

codex alimentarius commission

FOOD AND AGRICULTURE
ORGANIZATION
OF THE UNITED NATIONS

WORLD HEALTH
ORGANIZATION

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ALINORM 91/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Nineteenth Session

Rome, 1-10 July 1991

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

Seventeenth Session

Budapest, 8-12 April 1991

Note: This report incorporates Codex Circular Letter CL 1991/14-MAS.

w/z 7363

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CX 4/50.2

CL 1991/14-MAS
April 1991

TO: - Codex Contact Points
- Participants at the Seventeenth 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 Seventeenth Session of the Codex Committee on Methods of Analysis and Sampling (CCMAS)

The report of the Seventeenth Session of the above Committee (ALINORM 91/23) will be considered by the Nineteenth Session of the Codex Alimentarius Commission (Rome, 1-10 July 1991).

PART A: MATTERS OF INTEREST TO THE COMMISSION

(1) Sampling for Net Content (paras. 18-20, ALINORM 91/23)

The Committee agreed that there was no need for further work in the area of sampling for net content, because criteria had been established for such sampling plans (Appendix V, ALINORM 91/23), and the Committee did not support the proposal for adoption by the Commission of a general method for net weight determination as outlined in the OIML Recommendation.

(2) Endorsement of Methods of Sampling (paras. 21-23, ALINORM 91/23)

The Committee endorsed several sampling plans as proposed by the Coordinating Committee for Africa and the Codex Committee on Cereals, Pulses and Legumes. The Committee temporarily endorsed the sampling plans adopted for pesticide residues in food, for mercury, cadmium and lead, pending the outcome of its review of general sampling plans for contaminants.

(3) Amendment of the Definition of Type I Method (paras. 40-43, ALINORM 91/23)

The Committee noted that the definition of the Type I Method in the Procedural Manual (7th Edition, 1989, page 143) was incorrect as this method could not be used "calibration purposes". It recommended to the Commission that the definition should be amended by deleting the final phrase "and which can serve for calibration purposes" and substituting it with the phrase "and serves by definition as the only method for establishing the accepted value of the item measured".

PART B: MATTERS OF INTEREST TO GOVERNMENTS

(1) Recommendations for a checklist of information required to evaluate methods of analysis submitted to the CCMAS for endorsement (paras. 29-31, ALINORM 91/23)

At its 14th Session the CCMAS established a list of information required for submission by the Codex Commodity Committees of methods of analysis to be endorsed as Codex methods. The CCMAS considered that the Codex procedures established for the evaluation of methods submitted for endorsement should be revised, taking into

account the first two IUPAC protocols on "Harmonized Protocols for the Adoption of Standardized Methods and for Presentation of their Performance Characteristics" resulting from the Second International Workshop held in Washington, D.C., U.S.A., 17-18 April 1989 (CX/MAS 91/7).

Governments and interested international organizations are requested to send their comments on the current Checklist, attached as Appendix III to this report (ALINORM 91/23) to Dr. H. Horwitz, Food and Drug Administration, HFF-7, 200 C Street S.W., Washington, D.C. 20204, U.S.A., no later than 30 April 1992, with a copy to the Chief, Joint FAO/WHO Food Standards Programme, FAO, Via delle Terme di Caracalla, 00100 Rome, Italy.

(2) Review of General Methods for Contaminants (paras. 52-53, ALINORM 91/23)

The Delegation of Canada agreed to revise the list of methods on the basis of comments received at the 17th Session of the CCMAS. The list would be available to governments and interested international organizations for comments and for discussion at the 18th Session of the CCMAS.

(3) General Method of Ashing for the Determination of Heavy Metal Contaminants (para. 54, ALINORM 91/23)

The Delegation of the U.S.S.R. agreed to undertake a review of the proposed method on the basis of comments received at the 17th Session of the CCMAS. A revised version of the methods would be available to governments and interested international organizations for comments and for discussion at the 18th Session of the CCMAS.

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Seventeenth Session, Budapest, 8-12 April 1991

SUMMARY AND CONCLUSIONS

The Committee:

- expressed its interest in work on laboratory certification, proficiency programmes and guidelines on quality assurance in laboratories, and in combining its future work on methodology with the study of such laboratory systems. (Para 15)
- agreed that there was no need for further work in the area of sampling plans for net weight or net content, and stated that the provisions contained in the Codex General Labelling Standard were adequately covered by available national and international practices. (Para 20)
- endorsed the provisions concerning sampling in a number of Codex Standards (Para 22)
- endorsed the provisions concerning methods of analysis in a large number of Codex Standards (Para 49 and Appendix II)
- agreed to endorse the sampling plans adopted for pesticide residues in food (CAC/PR 5-1984) for mercury, cadmium and lead pending the outcome of its review of general sampling plans for contaminants. (Para 23)
- expressed its interest in the area of proprietary analytical techniques which, it was noted, were replacing traditional techniques in many laboratories for some specific analytes such as mycotoxins. It particularly noted the need for independently-derived information on the performance of these new techniques, especially for those agencies which intended to use them for enforcement purposes. (Para. 26)
- agreed to revise the checklist for information required to evaluate methods of analysis submitted to CCMAS for endorsement (Report of the 14th Session of the CCMAS, ALINORM 85/23, Appendix II). (Para 31)
- recommended to the Commission that the definition of Type I Method in the Procedural Manual (7th Edition, 1989, page 143) should be amended. (Para 43)
- confirmed that all methods which corresponded to defined Codex criteria were considered as suitable methods for Codex purposes. The Committee also supported the view that greater attention should be given to the elaboration and selection of general methods of analysis, multi-analyte methods for trace analysis, and ELISA methods. (Para 45-46)
- recommended adoption of a General Method for the Determination of Copper in Foods as a Type III Codex Method, noting some limitations of the method. (Para 55)

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX ALIMENTARIUS COMMISSION
Nineteenth Session, Rome, 1-10 July 1991

REPORT OF THE
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Seventeenth Session, Budapest, 8-12 April 1991

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Seventeenth Session from 8 - 12 April 1991 in Budapest, by courtesy of the Government of Hungary. The Session was attended by 87 delegates and observers from 22 countries and 8 international organizations. A complete list of participants, including the Secretariat is provided in Appendix I to this report.

2. Dr. K. Sütö, President of the Hungarian National Codex Committee, welcomed the participants, and introduced Dr. K. Szöke, Deputy Secretary of State for Agriculture. Dr. Szöke gave a short review of the changes of the political system in Hungary since the first free elections for 40 years, held in 1990. The agriculture and food industries, he noted, retained an important place in the Hungarian economy, accounting for 22% of employment and more than 30% of the value of Hungarian exports. Dr. Szöke stated that the major tasks confronting this sector in the coming years would be the adoption and application of EEC requirements in the field of food control, and that therefore Hungary's participation in the FAO/WHO Codex Alimentarius activities had become of increasing importance.

ADOPTION OF THE AGENDA (Agenda Item 2)

3. The Provisional Agenda, CX/MAS 91/1, was adopted as the Agenda for the Session. The Committee agreed to include a discussion of a paper prepared by The Netherlands (Conference Room Document No.7) on Analytical Criteria in Food Safety Control under Other Business, Agenda Item 17.

APPOINTMENT OF RAPORTEURS (Agenda Item 3)

4. The Committee appointed Dr. W. Dubbert (USA) and Dr. F. Bourguignon (France) and Rapporteurs for the English and French versions of the report of the Session, respectively.

MATTERS OF INTEREST TO THE COMMITTEE (Agenda Item 4)

5. The Committee had before it Working Paper CX/MAS 91/2 containing a summary of matters of interest arising from the Eighteenth Session of the Codex Alimentarius Commission (CAC) and several Codex Committees.

a. Codex Alimentarius Commission

6. The Committee noted, with interest, plans to publish Volume 13 of the Revised Codex Alimentarius which would contain a compilation of methods of analysis and sampling recommended and endorsed by the CAC. The Volume would contain a list, by reference, of Codex General Methods followed by methods for use in specific applications. A third section was envisaged which would contain, in full, methods elaborated by Codex or which were not generally available. The Committee noted

that the first stage of preparing for this volume, namely the review and tabulating of all existing methods, had been finalized.

b. Codex Committees

7. The Committee was informed that the recent Twenty-First Session of the Codex Committee on Food Labelling (CCFL), held in March 1991, had discussed methods of analysis for determination of nutrients used to determine conformity with claims made in labelling. A Working Group had been studying this question for some time. The CCFL had decided that, before reinstating the Working Group, comments would be requested concerning this issue from the Codex Committee on Methods of Analysis and Sampling (CCMAS) and the Codex Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU). The Committee had also agreed that the Executive Committee would be advised of this procedure, with a view to providing advice.

8. The Delegation of Sweden drew attention to the need for work in this area to be undertaken by people who had sufficient experience and knowledge in methods of analysis for nutrients. The Delegation recommended that this work should initially be undertaken by CCNFSDU and that the methods proposed should be subsequently endorsed by CCMAS. The Delegation of Switzerland supported this proposal. The Committee noted that its general responsibilities in this area would be defined as a result of the CAC response to the outcome of the FAO/WHO Conference on Food Standards, Chemicals in Food and Food Trade.

c. FAO/WHO Conference on Food Standards, Chemicals in Food and Food Trade

9. The Committee was briefly informed on the principal results of the FAO/WHO Conference on Food Standards, Chemicals in Food and Food Trade, which had been held at FAO Headquarters, Rome, 18-27 March 1991. It was noted that the Conference had reviewed several aspects of the work of the CAC and had made a number of significant recommendations on the structures and procedures of the CAC. Principal among these was the strengthening of the work of the CAC's General Subject Committees so that matters of general importance, such as labelling, additives, contaminants and methods of analysis and sampling would be handled entirely by the General Subject Committee concerned. The Conference recommended that these Committees would be the main source of direction in these areas and would not be dependent on proposals or provisions put forward by commodity committees. The Committee noted that specific proposals were being formulated for the consideration by the CAC at its Nineteenth Session in July 1991, and it was hoped that the necessary amendments to the Codex Procedural Manual could be made by the Commission's 20th session in July 1993 on the basis of recommendation from the Codex Committee on General Principles.

10. The Delegation of the United Kingdom drew attention to recent developments in the control of foodstuffs in the Member Countries of the European Community (EC), specifically as a result of a recently published Directive in this area. The Directive contained *inter alia* requirements for the certification of laboratories on the basis of performance and proficiency. The Delegation pointed out that the area of laboratory accreditation could become an important area of future work for CCMAS as the attention of governments moved from the reliability of methods to the proficiency of laboratories, and that the preparation internationally recognized protocols for the evaluation of proficiency could be included in the Terms of Reference of this Committee. The Delegation of Australia stated that the CCMAS should consider the scientific aspect of laboratory analysis in relation to import/export control. The Delegation expressed its support for any moves for work on laboratory certification, proficiency programmes and guidelines on quality assurance in laboratories. This was seen as a natural extension of the work of the CCMAS. The Committee expressed its interest in combining its future work on methodology with the study of such laboratory systems.

d. Matters arising from other bodies (Agenda Item 4c)

11. The Representative of IUPAC presented the report of activities of interest to the Codex Committee on Methods of Analysis and Sampling. The Commissions related to the work of the CCMAS were those on Food Chemistry; Oils, Fats and Derivatives; Agrochemicals; and Microchemical Techniques and Trace Analysis. The Commission of Food Chemistry devoted its work to those areas of food chemistry considered high priority in terms of international interests and placed great emphasis on food safety. Nine working groups were operating in this Commission and most of them were involved in the development and validation of methods of analysis. The Representative provided detailed information of the work of each of these groups. In much of the work undertaken by IUPAC in this area there was close cooperation with other international organizations, such as AOAC, IARC, FAO/WHO, EC, and ISO.

12. The Committee was also informed briefly of the activities of the Comité Européen de la Normalisation (European Committee on Standardization, CEN) in the fields of additives, contaminants, and mycotoxins.

GUIDELINES ON SAMPLING AND REVIEW OF CODEX STANDARDS WITH REGARD TO SAMPLING IN THE LIGHT OF THE INSTRUCTIONS ON CODEX SAMPLING PROCEDURES
(Agenda Items 5 and 6)

13. In view of the complementary nature of the items on the agenda, the Committee decided to discuss them together. The papers available for discussion were: CX/MAS 91/3 and CX/MAS 91/4, prepared by the Delegation of the United Kingdom, and document CX/MAS 91/3 Addendum 1 prepared by the Codex Secretariat.

14. The Committee noted 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 provision in individual Codex Standards (ALINORM 89/40 paras. 339-341). A comprehensive background and outline for this purpose had been prepared by the Delegation of the United Kingdom and presented in the documents cited above. The document provided for a review of existing sampling provisions in Codex Standards by individual Commodity Committees in accordance with current Codex General Principles and Instructions, and that following this review, the sampling procedures would be incorporated into a consolidated document. The document also provided for additional guidelines on sampling, mainly administrative, and not covered by the main part of the review. On the other hand the Secretariat paper called for the development of the least number of general sampling methods for all foods, when possible based on the work of other organizations, and the consideration of recommended Codex sampling plans as advisory methods which might be used in international trade.

15. The Committee supported the simplified approach proposed by the Secretariat, and noted that it would be necessary to prioritize some areas for development of general sampling plans. Several delegations recommended flexibility in the application of sampling plans, and it was noted by one delegation that procedures for the control of foods and import or export should not in principle be different to the control at the national level.

16. The Committee established an *ad hoc* Working Group to undertake the task of prioritization and to provide the outline of a uniform sampling document along the lines mentioned above. The Working Group, consisting of delegates from Canada, Finland, France, Hungary, The Netherlands, Poland, Sweden, United Kingdom (Chairman) and the United States of America met during the session. The Working Group agreed that guidance was required to ensure that its tasks would be focused on developing a document that would be of practical use to all potential users, including industry and regulatory authorities. It was agreed that the Secretariat should provide guidance on particular areas of concern to the CAC. The Working Group also agreed to work actively by correspondence between this and the next session of CCMAS to prepare the necessary discussion document(s).

17. In relation to the proposed work of the Working Group, the Delegation of Hungary pointed out that several sampling plans would be needed for different purposes. In addition to sampling for the control of food safety criteria, the Delegation of Poland stated that special attention should be given to sampling for the control of conformity to label declarations. The Delegation of The Netherlands referred to the successful work accomplished in certain areas, such as that for the sampling of quick-frozen fish blocks and food-grade salt, which should not be overlooked in the development of the new sampling document.

SAMPLING FOR NET CONTENTS: Recommendation of the OIML (Agenda Item 7)

18. The Committee had before it document CX/MAS 91/5 and CX/MAS 91/5 Addendum 1 (Conference Room Document No.4), containing comments received from governments at Step 3, in response to Circular Letter 90/30-MAS, requesting comments on the OIML¹ Recommendation on Sampling Plans for Net Content in Packages. Comments had been sent to the rapporteur for this subject, Dr. Alvin P. Rainosek (USA), from Czechoslovakia, Denmark, Spain and the United States of America. Dr. Rainosek reported that the comments indicated that there was no support for the adoption by the Commission of a general method for net weight determination as outlined in the OIML Recommendation.

19. The Committee recalled that it had previously discussed this matter in relation to the requirements of the Codex General Standard for the Labelling of Prepackaged Foods, and that it had established criteria for such sampling plans (ALINORM 83/23 Appendix V). The OIML method, while meeting these general criteria, was only one of several appropriate methods. The Delegation of Spain drew attention to EEC Directives in this area, and the Delegation of Switzerland stated its opinion concerning the OIML recommendation was not yet made, but estimated that EEC Regulations could be preferred.

20. The Committee agreed that there was no need for further work in this area, and that the provisions contained in the Codex General Labelling Standard were adequately covered by available national and international practices.

ENDORSEMENT OF METHODS OF SAMPLING IN CODEX STANDARDS (Agenda Item 8)

21. The Committee had before it document CX/MAS 91/6 which contained proposed provisions concerning sampling elaborated by the Codex Coordinating for Africa, the Codex Committee on Food Additives and Contaminants, and the Codex Committee on Cereals, Pulses and Legumes.

22. The Committee endorsed the provisions contained in the following texts:

- African Regional Standard for Gari (CODEX STAN 151-1985)
- African Regional Standard for Whole and Decorticated Whole Millet Grain (CODEX STAN 169-1989)
- African Regional Standard for Pearl Millet Flour (CODEX STAN 170-1989)
- Draft African Regional Standard for Edible Cassava Flour (Step 8); and
- Draft Standard for Durum Wheat Semolina and Durum Wheat Flour (Step 8).

The Secretariat was requested to determine which of the ICC Methods quoted in the standards above was equivalent to ISO 2170:1980.

23. In regard to the proposal to use the sampling plans adopted for pesticide residues in food (CAC/PR 5-1984) for mercury, cadmium and lead, the Committee was informed that this method was temporarily endorsed by the Executive Committee which noted the need for practical scientifically-based recommendations for use in international trade. The Committee agreed to endorse these recommendations

¹ Organisation Internationale de la Metrologie Légale
(International Organization for Legal Metrology).

temporarily pending the outcome of its review of general sampling plans for contaminants.

REPORT OF THE INTER-AGENCY MEETING ON METHODS OF ANALYSIS AND SAMPLING
(Agenda Item 9)

24. The Committee was provided with a report of the Eighth Inter-Agency Meeting (IAM) on Methods of Analysis and Sampling, held at the premises of the Hungarian Office for Standardization (MSZH) on Friday, 5 April 1991. The report was introduced by Dr. K. Lingner (ISO). The meeting had been attended by representatives of nine organizations active in the field of food analysis and control, and was chaired by Mr. Castan (ISO/AFNOR). The report of the meeting is attached to the present report as Appendix IV.

25. The IAM had considered matters specifically of interest to CCMAS, such as:

- international collaboration in the area of standards for methods of analysis and sampling;
- the exchange of information on collaborative studies;
- the status of joint work of international organizations on 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;
- the question of proprietary laboratory techniques vis-à-vis traditional methodology;
- methods for the detection and quantitative determination of irradiated foods.

26. The Committee expressed its interest in the area of proprietary analytical techniques which, it was noted, were replacing traditional techniques in many laboratories for some specific analytes such as mycotoxins. It particularly noted the need for independently-derived information on the performance of these new techniques, especially for those agencies which intended to use them for enforcement purposes. The Committee welcomed the proposal that a paper coordinated by AOAC be prepared for consideration at its next session, and stated that any mechanism adopted should provide for the rapid evaluation of any new proprietary technique or of any modification to existing validated proprietary techniques.

27. The Representative of the AOAC noted that a list of methods adopted by that organization since 1988 had been submitted to the IAM, together with information on interlaboratory studies completed and in progress, as they related to topics required by the CAC.

28. The Committee expressed its appreciation to the organizations represented on the IAM for their constructive and collaborative approach to meeting the needs of the CAC in the area of methods of analysis and sampling.

PROGRESS REPORT ON THE DEVELOPMENT OF GUIDELINES FOR THE VALIDATION AND COLLABORATIVE TESTING OF METHODS OF ANALYSIS (Agenda Item 9a)

29. The Committee had before it the document CX/MAS 91/7 which contained the report of IUPAC published as result of an International Workshop held in Washington D.C., 17-18 April 1989 on "Harmonized Protocols for the Adoption of Standardized Analytical Methods and for the Presentation of their Performance Characteristics". It was noted that an earlier IUPAC protocol² had been prepared dealing with procedures for carrying out interlaboratory collaborative studies, in which details such as the number of participating laboratories and the number of samples and

² "Protocol for the design, conduct, and interpretation of collaborative studies" Horwitz, W., Pure Appl. Chem., 1988, 60 (6) 855-64

levels to be tested were provided. The Committee noted that it was largely at the instigation of CCMAS that IUPAC had undertaken this important work.

30. The Committee considered this report in the context of a possible revision of its recommendations for a checklist for information required to evaluate methods of analysis submitted to CCMAS for endorsement (Report of the 14th Session of the CCMAS, ALINORM 85/23, Appendix II). The IUPAC report had shown that information accumulated over more than 3000 collaborative studies could be used to assess method performance in terms of the relative standard deviation of the main performance characteristics of the method. The Grubbs test instead of the Dixon test for the detection of "outliers" had also been recommended in the earlier IUPAC report and recent recommendations of ISO (ISO 5725 which is being revised) had used 2.8 instead of 2.83 for the conversion of certain precision parameters. The Delegation of The Netherlands pointed out that a computer programme was available in that country for the evaluation of collaborative studies according to ISO 5725, and for calculation of the Grubbs test. It was also noted that although the first IUPAC report had been completely accepted by AOAC, the second protocol had not yet been fully discussed and adopted by that organization.

31. The Committee noted that a further IUPAC/ISO/AOAC Workshop was to be held on quality assurance procedures and performance characteristics of laboratories, which would continue the development of work in this area. Part of this development would be the recognition of the need for certified reference materials for use in quality assurance/proficiency trials. Nevertheless it considered it to be important for CCMAS procedures for the evaluation of methods submitted for endorsement to be revised taking into account the first two IUPAC Protocols. It was agreed that Governments and interested international organizations, especially those participating in the Inter-Agency Meeting, should be requested to comment on the current Checklist, attached as Appendix III to this report. The Delegation of the United States kindly offered to review these comments and to prepare a revised draft checklist for the Committee's consideration at its next session.

PROGRESS REPORT ON REVIEW OF STANDARD METHODS BY INTERNATIONAL ORGANIZATIONS (Agenda Item 9b)

32. Document CX/MAS 91/8 containing a progress report on the review of standard methods by national and international organizations, was prepared and presented by the Delegation of the United Kingdom. The document had initially been prepared for the AOAC and an up-dated version was being prepared, and it was hoped that comments would be available before the next meeting of CCMAS. The document was before the Committee primarily for information.

33. It was noted that the information contained in the document was not complete, as the extensive work of ISO in this area had been published and was available separately. Also there were other international organizations working in the field which had not yet been contacted. It was suggested that the information in the document, presented on an organization-by-organization basis could be better presented on an analyte-by-analyte basis, however the information currently available did not allow this in all cases.

34. The Committee looked forward to being kept informed of future progress, especially in regard to the work of specific organizations. The Delegation of the Netherlands drew attention to the work of CEN in this regard (see para. 12 above).

HARMONIZATION OF TERMINOLOGY USED IN SAMPLING AND ANALYSIS (Agenda Item 9c)

35. The Committee noted that a report will shortly be published by IUPAC on nomenclature used in collaborative studies, comprising *inter alia* concepts and definitions relating to methods performance, laboratory performance, and reference materials specifications.

LIMIT OF DETERMINATION (Agenda Item 9d)

36. The Committee based its discussion on a paper prepared by the Secretariat (Conference Room Document No.3) which contained, as an annex, a report prepared by IUPAC on metrological limits on the chemical measurement process as a response to a request for the Sixteenth Session of CCMAS. The paper addressed the question of the use of Detection Limit and Quantification Limit, and concluded that the selection of one or the other of these for monitoring contaminants in food at a preset maximum permitted level was a matter of degree and not of kind.

37. The Delegation of the USSR stated that the proposals did not completely solve all problems facing the CCMAS and did not take into account the practical situation facing analytical chemists in their laboratories when analyzing food for safety characteristics. The Delegation referred to articles published in the Journal of the AOAC (1989) which summarized the approaches taken in the USSR in this area. The Delegation expressed its appreciation of the assistance given by the Leader of the US Delegation in facilitating the editing of the articles.

38. The Delegations of Czechoslovakia and The Netherlands and the Representative of ISO drew attention to the work of ISO Technical Committee No. 69 (Sub-Committee 6, Working Group 5 - *Measurement of Methods and Results: Limits of Determination*). It was noted that both IUPAC and AOAC were in liaison with this body. Noting that questions relating to the limits of detection or quantification were general for all analytical chemistry, and not only food chemistry, the Committee agreed to await further advice from ISO and IUPAC in this matter. It was noted, however, that an understanding of all relevant performance characteristics was essential for the evaluation of methods used for the determination of very low concentrations of substances in foods for conformity with specifications, including precision criteria on matrix blanks.

39. The Committee also agreed to forward the comments received on Conference Room Document 3 (the IUPAC paper) to IUPAC for further consideration and incorporation in to a revised document which it hoped to have available for review at its next session.

CLASSIFICATION OF THE CODEX METHODS OF ANALYSIS (Agenda Item 10)

40. The Committee considered the document CX/MAS 91/9 prepared by the Secretariat on the basis of discussions at the Sixteenth Session of CCMAS and government comments obtained in response to Circular Letter 1989/3-MAS. Comments had been received only from USSR at the time of preparation of the paper. The paper proposed a simplification of the classification system to provide for improved recognition of alternative methods which met the general criteria of CCMAS for control, inspection or regulatory methods. This would facilitate the selection and use of recommended Codex methods in industrialized and developing countries.

41. The Committee was generally in favour of simplifying the procedures for evaluating Codex methods, but noted that the classification system provided for a correct separation of Type I "Defining" methods from all other methods, and for establishment of "Reference Methods" to be used in Case of dispute. In particular, it noted that Alternative or Type III methods were not necessarily inferior in their performance characteristics to Type II methods and could therefore be used with confidence. Type III methods were optional, fully validated methods. When other performance characteristics were equal, Type II methods were selected on the basis of practicality.

42. The Delegations of Switzerland and the USSR recommended that methods should be published together with their metrological characteristics so that users would be able to assess the evaluation and the use of the method. The Committee noted however that the selection of "Type" depended also on the nature of the provision it was being used to measure, and on other non-metrological characteristics such as simplicity or ease of use when all other criteria were met.

43. The Committee noted that the definition of Type I Method in the Procedural Manual (7th Edition, 1989, page 143) was incorrect as these methods could not be used "for calibration purposes". It recommended to the Commission that the definition should be amended by deleting the final phrase "and which can serve for calibration purposes" and substituting it with the phrase "and serves by definition as the only method for establishing the accepted value of the item measured".

SIMPLE METHODS OF ANALYSIS (Agenda Item 11)

44. The Committee had before it a working paper prepared on this subject by the Secretariat (CX/MAS 91/10). The paper was based on discussions which took place in CCMAS on previous occasions and by several Codex Regional Coordinating Committees on the need for the development of "simple" methods of analysis for use in developing countries. In general, the Codex Regional Coordinating Committees had not supported the separate elaboration of simple methods for food control.

45. The Committee, taking into account its discussions on the previous item on its agenda, did not agree to amend the classification system for Codex methods of analysis, as was proposed in the paper. However, it confirmed that all methods which corresponded to defined Codex criteria were considered as suitable methods for Codex purposes.

46. The Committee supported the view that greater attention should be given to the elaboration and selection of general methods of analysis, multi-analyte methods for trace analysis, and ELISA methods, which in practical terms meant that expenses related to specialized equipment and reagents would be minimized. It also meant that staff could be trained more effectively in the analysis of many more products. Noting that the concept of "simple" in relation to methods was not easily defined and could be interpreted in different ways even when all metrological characteristics were available, the Committee agreed to continue to select Codex methods on the basis of its established criteria.

PROCEDURE FOR THE REVIEW OF METHODS OF ANALYSIS IN CODEX STANDARDS AND ENDORSEMENT OF CODEX STANDARDS (Agenda Items 12 and 13)

47. The Committee had before it the Conference Room Document 2 containing a Report of a meeting of the Working Group on Endorsements which met on 6 April 1991. The following Member countries and international organizations were represented:

Canada	AOAC
Finland	OIV
Hungary	IFG
Netherlands	ISO
Norway	
United Kingdom	
United States of America	

48. The Working Group, under the chairmanship of Dr. W. Horwitz (USA) had the following tasks to perform:

- (a) to consider endorsement of methods of analysis in Codex Standards, listed in parts A and B of document CX/MAS 91/11.
- (b) to review and consider for endorsement the methods of analysis for aflatoxins, selected by the Codex Committee on Food Additives and Contaminants and listed in Conference Room Document 5.

49. The Committee discussed the working papers and the report of the Working Group. The following remarks were made during the discussions:

- (a) In regard to the provision for Sodium Chloride in Bouillons and Consommés (CODEX STAN 117-1981), the Delegation of The Netherlands pointed out that the Codex General Method measured only Chloride and

that this should be so indicated. This position was supported by Finland, which pointed out that much more attention was being paid to the sodium content of foods in relation to nutrition labelling and claims. If the Codex General Method were used for the declaration of salt, the result could be misleading in cases where salt substitutes had been used. The Committee agreed that the method should be described as "Chloride calculated as Sodium Chloride".

- (b) In relation to the determination of Essential Oils in fruit juices, the Committee was informed that the IFJU Method No.45A (Steam Distillation Method) was valid for all citrus juices as a Type I Method, and that the alternative method proposed (IFJU Method No. 45B (Direct titration with bromate) was a simple routine method valid only for orange juices. The Committee endorsed only the first method.

Methods of Analysis for Aflatoxins

50. The Working Group had also considered a list of methods for aflatoxin determination prepared by the Codex Committee on Food Additives and Contaminants (CCFAC) and included in Conference Room Document No. 5. The Working Group felt unable to endorse the methods, noting that in some cases the limits under consideration by CCFAC were at or below the reliable limit of determination of the methods proposed. The Working Group had suggested that a list of general criteria for evaluating acceptable methods for determining aflatoxins should be prepared, together with an up-dated reference list of methods available, for consideration at the next session of CCMAS. The Delegation of the United Kingdom kindly offered to prepare a draft list of criteria and the Representative of the AOAC to prepare the list of methods.

51. 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 II to this report. The Committee expressed its appreciation to Dr. Horwitz, and to the Members of the Working Group.

REVIEW OF METHODS FOR CONTAMINANTS IN CODEX STANDARDS (Agenda Item 14)

a) General Methods

52. The Committee considered the document CX/MAS 91/12 and CX/MAS 91/12 Add. 1 (Conference Room Document 6) which contained an up-dated list of Codex General Methods for Contaminants and comments from Canada, Cuba, Czechoslovakia, Denmark, Finland, Sweden and the USA in response to Circular Letter 1989/3-MAS. The comments received generally supported the need to replace the existing methods which were generally recognized as out of date. Particular attention was drawn to the present use of multi-element methods by most up-to-date laboratories. Several delegations also drew attention to problems of laboratory safety in the use of these old methods, and the Delegation of Finland provided information on a multi-element method recently adopted by NMKL (No. 139 1991).

53. The Delegation of Canada agreed to revise the list of methods on the basis of comments received and in the light of other available published information such as in the EC Decision 90/515/EEC. This list would be made available to governments and interested international organizations for comments, and a more complete and current list would be presented to the Committee for discussion at its next session.

b) General Method of Ashing for the Determination of Heavy Metal Contaminants

54. The Committee considered the document CX/MAS 91/13 which contained government comments on a general mineralization proposed by the Delegation of the USSR at the Sixteenth Session of CCMAS, and later published in the Journal of the Association of Analytical Chemists (Vol. 72, No. 2, 1989). Comments were received from Canada,

Cuba, Czechoslovakia, Denmark, Finland, Sweden, Switzerland, USSR, and USA. The comments supported in general the adoption of the proposed method, but to specify and coordinate certain details it was recommended to organize a review of the method on the basis of comments received. The Delegation of the USSR kindly agreed to undertake this review and provide a revised version of the method for circulation for comments and discussion by the Committee at its next session. The Delegation of the USSR expressed the hope that the revised method would be published in the Journal of the AOAC.

c) **Determination of Copper in Foods**

55. The Committee considered a summary of comments presented in document CX/MAS 91/14 on the proposed AOAC Colorimetric Method (AOAC 960.40) on which comments had previously been requested at Step 3. Several delegations were of the opinion that the proposed method was old and out-dated and was not suitable for any commodity for which limits for copper were established at the order of 2.5 mg/kg or less. It was noted that the General Methods for Contaminants under consideration by the Committee included multi-element methods which were suitable for the analysis of copper. The Committee noted that the method had been validated collaboratively and decided to recommend adoption as a Type III Codex Method, noting the limitations of the method as mentioned above.

METHODS OF ANALYSIS FOR CODEX STANDARDS WHICH NEED TO BE DEVELOPED AND/OR VALIDATED
(Agenda Item 15)

56. The Committee discussed a list of methods of analysis required for the completion of Codex Standards as presented in Working Paper CX/MAS 91/15. The document had been prepared as part of the overall review of Codex Methods of Analysis, and highlighted those provisions in Codex Standards for which methods had never been identified or for which the identified method was known not to be validated. The document was primarily for the information of the Committee and to provide the basis of requests for work on method development and validation by the relevant organizations. The Committee was informed that this document had been provided to the Inter-Agency Meeting held in Budapest, 5 April 1991. The Representative of the AOAC and ISO stated that the list would be considered together with other "methodology needs" by these organizations.

57. The representative of IFG informed the Committee that this organization would undertake work to provide suitable methods for heavy metal determination in wheat gluten and gliadin in gluten-free foods. Several delegations also expressed interest in up-dating the existing review of available methods for determination of food additives in foods carried out by the Codex Committee on Food Additives and Contaminants.

58. The Committee noted that the recommendations of the FAO/WHO Conference on Food Standards, Chemicals in Food and Food Trade, if adopted by the CAC, would lead to a greater responsibility being delegated to the CCMAS in the identification and endorsement of a wider variety of methods for all foods, depending on the needs of member governments.

FUTURE WORK (Agenda Item 16)

59. The Committee noted that its future work would include the following:

- endorsement of methods of analysis and sampling
- elaboration of simplified guidelines on sampling
- consideration of procedures for endorsement of proprietary analytical techniques
- review of general methods for determination of metallic contaminants

- identification of methods of analysis required by Codex
- preparation of Codex guidelines for laboratory quality assurance programmes and proficiency testing
- limit of determination based on IUPAC and ISO follow-up.

OTHER BUSINESS (Agenda Item 17)

60. The Committee had before it a Working Paper (Conference Room Document No.7) prepared by The Netherlands, concerning the use of analytical quality criteria in food safety control. The subject had been briefly discussed at the Committee's previous session (ALINORM 89/23 para. 79-80). The Committee noted that it had requested to be kept informed of progress in this area.

61. The Delegation of The Netherlands noted that the paper referred to the use of highly sophisticated methods of analysis used for the determination of trace amounts of analytes which might be of food safety significance. In particular the paper provided a model to limit the number of false positive and false negative results which occur when the concentration of the analyte is near the limit of determination of the method. The Delegation also noted that this approach was useful for crisis situations in which collaboratively studied and validated methods were not available. The approach outlined in the paper had been published (JAOAC 72 1989, pp 487-490 and J. Anal. Chem. 330 1990, pp 370-377). The concept had been incorporated in a number of relevant EEC Decisions.

62. The Committee noted that these criteria had been taken up by the Codex Committee on Residues of Veterinary Drugs in Foods, and suggested that they might also be considered by the Codex Committee on Pesticide Residues. The Committee was also of the opinion that these criteria could be taken into account when establishing criteria for evaluating methods for contaminants.

DATE AND PLACE OF NEXT SESSION (Agenda Item 18)

63. The Committee was informed that its Eighteenth Session would be held in Budapest, most probably in November 1992.

SUMMARY STATUS OF WORK

Subject	Action to be taken by:	Document Reference (ALINORM 91/23)
Guidelines on Sampling	Working Group Codex Secretariat	paras. 13-17
Proprietary Analytical Techniques	AOAC	paras. 24-28
Checklist for information required to evaluate methods of analysis submitted to CCMAS for endorsement	Governments USA Codex Secretariat IAM	paras. 29-31
Limit of determination	Codex Secretariat IUPAC ISO	paras. 36-39
Amendment of the Definition of Type I Method (Procedural Manual, 7th Edition 1989, page 143)	CAC	para. 43
List of General Criteria for evaluating acceptable methods for determining aflatoxins	UK	para. 50
Review of General Methods for Contaminants	Canada Codex Secretariat Governments	paras. 52-53
Review of General Method of Ashing for the Determination of Heavy Metal Contaminants	USSR Codex Secretariat Governments	para. 54
Methods of Analysis for Codex standards which need to be developed and/or validated	IAM Codex Secretariat Governments	paras. 56-58
Preparation of Codex Guidelines for laboratory quality assurance programmes and proficiency testing	Codex Secretariat	para. 59

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LIST OF METHODS CONSIDERED BY THE COMMITTEE FOR ENDORSEMENT

COMMODITY	PROVISION	METHOD	TYPE & STATUS
Gari CODEX STAN 151-1985	Crude fibre	ISO 5498:1981 (B.5 Separation)	I E
Whole and Decorticated Pearl Millet Grain CODEX STAN 169-1989	Moisture	ICC Method No. 109/1 (1986)	NE ¹
	Ash	ISO 2171:1980	I TE ²
	Crude fibre	ISO 5498:1981 (B.5 Separation)	I E
	Protein	AOAC 920.87	I E ³
Pearl Millet Flour CODEX STAN 170-1989	Crude fat	AOAC 945.38F AOAC 920.39C	I E
	Moisture	ICC Method No. 109/1 (1986)	NE ⁴
	Ash	ISO 2171:1980	I TE ⁵
	Crude fibre	ISO 5498:1981 (B.5 Separation)	I E
	Protein	AOAC 920.87	I E ⁶
	Crude fat	AOAC 945.38F AOAC 920.39C	I E
	Colour	Modern Cereal Chemistry, 6th Ed., D.W. Kent Jones & A.J. Amos, pp 605- 612, Food Trade Press Ltd., London, 1969.	I NE ⁷
Vinegar CODEX STAN 162-1987	Residual alcohol	AOAC 942.06 OIV Method A2 (1978)	II E III E
	Sulphur dioxide	OIV Method A-17 (1990)	III NE ⁸
	Iron	IFJU Method No. 15 (1964)	II NE ⁹
Natural Mineral Waters CODEX STAN 108-1981	Total dissolved solids	Method described in the standard	I TE ¹⁰

Total organic matter	Handbuch der Lebensmittelchemie VIII/1 p.610	I TE ¹¹
Arsenic	AOAC 986.15 ISO 6595:1982	II E III NE ¹²
Barium	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 65-66	II TE ¹³
	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 67-68	III TE ¹³
Borate	Handbuch Spuranal., 1974	NE ¹⁴
Cadmium	ISO 8288:1986 AOAC 986.15 AOAC 974.27	II E III E III E ¹⁵
Chromium (VI)	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 86-87	II TE ¹⁶
Copper	AOAC 960.40	II TE ¹⁷
Fluoride	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 245-247	II E
	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 247-250	III E
Mercury	ISO 5666-3:1984 AOAC 977.22	II E III E
Manganese	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 121-122	II E
	ISO 6333:1986	III E

Nitrates	ISO 7890-2:1986	II TE ¹⁸
	Handbuch Lebensmittel Chemie (1969)	III TE
	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 280-283	III TE
Lead	ISO 8288:1986	II E
	AOAC 974.27	III E
Selenium	AOAC 986.15	II E
	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 320-322	III E
Sulphide	Handb. Spurenanal. 1974	II TE ¹⁹
Hydrogen carbonate (Bicarbonate HCO ₃ ⁻)	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 170-175	I TE ²⁰
Chloride	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 202-205	II E
	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 205-208	III E
	ISO 9297:1989 AOAC 973.51	III E ²¹ III E ²¹
Sulphates	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 331-333	II E
	ISO 9280:1990	III E ²²

Sodium	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 148-151	II E
	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 151-152	III E
Potassium	Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol. 2, pp. 142-145	II E
Calcium	ISO 6058:1984	II E
	ISO 7980:1986	III E
Magnesium	ISO 6059:1984	II E
	ISO 7980:1986	III E
Dissolved iron	ISO 6332:1988	II E
Total cyanide	ISO 6703-1:1984	II TE ²³
Nitrites	ISO 6777:1984	II TE ²⁴
Phenols	ISO 6439:1990	I E
Surface active agents	ISO 7875-1:1984	I E ²⁵
Vegetable protein products CODEX STAN 174-1989	Crude fibre	AACC (1982) 32-10 I E
Soy protein products CODEX STAN 175-1989	Crude fibre	ISO 5498:1981 I E/NE ²⁶
Quick frozen fish sticks (Fish fingers) and Fish portions, Breaded or in Batter CODEX STAN 166-1989	Fish core (Breeding)	AOAC 971.13 I E
	Parasites	Method described in the standard I E ²⁷
Fruit Juices	Carbon dioxide	IFJU Method No.42, 1976 II TE ²⁸
	Lead	ISO 6633:1984 II NE ²⁹
	Ethanol	IFJU Method No.53, 1983 II NE ³⁰

Foods for Special Dietary Uses	Crude fibre	AOAC 985.29	I	TE ³¹
	Loss on Drying	AOAC 934.01	I	TE ³²
	Linoleate	AOAC 922.06, 969.33, 963.22	II	E ³³
	Sodium & Potassium	AOAC 984.27	III	E
Processed Tomato Concentrates CODEX STAN 57-1981	Mineral impurities	AOAC 971.33	I	TE ³⁴
Table Olives CODEX STAN 66-1981 (Rev.1 1987)	Acidity of brine	Method described in the standard	I	TE ³⁵
	pH of brine	Method described in the standard	I	TE ³⁶
Bouillons and Consommés CODEX STAN 117-1981	Sodium chloride	Codex General Method (AOAC 971.27)	II	E ³⁷
		AIIBP Method No. 2/4	III	TE
Cocoa Butter CODEX STAN 86-1981	Copper	IUPAC Method Pure and Appl. Chem., <u>60</u> p.893 (1988)	II	TE ³⁸
	Lead	IUPAC Method in press	II	TE ³⁹
	Iron	IUPAC Method Pure and Appl. Chem., <u>60</u> p.893 (1988)	II	TE ⁴⁰
Chocolate CODEX STAN 87-1981	Lead	AOAC 986.15(E)	II	TE ⁴¹
Cocoa Powders (Cocoa) and Dry Cocoa-Sugar Mixtures CODEX STAN 105-1981	Cocoa butter	OICCC 8a (1972), 14 (1973), 16 (1973)	I	E ⁴²
	Lead	AOAC 986.15(E)	II	TE ⁴¹
Fats and Oils	Copper	IUPAC Method Pure and Appl. Chem., <u>60</u> p.893 (1988)	II	TE ⁴³
	Lead	IUPAC Method in press	II	TE ⁴⁴
Specified Vegetable Fat Products CODEX STAN 157-1987	Slip point	AOCS Cc 3-25	I	TE ⁴⁵
	Milk fat	Pure and Appl. Chem. <u>58</u> (1986); 10, 1419-1428	I	E ⁴⁶

Specified Animal or Mixed Animal and Vegetable Fat Products CODEX STAN 158-1987	Slip point	AOCS Cc 3-25	I	TE ⁴⁵
	Milk fat	Pure and Appl. Chem. 58 (1986); 10, 1419-1428	I	E ⁴⁶
Canned Mangoes CODEX STAN 159-1987	Syrup measurement	AOAC 932.14(c)	I	E
Mango Chutney CODEX STAN 160-1987	Total soluble solids	AOAC 932.14(c)	I	E
Sorghum Flour CODEX STAN 173-1989	Colour	Modern Cereal Chemistry, 6th Ed., D.W. Kent Jones & A.J. Amos, pp 605- 612, Food Trade Press Ltd., London, 1969.	I	NE ⁴⁷
Edible Low-Erucic Acid Rapeseed Oil CODEX STAN 123-1981	Erucic acid content	IUPAC 7th Ed., 1984 2.311	II	TE ⁴⁸
Cocoa Butter Confectionary CODEX STAN 147-1985	Sugars	Method to be developed		49

Draft Standards submitted for endorsement

Edible Cassava Flour (Step 8)	Granularity	ISO 2591-1:1988	I	E
	Moisture	ISO 712:1985	I	E
	Ash	ISO 2171:1980	I	TE ²
	Crude fibre	ISO 5498:1981 (B.5 separation)	I	E
Corned Beef (Step 8)	Lead	AOAC 934.07	II	E ⁵⁰
	Tin	AOAC 985.16	II	E
Luncheon Meat (Step 8)	Lead	AOAC 934.07	II	E ⁵⁰
	Tin	AOAC 985.16	II	E
Cooked Cured Ham (Step 8)	Lead	AOAC 934.07	II	E ⁵⁰
	Tin	AOAC 985.16	II	E
Cooked Cured Pork Shoulder (Step 8)	Lead	AOAC 934.07	II	E ⁵⁰
	Tin	AOAC 985.16	II	E

Cooked Cured Chopped Meat (Step 8)	Lead	AOAC 934.07	II	E ⁵⁰
	Tin	AOAC 985.16	II	E
Fruit Juices (Citrus Fruit Juices)	Essential oils	IFJU Method No. 45A (1972)	I	E

NOTES ON THE TABLE

1. The Working Group considered that the method was impractical for enforcement purposes because of extremely long drying times and recommended the use of a simpler method such as ISO 712 (equivalent to ICC 110/1).
2. The Working Group temporarily endorsed the lower temperature version (550°) of the ISO method which is equivalent to AOAC 923.03 and recommended that the Codex Committee for Cereals, Pulses and Legumes be requested to clarify which version of the ISO method should be used for millet. 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°) was included in the new text.
3. The Working Group requested the Commodity Committee to indicate the appropriate conversion factor for millet and its products.
4. The Working Group considered that the method was impractical for endorsement purposes because of extremely long drying times and recommended the use of a simpler more reliable method such as ISO 712 (equivalent to ICC 110/1).
5. The Working Group temporarily endorsed the lower temperature version (550°) of the ISO method which is equivalent to AOAC 923.03 and recommended that the Codex Committee for Cereals, Pulses and Legumes be requested to clarify which version of the ISO method should be used for millet. 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°) was included in the new text.
6. The Working Group requested the Commodity Committee to indicate the appropriate conversion factor for millet and its products.
7. The Working Group recommended that questions concerning the applicability of the method to this product should be referred to ICC.
8. The Working Group recommended that the Coordinating Committee for Europe consider AOAC Methods 990.28, 990.29, 990.31, from the First Supplement (1990) to the Fifteenth Edition, as these were less likely to be subject to interferences.
9. Status retained.
10. Retained as TE until results of collaborative study are provided.
11. The Working Group recommends that the Commodity Committee consider adopting ISO 8467:1986 or AOAC 973.47 methods which are collaboratively studied, and specify which of the available procedures contained in these methods are appropriate as Type I.
12. This method was not endorsed because of concerns about its applicability at the maximum permitted level of 0.05 mg/L.

13. The Secretariat is requested to obtain results of any collaborative studies of this method so as to finalize endorsement.
14. The Working Group recommended adopting a more modern collaboratively studied method such as ISO 9390:1990.
15. The Working Group recommended that ISO 5961:1985, developed for water, could be considered as another Type III method.
16. The method was temporarily endorsed, but the Working Group recommended that a more modern collaboratively studied general method such as ISO 9174:1990 or AOAC 947.27 should be considered.
17. The Working Group temporarily endorsed this method but recommended that a more generally applicable method designed for water analysis such as ISO 8288:1986 be considered as a Type II method and recommended that the AOAC method should be classified as Type III.
18. The Working Group temporarily endorsed these methods with the recommendation that the Commodity Committee use the collaboratively-studied method ISO 7890-2 as the Type II reference method, and provide data on collaborative studies conducted with the other methods for nitrates.
19. The Working Group temporarily endorsed the method with the request that collaborative study data be provided by the Commodity Committee.
20. The Working Group temporarily endorsed the method as Type I with the request that the Commodity Committee provide collaborative study data, or consider a collaboratively studied method such as AOAC 920.194.
21. ISO 9297:1989 and AOAC 973.51 were recommended as additional Type III methods.
22. ISO 9280:1990 was endorsed as an additional Type III method. The secretariat was asked to determine whether the two endorsed methods were equivalent.
23. The Working Group temporarily endorsed the method but requested data on the applicability at the level specified in the Codex Standard from the Commodity Committee or ISO.
24. Temporarily endorsed pending information from the Commodity Committee on reliability parameters of the method and applicability at the level specified in the Codex Standard.
25. The Working Group noted that Part 1 of ISO 7875:1984 referred to anionic surfactants only, and asked whether this was the intention of the Commodity Committee.
26. Endorsed only for application with Soy Protein Flours and Concentrates. Not endorsed for use with Soy Protein Isolates as available studies suggest that results are unreliable in when the amount of fibre to be determined from a test portion is less than 50 mg. The Working Group questioned whether a method was required for Soy Protein Isolates in commerce.
27. The Working Group suggested that the Secretariat compare the cited method for equivalency with AOAC 985.12 and the citation in the standard be clarified to indicate that the examination of the product should be conducted after removal of the breadding or batter.
28. The Working Group requested that the results of collaborative studies be provided.
29. No collaborative study data was provided. The General Method for Lead in Foods or AOAC 934.07 should be considered by the Committee.

30. The Working Group requested that the results of collaborative studies be provided. Alternative methods such as AOAC 942.06 or ISO 2448 could be considered if the IFJU method has not been collaboratively studied.
31. The method was temporarily endorsed and the Commodity Committee was requested to verify the application of the method which applies to (total) dietary fibre, not crude fibre.
32. The method is not applicable to high fructose products.
33. The IUPAC citation for this method to be added.
34. Temporary endorsement continued awaiting information concerning the basis for selection of the method.
35. The Working Group requested that the results of collaborative studies be provided.
36. The Working Group requested that the results of collaborative studies be provided.
37. The method determines chloride calculated as sodium chloride.
38. The Working Group temporarily endorsed the graphite furnace method published in *Pure and Applied Chemistry* Vol. 60, No.6, p.893, 1988 to be substituted in the standard for the method originally recommended by the Commodity Committee, unless the Commodity Committee advises the Commission to the contrary.
39. The Working Group temporarily endorsed the method to be published in *Pure and Applied Chemistry* to be substituted in the standard for the method originally recommended by the Commodity Committee, unless the Commodity Committee advises the Commission to the contrary.
40. The Working Group temporarily endorsed the graphite furnace method published in *Pure and Applied Chemistry* Vol. 60, No.6, p.893, 1988 to be substituted in the standard for the method originally recommended by the Commodity Committee, unless the Commodity Committee advises the Commission to the contrary.
41. The Working Group temporarily endorsed a substitute method and recommended that this should be permanently endorsed unless the Commission is advised to the contrary.
42. The Commodity Committee was advised that more modern methods are available for consideration.
43. The Working Group temporarily endorsed the graphite furnace method published in *Pure and Applied Chemistry* Vol. 60, No.6, p.893, 1988 to be substituted in the standard for the method originally recommended by the Commodity Committee, unless the Commodity Committee advises the Commission to the contrary.
44. The Working Group temporarily endorsed the method to be published in *Pure and Applied Chemistry* to be substituted in the standard for the method originally recommended by the Commodity Committee, unless the Commodity Committee advises the Commission to the contrary.
45. A substitute method from ISO (DIS 6321) will become available, and the Committee should consider replacement with this collaboratively studied method.
46. The Codex Committee on Fats and Oils should be requested to provide conversion factors from butyric acid to milk fat, which are not provided in the method.

47. The Working Group recommended that questions concerning the applicability of the method to this product should be referred to ICC.
48. The Committee on Fats and Oils was requested to provide reference to collaboratively studied data and compare ISO 8209:1986 for equivalency with this IUPAC method.
49. The Working Group noted that the method proposed by the Commodity Committee (OICC 7a to 7c 1960) did not exist, and noted that an appropriate collaboratively studied method needed to be developed.
50. The Committee suggested that consideration be given to the use of the Codex General Method for the determination of lead in this commodity.

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 BY THE CODEX
COMMODITY COMMITTEES TO THE CCMAS FOR CONSIDERATION
OF METHODS OF ANALYSIS FOR ENDORSEMENT

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 chemical component, constituent, or property 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 Determination (if needed)

To be defined.

(c) Classification (Type)

The method classification as defined in the Codex Alimentarius Commission Procedural Manual, Seventh Edition, pp. 143-144:

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 literature citation, indicating where the method will be found in the scientific or technical literature or in a Codex document. 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 AND RESULTS OF COLLABORATIVE STUDY SUPPORTING THE ENDORSEMENT OF THE METHOD

1.2.1 Bibliographic Reference to Collaborative Study

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. Sufficient documentation must be given so that a librarian can obtain the referenced document directly from the journal, by interlibrary loan, or by a request to the organization responsible for its production.

1.2.2 Design

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

The materials may be identified 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 and the outlier tests used. The commonly used tests for rejection of outliers are the Dixon (D) tests for extreme values, 3.1, 3.2 and the Cochran (C) test for extreme variance, 3.3, 3.4. If no outliers were rejected, report 0. Ordinarily, rejecting more than 20% of the data is considered excessive. Indicate identification number(s) of laboratories removed in order to detect a consistent systematic bias on the part of any of them. Also indicate CL used in outlier tests for rejection of data, if other than 99%. Both AOAC and ISO currently reject outliers at a probability level greater than 99%. In the example, outliers were rejected at the 95% CL.

1.2.5 Concentration of Analyte

If known or assumed. If it is the 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

(a) Repeatability (within-laboratory)

Standard deviation, in the same units as the average
Relative standard deviation (repeatability standard deviation
x 100/average found)
Interval (2.83 x repeatability standard deviation)

(b) Reproducibility (between-laboratory, including within-)

Standard deviation, in the same units as the average
Relative standard deviation (reproducibility standard deviation
x 100/average found)
Interval (2.83 x reproducibility standard deviation)

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 RSD between-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, 3.5 or ISO 5725, 3.2, section 7.2 (b).

The repeatability and reproducibility intervals indicate how well an analyst should check himself (r) and another analyst in another laboratory (R), both in absolute units, with 95% probability. These parameters, however, are usually concentration dependent, so if the expected concentration varies over a wide range in the study, the parameters must be associated with the corresponding average concentration, material by material, 3.2, section 3.

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

REPORT OF THE EIGHTH INTER-AGENCY MEETING (IAM) Budapest, 5 April 1991

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OPENING OF THE MEETING

1. The Meeting was opened by Mr Petrovai, President of the Hungarian Office for Standardization (MSZH). After extending a warm welcome to the Representatives of the various International Organizations (See Annex), Mr Petrovai emphasized the importance of the IAM for the work of CAC. He also mentioned that Hungary as a food exporting country is particularly interested in the food standardization work undertaken by CAC, ISO/TC 34 "Agricultural Food Products" and other Organizations specialized in this area.

ELECTION OF CHAIRMAN

2. Upon the proposal of Mr Petrovai, Mr G. Castan (AFNOR) was elected Chairman. Mr Petrovai recalled the important contributions Mr Castan had made over the past forty years to national and international standardization work in the area of food products. On behalf of the Hungarian Office for Standardization (MSZH) and the Hungarian food industry, Mr Petrovai thanked Mr Castan for all he had done for international cooperation and presented him with the MSZH medal for outstanding contributions to standardization.

3. Mr Castan thanked Mr Petrovai, MSZH and the members of the IAM for the confidence. He then gave credit to the joint efforts of the international team that had very much contributed to the success of the IAM.

ADOPTION OF THE AGENDA

4. The agenda was adopted without amendment. Following a proposal by the Representative of IAEA it was agreed to consider the topic of food irradiation under the item "Other activities of interest to the IAM".

REVIEW OF MEMBERSHIP OF THE IAM

5. The IAM noted that the European Committee for Standardization (CEN) had recently established a Technical Committee CEN/TC 275 "Food analysis - Horizontal aspects" the Secretariat of which had been assigned to the German Standards Institute (DIN). The Secretary of IAM had already contacted the CEN Central Secretariat and the Secretariat of CEN/TC 275 with a view to establishing close cooperation with the IAM.

6. After discussing the activities, structure, composition and working procedures of CEN, the IAM decided to include CEN in the list of Organizations to be invited to the next IAM. It also agreed that there was a need for close cooperation between CEN/TC 275 and ISO, AOAC, CAC, IDF and other Organizations in order to avoid the risk of duplication of work in the field of methods of analysis and sampling.

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

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

- The report of the Seventh IAM had been circulated as Appendix VII of the report of the Sixteenth Session of CCMAS (document ALINORM 89/23);
- A computerized list of the more than 400 existing Codex methods had been prepared. Volume 13 of the Codex will shortly be published with references to all these Codex methods;
- The CCMAS Working Group on the endorsement of methods will be meeting on 6 April 1991;
- The topic of "limit of determination" and the Guidelines for the validation of Collaborative testing of Methods of Analysis had been included in the agenda of CCMAS
- A list of methods required by the CCMAS (document CX/MAS 91/15) had been prepared.

8. The Secretary introduced the "Selective list of ISO Standards on Food Technology" which contains the titles and reference numbers of all relevant ISO

Standards and drafts and suggested that this may be used for the updating of information on relevant ISO Standards in Codex documents.

REPORT ON EXCHANGE OF INFORMATION ON COLLABORATIVE STUDIES

9. The Representative of AOAC informed the IAM that she had received information on planned and current collaborative studies from several Organizations. She noted, however, that some Organizations had not yet responded to the request of IAM for the exchange of information in the formal agreed upon earlier. Therefore, copies of the relevant form were distributed to all participants.

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

10. The IAM noted that the harmonized Protocol which had been published by IUPAC had also been accepted by AOAC and NMKL. IDF had prepared an identical Protocol and ICUMSA was using the original IUPAC Protocol. Other Organizations, including ISO, were encouraged to adopt and use the Protocol. In this context reference was made to the International Symposium on Harmonization to be held at the ISO Central Secretariat in Geneva, on 2 and 3 May 1991. All interested Organizations had been invited to attend this Symposium.

11. The Representative of ISO mentioned that his Organization was also exploring various possibilities of publishing and adopting the Protocol, i.e. in the form of an ISO Guide or as an extension of relevant Standards prepared by ISO/TC 47 "Chemistry" or ISO/TC 69 "Applications of statistical methods". This matter is also under consideration by the Standing Editing Committee (SEDCO) of ISO/TC 34 which is the body responsible for the uniform drafting of the ISO standards in the area of food products.

METHODS OF ANALYSIS AND SAMPLING REQUIRED BY THE CAC

12. The Chairman invited the Representatives of the various specialized Organizations to report on the progress achieved with respect to methods required by the CAC. In this context, reference was also made to document CX/MAS 91/15 which contains a list of methods to be developed and/or Validated.

Methods of Sampling

13. The IAM noted that the general outline on sampling prepared by CCMAS does not include aspects of sampling techniques. The Representative of the Codex Secretariat drew attention to the maximum limits for contaminants in food packaging materials as laid down by the Codex Committee on Food Additives and Contaminants. These maximum limits will require the establishment of appropriate sampling Plans.

Pesticide Residues

14. The Representative of the Codex Secretariat informed the IAM that general Codex methods and Guides exist on the sampling and analysis for pesticide residues and that no specific request had therefore been made to CCMAS. He mentioned, however, that screening methods and multiple-residue detection methods would be required in future. The Representative of AOAC drew attention to the 15th edition of the AOAC methods which uses a new numbering system that facilitates updating of References.

Residues of Veterinary Drugs

15. The IAM noted the relevant activities of AOAC and IDF. The Representative of IDF drew attention to a new Compendium of methods on inhibitors in milk his Organization had produced. The IAM also noted that the Codex Committee on Veterinary Drugs in Foods had established a working group on methods of analysis

and that the Code of Hygienic Practice of Aquaculture prepared by the Codex Committee on Fish and Fishery Products includes provisions for veterinary drugs. Furthermore, the Bureau of Reference Materials (BCR) of the EEC is currently preparing a handbook on criteria and methods for the determination of veterinary drugs. Therefore, close collaboration had been established between BCR and the Codex Committee on Veterinary Drugs in Foods.

Methods of Analysis for Nutritional Food Labelling

16. The Chairman recalled that this item had been included in the agenda following a decision of the Seventh IAM. The IAM noted that in view of its particular interest in this subject, IDF had established relations with the Working Group on Methods of Analysis for Nutritional Labelling of the Codex Committee on Food Labelling.

Food Additives and Contaminants

17. The Representative of IFG said that the determination of heavy metal contaminants in wheat gluten referred to in document CX/MAS 91/15 is of particular interest to his Organization and ISO/TC 93/WG 3. The Representatives of AOAC and NMKL informed the IAM of relevant activities concerning TLC methods for mycotoxins and recent collaborative studies that had been carried out with methods for the determination of heavy metals, respectively. The IAM noted that CCMAS is currently reviewing its existing general methods for the determination of contaminants. For this purpose, a document (CX/MAS 91/2) had been included in the agenda of CCMAS.

Microbiology

18. The Representative of the Codex Secretariat was pleased to note that suitable methodology exists for most of the provisions specified by Codex. He also said that the Codex Committee on Food Hygiene will eventually make more use of the existing work. The Representative of ISO gave a brief account of the current work of Subcommittee ISO/TC 34/SC 9 "Microbiology". The relevant activities of ISO/TC 147/SC 4 "Water quality - Microbiological methods" were also mentioned in this context as well as methods developed by NMKL.

Nutrition and Foods for Special Dietary Uses

19. The IAM was informed by the Representative of IFG that the European Starch Association has been supporting a study on the determination of gliadin in wheat gluten. A suitable method is expected to be developed shortly. The Representative of the Codex Secretariat said that it has been decided to revise the Codex Standard on gluten free Foods.

Cocoa Products and Chocolate

20. The IAM noted with regret that OICCC had not responded to the invitation to attend and not submitted a written report on relevant activities. The Representative of the Codex Secretariat said that the proposed method for the determination of lead in cocoa powder had not been endorsed and that further study was Necessary.

Milk and Milk Products

21. The Representative of IDF informed the IAM of the results of the Meeting of the Joint FAO/WHO Expert Committee on Milk and Milk Products held in November 1990. He also referred to the new edition of the Inventory of methods jointly developed by IDF, AOAC and ISO. Copies of this Inventory had been mailed to all interested Organizations.

22. The Representative of IDF then reviewed the activities of the more than 40 IDF/ISO/AOAC Groups of Experts that has led to the publication of 115 Standards so

far. At present, there are over 100 projected standard methods, some of these being revisions. During the "Analytical Week 1991" (Milan 11 to 15 March 1991) 29 meetings were held with 166 participants. A new IDF sectional list and the programme of work will shortly become available .

Edible Ices

23. The Representative of IDF informed the IAM that the Weibull-Bentrop method for the determination of fat content of edible ices had been Published.

Fats and Oils

24. The IAM noted the existing good collaboration between IUPAC, AOAC and ISO but expressed its regret that no Representative of IUPAC was present at the IAM. The Secretary recalled that the Codex Secretariat had accepted the proposal of the IAM to include multiple references to all technically identical methods developed by different Organizations in Codex Standards. He noted that this decision had not yet been fully implemented in the documents prepared by the Codex Committee on Fats and Oils.

25. The IAM noted that ISO drafts on the determination of heavy metals (DIS 8294), and lead (CD 10229) in fats and oils are in preparation and could be made available for endorsement at a later Stage.

Fruit Juices

26. The IAM was informed of the relevant activities of AOAC, IFJU and CEN. It was suggested that new methods of analysis for fruit juices should be developed only if these are required for the verification of existing Provisions.

Processed Fruits and Vegetables

27. The Representative of the Codex Secretariat said that while some methods may still be lacking, no action by the IAM was required since some of the relevant Codex Standards are currently being reexamined for certain Criteria.

Processed Meat and Poultry Products

28. The Representative of ISO gave a brief account of the work of ISO/TC 34/SC 6 "Meat and meat products" which was developing Standards on chemical and microbiological methods. Progress has been made on methods for the determination of chloride, hydroxyproline, coloring agents, ascorbic acid, *Pseudomonas*, lactic acid bacteria, chloramphenicol and *Brochothrix*. New work will eventually cover subject such as histamines, ascorbic acid and the detection of the presence of parasites in meat and meat products.

Fish and Fishery Products

29. The IAM noted that apart from a method for the determination of water capacity of containers, no particular methods were required in this Area.

Sugars

30. The Representative of AOAC, speaking on behalf of ICUMSA said that a number of methods were still required for powdered sugars. However, neither ICUMSA nor ISO have been able to provide appropriate methods so far. The Representative of IFG offered his assistance in the development of a method for the determination of starch in icing sugars if such a method should be Required.

Starch Hydrolysis Products

31. The Representative of ISO reviewed the work of ISO/TC 93 "Starch, including derivatives and by-products" and drew attention to a possible future CEN activity in this area. The Representative of IFG said that ISO/TC 93/WG 1 was developing a method for the determination of starch content of starches that was required by the EEC with a view to a future Directive.

Cereals, Pulses and Legumes

32. The Representative of ICC gave a brief account of the work of his Organization, emphasizing the existing close collaboration with AOAC and ISO. He also said that ICC had used ISO 5725 as the basis for its Standard on repeatability and reproducibility.

33. The IAM also noted the relevant activities of ISO/TC 34/SC 4 "Cereals and pulses" and, in particular, the establishment of a working group under ISO/TC 34 that deals with the determination of protein, moisture and oil contents of cereals, pulses and oilseeds.

34. The Representative of the Codex Secretariat invited the IAM to provide the Codex Secretariat with a comparative table of different methods for the determination of fat acidity. The method which had been proposed (ISO 7305:1986) to replace the current methods uses a different principle. This had caused a problem since it would require a change of the level of the provisions for fat acidity in the respective commodity standards.

35. The Representative of the Codex Secretariat also informed the IAM that an alternative method of colour determination in pearl millet flour would be required. The modification to the current method proposed by Senegal had led to some problems. ICC was invited to assist the Codex Secretariat in this Matter.

Mineral Waters

36. The IAM noted that references to chemical and microbiological methods developed by ISO/TC 147 "Water quality" had been included in the relevant Codex documents. Existing ISO standards and drafts on the determination of radionuclides were also of interest in this Context.

Wines and Spirits

37. The Representative of OIV informed the IAM of recent developments in her Organization. A new edition of the OIV methods for wines and musts had been published which comprises new and current techniques such as GLC, HPLC and AAS. The OIV Sub-Commission on Analytical Methods is currently revising the Oenology Codex which includes test methods and specifications for the quality of products used in wine making. The Sub-Commission is also considering the lead content of these products in order to reduce the sources of contamination of wines and to reduce the maximum levels of contaminants. Another important subject is the content of ethyl carbamate in wines and spirits. While this does not constitute a problem in the case of wines, the technological process of the manufacture of spirits will be modified in order to reduce maximum levels of ethyl carbamate. This project is being carried out in the region of Bordeaux and coordinated by Mr Bertrand.

38. AOAC expressed interest in the determination of ethyl carbamate and agreed to establish direct contacts with Mr Bertrand on this subject. Contacts already exist between OIV and AOAC for collaborative studies which are being coordinated by Dr Junge of the Federal Health Office in Berlin, Germany.

OTHER ACTIVITIES OF INTEREST TO THE IAM

39. The Representative of IAEA (Dr L.G. Ladomery) informed the IAM of developments in the analysis of irradiated foods. The requirement that irradiated food be so labelled (Ref. Codex General Standard for the labelling of prepackaged foods) and that the overall average limit of 10 kGy for absorbed dose should not be exceeded are among the reasons why there is a need for methods for the detection of irradiated foods and quantitative methods to determine absorbed dose of radiation. Under the coordinated Research Programmes of IAEA and Community Bureau of Reference Materials (BCR) of the EEC promising methods are being developed and subjected to preliminary collaborative or cooperative testing. Among these are the electron spin resonance (ESR) method for foods containing bone, exoskeleton, seeds or stones, the thermoluminescence (TL) method for spices and field crops, lipid-derived volatiles, cyclobutanones from fatty acids and *o*-tyrosine for fatty food products. These and others may be ready for full collaborative testing. The Representative of IAEA expressed the hope that these methods will be tested collaboratively by interested International Organizations and that the IAM will be of assistance in this respect.

40. The Codex Secretariat confirmed its interest in these methods and indicated that any collaboratively tested methods would be considered by the CCMAS in due course. The Representative of AOAC expressed her Organization's interest in the methods and invited the IAEA to submit a brief article on the subject indicating the results obtained, foods and methods to be tested collaboratively for publication in the "Referee".

41. The IAM decided to put the question of methods of analysis of irradiated foods on its agenda for the next Meeting and invited the Representative of IAEA to keep the IAM informed of further developments.

42. An Observer to the IAM, Mr de Ruig (Netherlands) introduced his papers which he felt could be of interest to the future work of IAM and CCMAS. These papers cover topics such as a chemometric model for estimating the false-positive fraction and the false-negative fraction in food residues control programmes and criteria for the detection of analytes in test samples.

43. The Representative of NMKL informed the IAM that her Organization had recently published a NMKL Report No. 8 entitled "Quality Assurance Principles for chemical food laboratories". This manual is widely used in the Nordic countries. Any comments are welcome and should be submitted to the NMKL Secretariat.

HARMONIZED TERMINOLOGY IN THE FIELD OF METHODS OF ANALYSIS AND SAMPLING

44. The Representative of AOAC gave a brief account of the joint efforts of IUPAC, AOAC and ISO towards the harmonization of terminology, including the definitions. A document will shortly be published by IUPAC on nomenclature used in collaborative studies. This will, among others comprise concepts and definitions related to methods performance, laboratory performance and reference material specifications.

EXCHANGE OF VIEWS ON PROPRIETARY LABORATORY TECHNIQUES VERSUS TRADITIONAL METHODOLOGY

45. The Representative of IDF recalled that his Organization had established a policy document on the use of proprietary laboratory techniques which outlined a three stage approach to this subject:

- the review of the supplier's literature to support performance claims;
- the study by an independent body to check the performance;

- the study to be published by IDF concerning the performance.

46. The Representative of AOAC said that a special task force of his Organization was currently reviewing the entire approval process and offered to provide relevant information. NMKL and ISO also expressed their interest in this matter.

47. The Codex Secretariat welcomed discussion of this item and proposed that a review be developed through CCMAS and IAM that explains the current status of proprietary laboratory methods within the various interested Organizations. Several Organizations expressed their concern that the present uncoordinated activities might eventually lead to an undesirable duplication of work between the Organizations concerned.

48. As a result of the discussion, AOAC, IDF, ISO and IUPAC were requested to jointly develop a document on the principles of validation of proprietary laboratory techniques. Mrs M. Lauwaars (AOAC) agreed to coordinate the work on this document.

ANY OTHER BUSINESS

49. The Secretary urged the Representatives of the various Organizations participating in the IAM to prepare written contributions for the next IAM. This was considered to be important as the efficient work of the IAM very much depends on the input the Secretariat receives from the specialized Organizations.

50. The Representative of IAEA raised the question of methods for the detection and determination of radionuclide contamination of foods. The IAM was informed that Guidelines for maximum levels of contamination were available in CAC and FAO but that methods had not been requested so far. If these should be required, reference could be made to existing AOAC work. It was also pointed out that caesium constituted the only real current problem in trade in this respect. The IAM agreed that an item dealing with radionuclide contamination should be included in the agenda of the next Meeting.

DATE AND PLACE OF THE NEXT MEETING

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

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Budapest, 5 April 1991

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