 66-68 amended by ICUMSA (20th) 190-193.	Polarimetric	II	E*

((note reference)) ; (standard-year-volume) ;

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
White sugar ((65)) (004-1981-III)	Polarisation in sugars requiring clarification	ICUMSA (1979) 25-30, amended ICUMSA (18th) 175-180, 189-190 and ICUMSA (19th) 66-68 and 197	Polarimetric	II	E*
White sugar ((68)) (004-1981-III)	Invert sugar in sugars with content >10%	ICUMSA (17th) S. 14 Rept. Appendix 1 amended ICUMSA (20th) S. 15 Rept. Table 6 and Rec. 4	Titrimetric	I	E*
White sugar ((69)) (004-1981-III)	Conductivity ash < 0.04 - 0.1 %	ICUMSA (1979) 85-86	Conductimetric	I	E*
White sugar ((71)) (004-1981-III)	Colour 60 - 150 ICUMSA units	ICUMSA (20th), General Subject 2 Report, Appendix 1 and Recommendation No 2	Spectrophotometric	I	E*
Fruit juices ((265)) (990-X)	Carbon dioxide	IFJU Method No 42, 1976	Back-titration after precipitation	II	NE
Fruit juices ((267)) (990-X)	Ethanol < 2 - 5 g/kg	IFJU Method No 53, 1983	Enzymatic	II	NE
Fruit juices ((281)) (990-X)	Lead < 0.2 - < 0.3 mg/kg	ISO 6633:1984	Atomic absorption	II	NE
Natural Mineral Waters (European Regional Standard) ((453)) (108-1981-XII)	Arsenic 0.05 mg/l	ISO 6595:1982		III	NE
Vinegar ((390)) (162-1987-XII)	Iron < 10 mg/kg	IFJU Method No 15, 1964	Photometric	II	NE
Powdered sugar (Icing sugar) ((77)) (005-1981-III)	Loss on drying < 0.1 %	ICUMSA (1979) 113-115, amended ICUMSA (19th) 348	Gravimetric	I	TBE
Bouillons and consommés ((138)) (117-1981-IV)	Sodium chloride < 12.5 g/L	A11BP Method No 2/4	Volhard titration	III	TE
Dextrose anhydrous ((89)) (007-1981-III)	D-glucose > 99.5 % (m/m)	ISO 5377:1981	Titrimetric	I	TE
Dextrose anhydrous ((90)) (007-1981-III)	Total solids > 98.0 %	ISO 1741:1980	Vacuum oven	I	TE

((note reference)) ; (standard-year-volume) ;

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
Dextrose anhydrous ((91)) (007-1981-III)	Sulphated ash < 0.25 % (m/m)	ISO 5809:1982	Single sulphonation	I	TE
Dextrose monohydrate ((93)) (008-1981-III)	D-glucose > 99.5 % (m/m)	ISO 5377:1981	Titrimetric	I	TE
Dextrose monohydrate ((94)) (008-1981-III)	Total solids > 90.0 % (m/m)	ISO 1741:1980	Vacuum oven	I	TE
Dextrose monohydrate ((95)) (008-1981-III)	Sulphated ash < 0.25 % (m/m)	ISO 5809:1982	Single sulphonation	I	TE
Dried glucose syrup ((101)) (010-1981-III)	Total solids > 93.0 % (m/m)	ISO 1742:1980	Vacuum oven	I	TE
Dried glucose syrup ((102)) (010-1981-III)	Reducing sugar > 20.0 %	ISO 5377:1981	Titrimetric	I	TE
Dried glucose syrup ((103)) (010-1981-III)	Sulphated ash < 1.0 % (m/m)	ISO 5809:1982	Single sulphonation	I	TE
Edible Cassava flour ((503)) (176-1991-XII)	Ash < 3 % (m/m)	AOAC (1990) 923.03		I	TE
Foods with low-sodium content (including salt substitutes) ((251)) (053-1981-IX)	Sodium and potassium Na: < 120 mg/100 g, K: No limit	AOAC (1990) 984.27	ICP emission spectrometry	II	TE
Foods with low-sodium content (including salt substitutes) ((252)) (053-1981-IX)	Calcium and magnesium Mg: < 20 % of sum of potassium, calcium, ammonium cations	AOAC (1990) 965.09	Atomic absorption	II	TE
Foods with low-sodium content (including salt substitutes) ((253)) (053-1981-IX)	Ammonium < 3 % (m/m)	AOAC (1990) 920.03	Magnesium oxide	II	TE
Foods with low-sodium content (including salt substitutes) ((254)) (053-1981-IX)	Phosphorous < 4 % (m/m)	AOAC (1990) 984.27	ICP emission spectrometry	II	TE

((note reference)); (standard-year-volume);

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
Fructose ((123)) (102-1981-III)	Specific rotation -89 - -93.5 degrees	Zuckerindustrie 113 (1988):1, 49-50	Polarimetric	II	TE
Fructose ((124)) (102-1981-III)	Loss on drying < 0.5 % (m/m)	ISO 1742:1980	Gravimetric	I	TE
Fructose ((128)) (102-1981-III)	Sulphur dioxide < 20 mg/kg	ISO 5379:1983	Colorimetric	II	TE
Glucose syrup ((97)) (009-1981-III)	Total solids > 70.0 % (m/m)	ISO 1742:1980	Vacuum oven	I	TE
Glucose syrup ((98)) (009-1981-III)	Reducing sugar > 20.0 % (m/m)	ISO 5377:1981	Titrimetric	I	TE
Glucose syrup ((99)) (009-1981-III)	Sulphated ash < 1.0 % (m/m)	ISO 5809:1982	Single sulphonation	I	TE
Grated desiccated coconut ((509)) (177-1991-II)	Granularity Extra fine, fine and medium	ISO 2591-1:1988 Test Sieving According to British Standard Mesh Nominal Test Sieves: BS410-1986	Sieving method	I	TE
Natural Mineral Waters (European Regional Standard) ((452)) (108-1981-XII)	Barium 1.0 mg/l	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 65-66		II	TE
Natural Mineral Waters (European Regional Standard) ((454)) (108-1981-XII)	Barium 1.0 mg/l	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol.2, pp. 67-68		III	TE
Natural Mineral Waters (European Regional Standard) ((459)) (108-1981-XII)	Chromium (VI) 0.05 mg/l	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 86-87.		II	TE
Natural Mineral Waters (European Regional Standard) ((468)) (108-1981-XII)	Nitrates 45 mg/l, calculated as NO3-	Handbuch Lebensmittel Chemie (1969)		III	TE
Natural Mineral Waters (European Regional Standard) ((469)) (108-1981-XII)	Nitrates 45 mg/l, calculated as NO3-	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 280-283		III	TE

((note reference)) ; (standard-year-volume) ;

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
Natural Mineral Waters (European Regional Standard) ((474)) (108-1981-XII)	Sulphide 0.05 mg/l, calculated as H ₂ S	Handb. Spurenanal. 1974		II	TE
Natural Mineral Waters (European Regional Standard) ((475)) (108-1981-XII)	Hydrogen carbonate (Bicarbonate HCO ₃)	Examination of Water Pollution Control, WHO Pergamon Press (1982) Vol. 2, pp. 170-175		I	TE
Natural Mineral Waters (European Regional Standard) ((489)) (108-1981-XII)	Total cyanide not more than 0.01 mg/l, calculated as CN	ISO 6703-1:1984		II	TE
Natural Mineral Waters (European Regional Standard) ((490)) (108-1981-XII)	Nitrites Not more than 0.005 mg/l, calculated as NO-2	ISO 6777:1984		II	TE
Natural mineral waters (European Regional Standard) ((353)) (108-1981-XII)	Total dissolved solids < 1000 mg/L	Method described in the standard	Gravimetric	I	TE
Natural mineral waters (European Regional Standard) ((354)) (108-1981-XII)	Total organic matter ≤ 3 mg/L as oxygen	ISO 8467:1986 or AOAC (1990) 973.47	Permanganate digestion	I	TE
Powdered sugar (Icing sugar) ((79)) (005-1981-III)	Sulphur dioxide < 20 mg/kg	ICUMSA (1979) 98-99 (for additive-free products)	Colorimetric	II	TE
Soft sugars ((82)) (006-1981-III)	Invert sugar in sugars with content <10% 0.3 - 12 %	ICUMSA (1979) 55-59	Titrimetric	I	TE
Soft sugars ((86)) (006-1981-III)	Loss on drying < 4.5 %, < 3.0 %	ICUMSA (1979) 113-115, amended ICUMSA (19th) 348	Gravimetric	I	TE
Soft sugars ((88)) (006-1981-III)	Sulphur dioxide < 40 mg/kg	ICUMSA (1979) 98-99 (to be confirmed)	Colorimetric	II	TE
Special Foods ((249)) (980-IX)	Copper, manganese, zinc, magnesium, iron Cu: >60 mg, Mn: >5 ug, Zn: >0.5 mg, Mg: >6 mg and Fe: >0.15 mg/100 kcal	AOAC (1990) 984.27	ICP emission spectrometry	II	TE
Specified animal or mixed animal and vegetable fat products ((341)) (158-1987-XI)	Slip point 31 - 44 C	AOCS Official method Cc 3-25	Open tube	I	TE

((note reference)) ; (standard-year-volume) ;

METHODS OF ANALYSIS CONSIDERED BY CCMAS LISTED BY STATUS

COMMODITY	PROVISION AND LEVEL	METHOD	PRINCIPLE	TY	STATUS
Specified vegetable fat products ((327)) (157-1987-XI)	Slip point 31- 44 C	AOCS Official method Cc 3-25	Open tube	I	TE
White sugar ((66)) (004-1981-III)	Invert sugar in sugars with content < 0.04% (m/m)	ICUMSA (1979) 59-61	Titrimetric	I	TE
White sugar ((67)) (004-1981-III)	Invert sugar in sugars with content 0.1% (m/m)	ICUMSA (1979) 55-56	Titrimetric	I	TE
White sugar ((70)) (004-1981-III)	Loss on drying < 0.1 %	ICUMSA (1979) 113-115, amended ICUMSA (19th) 348	Gravimetric	I	TE
White sugar ((72)) (004-1981-III)	Sulphur dioxide 20 - 70 mg/kg	ICUMSA (1979) 98-99	Colorimetric	II	TE

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- 66 **Invert sugar in sugars with content**
ICUMSA informed the WG that the Official method will be modified during 1992-94, to raise limit from < 0.02 to < 0.08%
- 67 **Invert sugar in sugars with content**
See No 66
- 70 **Loss on drying**
The Working Group was informed by ICUMSA that the Official method should be re-tested during 1992-94.
- 72 **Sulphur dioxide**
The Working Group was informed by IUMSA that the Official method and a CEN alternative method should be tested during 1992-94.
- 77 **Loss on drying**
Status retained until results of collaborative study are provided from ICUMSA through the delegation of the United Kingdom.
- 79 **Sulphur dioxide**
See No 72
- 82 **Invert sugar in sugars with content <10%**
The Working Group was informed by ICUMSA that a new method will be tested during 1992-94.
- 84 **Sulphated ash**
The WG endorsed the method as Type I and requested information by ICUMSA on the applicability of the method to soft sugars.
- 86 **Loss on drying**
See No 70
- 88 **Sulphur dioxide**
See No 72
- 92 **Sulphur dioxide**
The Working Group did not have data indicating that this method had been collaboratively studied and therefore classified it as Type IV.
- 96 **Sulphur dioxide**
See No 92
- 104 **Sulphur dioxide**
See No 92
- 105 **Anhydrous lactose**
The Working Group endorsed the method as Type IV requesting comments provided by ICUMSA on the applicability of the method.
- 106 **Sulphated ash**
The WG endorsed the method as Type IV and requested information by ICUMSA on the applicability of the method to lactose.
- 108 **pH**
The Working Group endorsed the method as Type IV, but requested ICUMSA to provide information on the applicability of the method to lactose.
- 121 **Sulphur dioxide**
See No 92
- 127 **pH**
The Working Group endorsed the method as Type IV and requested ICUMSA to provide information on the applicability

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of the method to fructose.

- 249 **Copper, manganese, zinc, magnesium, iron**
The Working Group temporarily endorsed the method as Type II with the request that the Codex Committee on Food and Nutrition for Special Dietary Uses should confirm the applicability of the method to the standard and consider adopting a more generally available AAS method.
- 251 **Sodium and potassium**
See No 249
- 252 **Calcium and magnesium**
See No 249
- 253 **Ammonium**
See No 249
- 254 **Phosphorous**
See No 249
- 265 **Carbon dioxide**
The Working Group noted that the results of collaborative studies were not provided, and decided to include the method in an Appendix to the endorsed Codex methods.
- 267 **Ethanol**
The Working Group was informed that a recent method has been submitted to collaborative study and data will be available at the next session, provided by the delegation of the United Kingdom.
- 281 **Lead**
No collaborative study data was provided. The Working Group considered that the General Method (AOAC 1990 972.25) was a good method to measure lead in Fruit juices, and recommended that reference to IFJU method should be withdrawn by the Codex publication.
- 327 **Slip point**
A substitute method from ISO 6321:1991 was available, and the WG recommended that the Commodity Committee should consider replacement with this collaboratively studied method.
- 341 **Slip point**
See No 327
- 353 **Total dissolved solids**
Retained as temporary until results of collaborative study are provided. The WG recommended to include the method in an Appendix to the endorsed Codex methods.
- 354 **Total organic matter**
The Working Group recommended that the Commodity Committee specify which of the available procedures contained in the proposed ISO or AOAC methods are appropriate.
See also No 353
- 390 **Iron**
Status retained because the results of collaborative study were not provided. In view of the repeated request the WG recommended to put this method in an Appendix to the list of endorsed Codex Methods awaiting further development.
- 418 **Colour**
The Working Group received information by the ICC, however no collaborative study data was provided. The WG proposed the endorsement of the proposed method as Type IV, pending further development.
- 432 **Colour**
See No 418

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- 452 Barium See No 353
- 453 Arsenic This method was not endorsed because of concerns about its applicability at the maximum permitted level of 0.05 mg/l. The WG decided to include the method in an Appendix to the endorsed Codex methods, awaiting further development.
- 454 Barium See No 353
- 459 Chromium (VI) See No 353 The Working Group recommended that a more modern collaboratively studied general method such as ISO 9174:1990 or AOAC 947.27 should be considered by the Commodity Committee.
- 468 Nitrates See No 353. The Working Group noted that a spectrophotometric method was available ISO 7890:1991 (Part 1 and 2) and this should be recommended to the Commodity Committee.
- 469 Nitrates See No 353 and 459
- 474 Sulphide See No 353 The Working Group noted that a new method ISO 10530:1992 was available and should be considered by the Commodity Committee.
- 475 Hydrogen carbonate (Bicarbonate HC03) See No 353 The Working Group noted that a collaboratively studied method such as AOAC 920.194 was available.
- 489 Total cyanide Information from ISO indicates that the ISO method is applicable at levels as low as 10 microgram/L, but samples with such a low concentration of cyanide cannot be tested in a collaborative study for rapid decomposition of cyanide in samples. A ion chromatography method is being developed.
- 490 Nitrites SEE No 353
- 500 Erucic acid content The reference for the collaborative study for the IUPAC Method for Erucic Acid is Pure & Applied Chemistry, Vol 56, No 2, pp 301-304, 1984. The IUPAC Method 2.311 and the ISO 8209:1986 are technically equivalent, indeed virtually identical apart from the format.
- 503 Ash It was noted that the ISO method was under revision on the basis of ICC 104/1 and that only the high temperature procedure (900 C) was included in the new text. The WG temporarily endorsed an AOAC method working at 550 C and requested the Commodity Committee on the applicability of the method.
- 509 Granularity The WG temporarily endorsed this method but recommended that ISO should refer at the next session on the correlation for test sieves of ISO and British Standard.
- 513 Ash The WG noted that ISO method was under revision and that only the high temperature method was included in the revision. The WG endorsed an AOAC method working at 550 C, identical to the unrevised ISO method. AOAC was requested to include this

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surplus method in the next edition of Off.Meth. of Analysis.

- 518 **Water capacity of container**
Ref. Alinorm 91/18 para. 135 and Alinorm 91/40 para 316
- 521 **Net contents**
Ref. Alinorm 91/40 para 316
- 522 **Net contents**
Ref. Alinorm 91/40 para. 316
- 523 **Net contents**
Ref. Alinorm 91/40 para 316.
- 524 **Net contents**
Ref. Alinorm 91/40 para. 316
- 528 **Vinyl Chloride Monomer**
This method was only endorsed for vinyl chloride monomer in packaging material.
- 530 **Vinyl Chloride Monomer**
Results of collaborative study were published in J. Assoc. Publ. Analysts, 1982, 20, 55-65