

codex alimentarius commission

FOOD AND AGRICULTURE
ORGANIZATION
OF THE UNITED NATIONS

WORLD HEALTH
ORGANIZATION

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ALINORM 95/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION
Twenty-first Session
Rome, 3-12 July 1995

**REPORT OF THE 19TH SESSION OF THE
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Budapest, Hungary, 21-25 March 1994**

Note: This report incorporates Codex Circular Letter CL 1994/10-MAS.

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CX 4/50.2

CL 1994/10-MAS
May 1994

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

The report of the Nineteenth Session of the above Committee (ALINORM 95/23) will be considered by the Twenty-First Session of the Codex Alimentarius Commission (Rome, 3-12 July 1995).

PART A: MATTERS FOR ADOPTION BY THE COMMISSION

The following matters will be brought to the attention of the 21st Session of the Codex Alimentarius Commission for adoption:

- i) The Proposed Revised Protocol for the Design, Conduct and Interpretation of Collaborative Studies (para. 38, ALINORM 95/23).
- ii) The Proficiency Testing Harmonized Protocol for Laboratory Analysis (para. 43, ALINORM 95/23).
- iii) Five Codex General Methods of Analysis for Contaminants (para. 68 and Appendix III of ALINORM 95/23).

The Committee also endorsed provisions concerning methods of analysis for 50 Commodity Codex Standards (para. 62 and Appendix IV of ALINORM 95/23).

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 Alimentarius Commission Procedural Manual, to the Chief, Joint FAO/WHO Food Standards Programme, FAO, Via delle Terme di Caracalla, 00100 Rome, Italy, **no later than 31 October 1994.**

PART B: REQUEST FOR COMMENTS AND INFORMATION

I. CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS FOR CODEX PURPOSES

The Codex Secretariat and the Delegation of the United Kingdom with collaboration by Canada will study the impact of the implementation of the proposed Criteria for evaluating acceptable methods of analysis and other methods of analysis and prepare a revised paper to be circulated before the end of 1994 for comments (paras 29-33, ALINORM 95/23).

II. DRAFT CODEX GENERAL GUIDELINES ON SAMPLING

The Committee agreed to submit to governments amended Draft Codex General Guidelines on Sampling at Step 3 (paras 21-28, ALINORM 95/23).

III. DEVELOPMENT OF OBJECTIVE CRITERIA FOR ASSESSING THE COMPETENCE OF TESTING LABORATORIES INVOLVED IN THE IMPORT AND EXPORT CONTROL OF FOODS

The Committee agreed to circulate to governments and interested international organizations for comments "Development of Objective Criteria for Assessing the Competence of Testing Laboratories involved in the Import and Export Control of Foods" (paras 69-75, Appendix VII). Governments and interested international organizations are invited to forward their comments in writing to this office **by 31 October 1994.**

SUMMARY AND CONCLUSIONS

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

MATTERS FOR CONSIDERATION BY THE EXECUTIVE COMMITTEE AND THE COMMISSION:

- Recommended the adoption of the Proposed Revised Protocol for the Design, Conduct and Interpretation of Collaborative Studies, subject to no significant changes in the final review process by IUPAC (paras 34-38);
- Recommended the adoption for Codex purposes of the IUPAC/ISO/AOAC Harmonized Protocol for the Quality Control of (Chemical) Analytical Data (paras 44-47);
- Recommended adoption by reference of the Proficiency Testing Harmonized Protocol for Laboratory Analysis as being suitable for use for Codex purposes (paras 39-43);
- Recommended adoption of 5 General Methods of Analysis for Contaminants in Codex Standards at Step 8 as Codex General Methods; and
- Agreed to two editorial changes in the Checklist of Information Required and Guidelines to Evaluate Methods of Analysis (ALINORM 93/23, Appendices II and III), and proposed that these be presented to the Executive Committee so that the texts could be published in final form as soon as possible (para. 9 and Appendix II).

OTHER MATTERS OF INTEREST TO THE COMMISSION

- Recommended that the Report of the FAO Technical Consultation on Sampling Plans for Aflatoxin Analysis in Peanuts and Corn (FAO Food and Nutrition Paper No. 55) be also considered by the Codex Committee on Cereals, Pulses and Legumes when proposing maximum limits for aflatoxins in peanuts (paras 10 and 11);
- Agreed to request FAO and other International Organizations to seek possibility of publishing an exhaustive list of multi-element, multi-food and multi-element/multi-food analytical methods (para. 67);
- Noted the implications to food analysis of the prohibition of the use of toxic elements or reagents which are detrimental to the environment (para. 79);
- Recommended that the document CX/MAS 94/13 - Development of Objective Criteria for Assessing the Competence of Testing Laboratories involved in the Import and Export Control of Foods, be circulated to governments and interested international organizations for comments and that the paper should be referred to the Codex Committee on Food Import and Export Inspection and Certification Systems, for comments (paras 69-75);

SUMMARY AND CONCLUSIONS (Cont.d)

- Endorsed the following recommendations in the Proposed Draft Codex General Guidelines on Sampling:
 - * Simplification of existing sampling plans and the development of a hierarchy of plans to correspond to the level of sophistication and resources in the areas in which they are to be applied;
 - * Development of plans and procedures requisite for use in programs of certification of food processing at the producer, quality management system certification, and harmonization with existing certification and audit procedures applied to other aspects of the industry;
 - * Development of plans and procedures to encourage, simplify and make more effective process control procedures at the producer; and
 - * Study the existing standards to aid the Committees in specifying and improving sampling in their area of expertise; and

- Proposed to undertake the following new work:
 - * Harmonization of analytical terminology in accordance with international standards;
 - * Development of uniform criteria for the reporting of test results especially when the provision or specification to be tested, was not identical to the analyte; and
 - * Harmonization of reporting of tested results corrected for recovery factors.

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INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling held its Nineteenth Session from 21 to 25 March 1994 in Budapest, under the Chairmanship of Professor Peter Biacs, by courtesy of the Government of Hungary. The Session was attended by 83 delegates and observers from 33 countries and 8 International Organizations. A complete List of Participants, including the Secretariat, is provided in Appendix I to this report.

2. Mr. Auriel Salamon, President of the Hungarian National Codex Committee welcomed the Delegates and expressed the hope that by the end of the Session, Delegates would have made important and significant contributions to the work of the Codex Alimentarius Commission. Mr. Salamon underlined that the Hungarian National Codex Committee operated 17 Working Groups and had participated actively in the work of the Codex Committee on Import and Export Food Inspection and Certification Systems. He noted that Hungary attached a great importance to the results and recommendations provided by Codex Alimentarius for the development of Hungarian food regulations.

3. Mr. Salamon introduced Dr. Csaba Lovász, Vice Under-Secretary of State for Agriculture. Dr. Lovász emphasized the importance of international food trade for which modern methods of analysis and sampling were essential. He noted that food legislation had a long tradition in Hungary, the first Hungarian Food Act prepared and elaborated by manufacturers, adopted in 1895, being among the first in Europe. Hungarian specifications contained strict requirements to protect the health and interests of consumers.

4. With the cooperation of eminent food scientists and other experts, the Hungarian Ministry of Agriculture had prepared a draft of a new Food Act which had been submitted to the Hungarian Parliament for adoption. The main basic principles of this new food act included requirements covering the free circulation of foodstuffs in the territory of Hungary as an Associate Member of the European Union. The Hungarian Food Book (Codex Alimentarius Hungaricus) - being an important tool for food regulation in Hungary - will be compiled and edited in this manner. The official methods of analysis and sampling will be included in the Third Volume and would take into account the recommendations of the CCMAS.

ADOPTION OF THE AGENDA (Agenda Item 2)

5. The Committee **adopted** the Provisional Agenda (CX/MAS 94/1) as proposed. In order to facilitate discussions concerning endorsement of methods of analysis, the Committee **agreed** to discuss Agenda Item 14 directly after Agenda Item 12. The Committee was informed that Annex I of document CX/MAS 94/10 was amended and that the new copies and an Annex III to the same paper were available for distribution.

APPOINTMENT OF RAPPORTEUR (Agenda Item 3)

6. The Committee **agreed** with the proposal of the Chairman to appoint Ms. J.A. Springer (USA) as Rapporteur.

**MATTERS OF INTEREST ARISING FROM THE 20TH SESSION OF THE CODEX ALIMENTARIUS COMMISSION, CODEX COMMITTEES AND OTHER BODIES
(Agenda Item 4)**

a. Matters Arising from the 20th Session of the Codex Alimentarius Commission and Other Codex Committees

7. The Secretariat introduced documents CX/MAS 94/2 and CX/MAS 94/2 - Addendum 1 which contained extracts from the reports of the 20th Session of the Commission (ALINORM 93/40), the 25th Session of the Committee on Food Additives and Contaminants (ALINORM 93/12A), and the 25th Session of the Committee on Pesticide Residues (ALINORM 93/24A). The Committee was also provided with an oral report on the outcome of the 26th Session of the Committee on Food Additives and Contaminants which had been held in The Hague, 7-11 March 1994.

8. The Committee welcomed the change in its Terms of Reference as had been proposed at its previous session. It noted, however, that the Terms of Reference of the Codex Committees on Residues of Veterinary Drugs in Foods, Pesticide Residues, and Food Hygiene still provided for the autonomous development of methods for analysis and sampling by these Committees.

9. The Committee noted that the Commission had adopted the proposed texts of the **Checklist of Information Required and Guidelines to Evaluate Methods of Analysis** (ALINORM 93/23, Appendices II and III), but had requested the Committee to clarify certain inconsistencies in these texts and verify the method for the removal of outliers and the example attached to the text (ALINORM 93/40, paras. 263-264). The Committee was informed that the procedure used for control of outliers was consistent with internationally adopted methodology and that the example given in the text appeared to be accurate. In regard to the inconsistencies between the texts of Appendices II and III, the Committee agreed to two editorial changes and proposed that these be presented to the Executive Committee so that the texts could be published in final form as soon as possible. The text of the amended paragraphs is presented in Appendix II to this report.

10. The Committee noted that the Committee on Food Additives and Contaminants (CCFAC) had decided to continue consideration of methods of analysis for the determination of food additives in food, with special priority being given to additives which had a potential to create problems in international trade. It was noted that the methods, once identified by the CCFAC, would be submitted to the Committee for endorsement. Governments had been invited to provide comments and proposals on recommended and up-dated methods to the Canadian Delegation which had been requested by the CCFAC to develop the list of methods.

11. The Committee further noted that the CCFAC had not been able to recommend either limits or methods for sampling for aflatoxins. The CCFAC had, however, noted the report of the FAO Technical Consultation on Sampling Plans for Aflatoxin Analysis in Peanuts and Corn (FAO Food and Nutrition Paper No. 55). The Committee recommended that this report be considered by the Codex Committee on Cereals, Pulses and Legumes when proposing maximum limits for aflatoxins in peanuts.

b. Matters Arising from Other Bodies

AOAC International

12. The representative of AOAC International provided details of recent work undertaken by the organization. Attention was drawn to the development of two new categories for the validation of methods. The AOAC test kit performance programme certified manufacturers claims regarding the test kit. This involved expert review of performance characteristics developed by the manufacturer and

independent performance testing by at least one other laboratory. The representative provided a list of the test kits certified by the organization as part of the information document presented to the Committee. AOAC peer-verified methods formed the second new category of methods validated by the organization. The validation procedure involved in-house validation followed by validation in at least one other laboratory.

13. The representative also reported that AOAC International had continued its collaboration with ISO and IUPAC on the development of laboratory performance Protocols. The final report of a task force on methods for analysis of nutrients listed in food labels had been published in 1993, and it was reported that the organization had established a new technical division on biological and environmental reference materials.

European Economic Community

14. The observer reported that a compendium of methods for the determination of veterinary drug residues had recently been published.

International Atomic Energy Agency (IAEA)

15. The Representative of the Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture gave a brief account of developments in the field of analytical methods for irradiated food. The Representative pointed out that, as irradiation treatment did not change the nature or identity of food, any standards, codes, methods of analysis or sampling applied equally to irradiated food. There was, however, a need for verification of label declarations in regard to irradiated foods by the use of qualitative identification procedures. On the other hand, where upper or lower limits of the absorbed dose of irradiation were specified, quantitative methods were required.

16. The Committee was informed that several groups including the EEC, the European Committee for Standardization and a FAO/IAEA Coordinated Research Programme had been developing collaboratively tested methods for the identification of irradiated food. One method, based on the determination of volatile hydrocarbons, had been submitted to AOAC International for consideration. Another method, a microbiological method based on the Direct Epifluorescent Filter Technique/Aerobic Plate Count (DEFT/APC) approach, had been published in the Journal of the AOAC. Other methods being evaluated for different commodities included electron spin resonance, thermal luminescence, changes in viscosity, cyclisation of fat components, DNA fragmentation and changes in electrical impedance. Although some of the characteristics being tested could arise through other food processing techniques, their presence in otherwise unprocessed food was strongly indicative of the use of irradiation, and in one case (cyclisation of fat components) appeared to be specific to the irradiation process. The Committee noted that it was quite likely that there would be several collaboratively tested methods available for consideration and endorsement at the Committee's next Session.

17. On the other hand, it was unlikely that quantitative methods for the determination of absorbed dose would be available for the Committee's consideration in the near future.

Office International du Vin et de la Vigne (OIV)

18. The Committee was informed that a comprehensive list of methods of analysis for alcoholic beverages had been published in 1993, and that a second edition (in French only) would become available in 1994. The OIV had also prepared a comprehensive study of maximum residue limits for pesticides in wine and in fruit, mainly grapes, covering 700 different limits applied in some 36 countries. This list would be published in 1994.

International Union of Pure and Applied Chemistry (IUPAC)

19. IUPAC continued to work on the development of the Revised Protocol for Collaborative Studies (see Item 7 of the Agenda) and with ISO and AOAC International on Harmonized Protocol for the Determination of Laboratory Proficiency and Quality Control of Chemical Analytical Data (see Items 8 and 9). IUPAC's Commission on Food Chemistry was developing methods for the determination of mycotoxins other than aflatoxins, phycotoxins, residues of veterinary drugs and trace elements. IUPAC's Commissions on Fats and Oils and on Agrochemicals continued to develop methodology in these areas. It was noted that Division V.1 of IUPAC, i.e. Division on General Aspects of Analytical Chemistry, was working on limits of detection and reporting of results.

IDF/ISO/AOAC Cooperation on Methods of Analysis for Milk Products

20. The Representative of AOAC International reported that meetings of joint groups of experts of these organizations had been held with the intention of providing methodology for use by the Codex Committee on Milk and Milk Products. An updated edition of the IDF/ISO/AOAC Inventory of Methods of Analysis had been published (IDF Bulletin 286/1993). The Inventory contained references to IDF, ISO and AOAC International methods listed on the basis of both product and analyte.

PROPOSED DRAFT CODEX GENERAL GUIDELINES ON SAMPLING (Agenda Item 5)

21. The Committee had before it document CX/MAS 94/3 prepared by a Consultant, Dr. Edward Schilling¹ and CX/MAS 94/3-Addendum 1 prepared by FAO (Food Quality and Liaison Group of the Food Policy and Nutrition Division). The Committee noted that at its 18th Session it had discussed a similar paper and delegates attending that meeting were of the opinion that in view of the horizontal approach recommended by the Commission, the Codex advisory text on sampling should be applicable to all commodities including pesticide residues, veterinary drug residues, aflatoxins and toxic elements.

22. In introducing the document, Dr. Schilling said that it had been prepared using existing materials and discussions on the matter. Dr. Schilling emphasized that the document was a first draft and still required modification.

23. In his presentation, Dr. Schilling highlighted the arrangement of the paper and its usefulness not only for Codex purposes but also for sampling in other fields where the document was applicable. Secretariat informed the Committee that the paper when finalized would be a useful guide to governments (food inspectors), traders (importers and exporters) and food producers who wish to maintain the quality of their products. It was made clear that the draft document was intended for use by governments or organizations in developing their national or organizational policy guidelines on sampling.

24. The paper was discussed in-depth by the Committee. Some delegations submitted written comments which were categorized into editorial and substantive or long term items. Some of the amendments which were proposed included:

- to include preliminary documentation to define parameters of the plan;
- to include considerations of destructive and non-destructive sampling and/or analysis;

¹ Edward G. Schilling, Ph.D., P.E., Director and Professor, Center for Quality and Applied Statistics, Rochester Institute of Technology, New York, NY.

- to give attention to the Codex methods of sampling for pesticide residues;
- to include procedures for semi-curtaiment in the document;
- to delete the discussion part on "Net Contents" in the document (page 13);
- the document should also address acceptance of a variables sample with a defect in it (possibly in mixed plans);
- 5.3.3(i) third line, the word "contaminated" to be replaced with "defect";
- 5.4.1.1, 2nd paragraph, 5th line substitute "production" for "industry";
- 5.4.1.2, 7th line from top of page 11, add to end of the sentence the following, "or with the relevant regulations of the importing country";
- 5.5.6 - the title "Preparation of Bulk Sample" to read "Preparation and Submission of Bulk Sample";
- 5.5.6.2 add after the section the following, "The bulk sample should be submitted to the laboratory and the final sample prepared under laboratory conditions";
- 5.5.7.2 - to be deleted, and 5.5.7.3 to be renumbered;
- the substitution of equivalent terminology from ISO 7002, 8759, 3534 in the list of definitions;
- ISO and IUPAC would be invited to supply lists of documents on sampling for possible harmonization with the documents;
- consideration should be given to the impact of risk (as OC curve);
- review of the statement that sampling plans can be compared with Type I methods;
- to develop plans for changing sampling rates (or refer to ISO 2859 Part 1);
- to mention other considerations in determining consumer risk (5.4.1.2);
- to extend Table 4 to other probability levels;
- to include a test for normality;
- address heterogeneity or clustering of populations; and
- it was noted that the "Recommendations" on page 13 would not be reflected in the revised document.

25. Those comments which were long term issues and probably would have to be taken up by the Committee in the future included:

- determination of the incidence rate in Table 2;

- economic feasibility of the sample size in Table 6;
- incorporation of average level into the plan;
- provision of a guide to sampling procedures as part of a "Manual on Sampling";
- incorporation of a glossary of sampling terms;
- inclusion of sequential plans;
- development of special plans and simple criteria or approximation for use with non-normal distributions;
- consideration of large scale effects in systematic sampling in 5.3.2b.

26. The Committee endorsed the following recommendations:

- a) Simplification of existing sampling plans and the development of a hierarchy of plans to correspond to the level of sophistication and resources in the areas in which they are to be applied.
- b) Development of plans and procedures requisite for use in programs of certification of food processing at the producer, quality management system certification, and harmonization with existing certification and audit procedures applied to other aspects of the industry.

Note: This has been started in this document with an emphasis on the translation of Codex sampling procedures into ISO 2859 and similar documents.

- c) Development of plans and procedures to encourage, simplify and make more effective process control procedures at the producer.
- d) Study the existing standards to aid the Committees in specifying and improving sampling in their area of expertise.

27. It was noted that the recommendation to develop a programme of training and consultation in sampling and statistical process control fell outside the terms of reference of the Codex Alimentarius Commission, but could be taken up by FAO or other responsible organizations.

28. Delegates expressed their appreciation to Dr. Schilling for the excellent paper and agreed that the document should be revised in the light of comments made and circulated for comments at Step 3. The finalized documents would be recommended for inclusion in Volume 13 of Codex Alimentarius.

CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS FOR CODEX PURPOSES (Agenda Item 6)

29. The Committee had before it document CX/MAS 94/4 which was prepared by the Delegation of the United Kingdom. The Committee recalled that this item had been discussed at the last session without reaching an agreement. The Delegation of the United Kingdom introduced the paper stressing that in the light of GATT Agreement on Application of Sanitary and Phytosanitary Measures, mutual recognition across governments and concept of equivalence should be incorporated by Codex. As Codex had already reached the point where there was less development of traditional type methods and more emphasis was being placed on a horizontal approach. In order to facilitate Codex work and to

overcome disadvantage in current procedures, an alternative approach was proposed - to define criteria and to choose methods which met criteria instead of specifying individual methods. This approach was explained by using three examples, toxic elements, aflatoxins and residues of veterinary drugs with an emphasis that one of Codex Committees had already taken this approach.

30. Majority of delegations were in favour of this new approach. The Committee noted that at EC level, criteria for methods are already used in some areas such as residues in animal and animal products intended for human consumption. It was suggested for better evaluation of methods by analysts that collaborative studies be published in scientific literature.

31. The Delegation of the United States strongly opposed this approach. It was stressed that in case of dispute only one method should be chosen from acceptable methods (in the current system, Type II method) and should be used by all parties involved. It was stated that the proposed approach would transfer arguments from this Committee to a forum which dealt with the legal settlement of disputes. It was pointed out that the criteria contained in the paper would not help if the level of the analyte was near the limit of determination. Even if a method met criteria, it could possibly give an apparently unrealistic result depending on mean and variability.

32. Several countries questioned consequences of the implementation of this approach though it was suggested that while in practice there would not be much change for the time being, there may be changes in the future. The Delegation of Canada was requested to provide performance data of methods of analysis for contaminants (see Appendix III) for preparation of the paper.

33. The Committee agreed that the Codex Secretariat and the Delegation of the United Kingdom with collaboration by Canada should study impact of the implementation of the proposed approach on the existing Codex methods of analysis and other methods of analysis and prepare a revised paper to be circulated before the end of 1994 for comments.

PROPOSED REVISED PROTOCOL FOR COLLABORATIVE STUDIES (Agenda Item 7)

34. The Committee had before it document CX/MAS 94/5. The Secretariat recalled the Committee's earlier consideration of the Protocol during the 18th Session when the Committee had agreed that the revised Protocol should be transmitted to IUPAC for consideration (ALINORM 93/23, para. 39). The Representative of IUPAC informed the Committee that the amendments were reflected in the introduction section of the present document. The revised Protocol had been circulated to IUPAC members and other interested organizations for final review prior to its final publication.

35. Delegations welcomed the development of the Protocol and were in favour of recommending its use for the evaluation of methods of analysis to be used for Codex purposes. The Committee noted the significance of changes made to Section 2 and to the first paragraph of Section 4 of the document.

36. The representative of AOAC International expressed the view that comments received by IUPAC should also be provided to other interested organizations developing similar Protocols in order to assure harmonization. The Representative further informed the Committee that ISO and AOAC were in the process of examining the Protocol with the view of reaching concurrence on the document. In particular AOAC International was in the process of examining the treatment of outliers contained in the Protocol which, in its present form, would eliminate more laboratories as outliers than in previous versions.

37. The Committee also noted that the Protocol had been developed from an earlier ISO standard (ISO 5725:1986) and that the ISO document would need to be revised. However, the Committee was informed that responsible ISO Technical Committee and IUPAC were cooperating satisfactorily in this

regard and it would appear that the CCMAS was serving as the catalyst for the harmonization of work between the interested organizations.

Status of the Protocol for the Design, Conduct and Interpretation of Collaborative Studies

38. The Committee agreed that the Protocol would be useful to Member Governments in selecting suitable methods for analysis of foods and for Codex purposes. The Committee provisionally recommended that the Protocol be submitted to the Commission at its 21st Session in 1995 for adoption provided that there were no significant changes in the final review process by IUPAC. IUPAC was invited to communicate the final action taken on the Protocol to the Executive Committee through the Codex Secretariat.

PROGRESS REPORT ON THE DEVELOPMENT OF A PROFICIENCY TESTING HARMONIZED PROTOCOL FOR LABORATORY ANALYSIS (Agenda Item 8)

39. The Committee had before it document CX/MAS 94/6 and the full text of the Protocol as approved by IUPAC/ISO/AOAC and presented in a Conference Room Document (un-numbered). In introducing the document the Delegation of the United Kingdom stated that one aspect of ensuring the quality of laboratory data was the requirement for proficiency testing of laboratories. It was noted that although proficiency testing was a requirement of laboratory accreditation it was in itself not accreditation. Participation in proficiency testing schemes provided laboratories with the means to objectively assess and demonstrate their ability to produce consistent and reliable results.

40. The Committee noted that the Protocol would be of considerable interest to other Codex Committees, and in particular to the Codex Committee on Food Export/Import Inspection and Certification Systems (CCFICS). Certification systems were frequently supported by laboratory analyses which needed to be of an assured quality to provide confidence in the certification system itself. It was noted that CCFICS was concerned with the general principles and guidelines relating to the inspection and certification programmes, of which laboratory quality assurance and proficiency programmes could be essential aspects, but that the preparation of codes of practice and related texts for food laboratory practices, including analytical quality assurance and proficiency programmes, were the responsibility of the Codex Committee on Methods of Analysis and Sampling in cooperation with other international organizations competent in this field.

41. The Committee was informed by the U.K. Delegation that the need for proficiency testing had been recognized in the EEC and that a similar Protocol had been approved in the Council Working Group discussions on the EC Directive concerned with the additional measures for food control. Food control laboratories in the EEC would have to operate under defined guidelines which include:

- accreditation to EN 45000 and some aspects of the OECD GLP Guidelines;
- Good Laboratory Practice (GLP);
- participation in proficiency testing schemes; and
- use of validated methods of analysis.

42. Some delegations questioned the use of combined scores in Appendix III of the document as this practice did not conform to the EC Directive, nor was it commonly used in food laboratories. It was pointed out that the Draft Protocol was intended for a wide range of uses, including non-food uses, for which combined scores were appropriate.

Status of the Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories

43. The Committee recommended to the Commission that the Protocol be adopted by reference as being suitable for use for Codex purposes.

PROGRESS REPORT ON THE DEVELOPMENT OF AN IUPAC/ISO/AOAC HARMONIZED PROTOCOL FOR THE QUALITY CONTROL OF (CHEMICAL) ANALYTICAL DATA (Agenda Item 9)

44. The Committee had before it document CX/MAS 94/7, prepared by the Delegation of the United Kingdom, and a room document which contained the **IUPAC/ISO/AOAC Harmonized Guidelines for Internal Quality Control in Analytical Chemistry Laboratories**. The Delegation of the United Kingdom introduced the documents and informed the Committee that the objective of the Protocol was to aid laboratories in their work. The Protocol had been drafted and revised by a joint IUPAC/ISO/AOAC Working Group which had also developed other Protocols discussed at this session. It was emphasized that the Protocol was still at draft stage. The Delegation also informed that this revised Protocol would be discussed by IUPAC in May 1994.

45. Many delegations welcomed the Protocol, expressed their willingness to study and comment on it and agreed that consideration of the Protocol should be included in future work of the Committee.

46. Noting concern about possible conflict between the corresponding EC Protocol and the present Protocol should it be recommended by the Codex at laboratory level, the Committee noted that the document was not in conflict with existing texts but complementary to ISO 25 series and the like.

47. It was agreed that after a revised Protocol was adopted by IUPAC, the Committee should consider the adopted Protocol with a view towards adopting it for Codex purposes.

REPORT OF THE TENTH MEETING OF INTERNATIONAL ORGANIZATIONS WORKING IN THE FIELD OF ANALYSIS AND SAMPLING (INTER-AGENCY MEETING) (Agenda Item 10)

48. The Committee had before it Conference Room Document 1 containing the Report of the Tenth Inter-Agency Meeting (IAM), held at the Conference Room of the Hungarian Office for Standardization (MSZH) on Friday 18 March 1994. The Report was presented by Mr. K.-G. Lingner (ISO), Secretary of the IAM. The meeting which was attended by representatives of eleven international organizations actively working in the field of food analysis and control was chaired by Mr G. Castan (ISO). The Report of the IAM is attached to the report as Appendix VI.

49. The IAM had considered matters of interest to CCMAS such as:

- exchange of information on collaborative studies;
- status of joint work by AOAC International, IUPAC and ISO towards a harmonized Protocol on internal laboratory quality control;
- methods of analysis and sampling required by some of the Codex subsidiary bodies;
- an exchange of views on proprietary laboratory techniques versus traditional methodology.

50. The IAM, in accordance with its terms of reference, confirmed its availability to provide advice on matters relating to methods of analysis and sampling that might be referred to it by the CCMAS. The Committee was informed that written report of activities had been provided by AOAC International, IUPAC, EOQ, Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture, ICC, IDF, ISO, NMKL and OIV.

51. In the discussion which followed, Delegates expressed appreciation for the work of the IAM and looked forward to its future contribution to the work of Codex. Some Representatives of international organizations requested that a list of documents of work being carried out by organizations be listed as an appendix to the report of the IAM.

52. The Committee noted the report and expressed its appreciation to the organizations represented at the Inter-Agency Meeting (IAM) for their collaboration in meeting the needs of CAC in the area of methods of analysis and sampling.

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

53. The Committee had before it document CX/MAS 94/8 prepared by the Delegation of the United Kingdom which contained information on the activities of interest to the CCMAS being undertaken by national and international institutions. The Committee was informed that extensive information had been provided by these institutions which was useful in coordinating methods development and preventing duplication of work.

54. Several Delegations indicated their support for this exercise and requested that it should be continued. Organizations (including national and international institutions) having information were encouraged to respond to future requests and it was suggested that the information might be coordinated through the IAM. The Committee expressed its continued interest in the field and requested to be kept informed on future progress.

ENDORSEMENT OF CODEX METHODS AND THEIR CLASSIFICATION (Agenda Item 11)

55. The Committee had before it document CX/MAS 94/9 prepared by The Netherlands, after consultation with Egypt, Hungary, U.K., U.S.A. and the IFG. Written comments from the Russian Federation were also provided. The Committee noted that matters strongly related to this item had also been discussed under Item 6. The Delegation of The Netherlands, in introducing the document, emphasized that in the light of new Agreement on the Application of Sanitary and Phytosanitary Measures, Codex methods of analysis would be used for reference especially in cases of dispute. The paper proposed that method Types II and III could be considered to be equivalent and therefore should be merged to form a new Type II to simplify the procedure and to give analysts wider choice and allow the Committee to endorse all methods meeting criteria.

56. The Delegation of the United States reminded the Committee that the purpose of creating Type III was to eliminate such problems as when two equivalent methods gave different results and legal disputes arose about the selection of method. Type III methods could be used generally for trade purposes, but in case of dispute Type II methods were required. It was noted that the selection of a Type II method from available Type III methods was often based on subjective criteria such as cost, ease of use or availability of reagents.

57. The Committee noted that a considerable number of Codex methods were Type I methods which had not been collaboratively studied but had been used extensively for many years and were generally considered to be reliable in their performance. Similarly, it was recognized that a number of Type IV

methods had not been evaluated and therefore could not be considered for inclusion as either Type II or III. It was suggested that through proficiency testing, quantitative data on reliability of these methods might be available.

58. The Committee encouraged organizations concerned to use data from proficiency testing in order to evaluate traditional Type I and Type IV methods which had not been collaboratively tested. It decided to await future progress in developing criteria for the evaluation of methods (see para. 33 above) before deciding on whether Types II and III should be merged to form one Type. These matters would be discussed together at the Committee's next session.

ENDORSEMENT OF METHODS OF ANALYSIS IN CODEX STANDARDS AND METHODS OF ANALYSIS REQUIRED FOR CODEX STANDARDS (Agenda Items 12 and 14)

59. The Committee had before it the Conference Room Document 2 containing a report of a meeting of the Working Group on Endorsements which had met on 19 March 1994. The following Member Countries and International Organizations were represented: Canada, Finland, France, Hungary, The Netherlands, Thailand, United Kingdom, United States of America, AOAC International, ISO, IUPAC, OIV and NMKL.

60. The Report of the Working Group was presented by its Chairman, Dr. William Horwitz (USA). Dr. G. Diachenko (USA) served as Rapporteur. The Working Group had considered:

- (i) Methods of analysis submitted for endorsement as contained in document CX/MAS 94/10;
- (ii) Methods of analysis for foods for special dietary uses contained in ALINORM 93/26, Appendix IV; and
- (iii) Methods of analysis required by Codex which needed to be developed and/or validated as presented in document CX/MAS 94/12.

61. The Committee discussed the Report of the Working Group. The following remarks were made during the discussions:

- (a) It was proposed that reference to sodium chloride in the provisions for sodium chloride in bouillons and consommées should be "Chloride calculated as Sodium Chloride". The Secretariat was requested to ascertain the correct wording in the Standard and advise the respective Commodity Committee and amend the records.
- (b) The Working Group informed the Committee that if the protein conversion factor is specified in the method then the method is classified as a Type I method, but if prescribed in the commodity standard independent of the method then it should be classified as a Type II method.
- (c) With regard to methods of analysis of fats and oils the Committee noted that in addition to the recognized international organization, the CEN was also undertaking work in this area.
- (d) AOAC International confirmed that the AOCS method for lead in fats and oils was similar to AOAC 994.02, IUPAC 2.632 and ISO 12193 methods.
- (e) Some delegations requested that the Secretariat confirm the Commission's view on the analyte - mercury/methyl mercury in fish.
- (f) The Committee noted that some methods of analysis such as AOAC 925.25 required the use of banned chemicals such as chloroform and asbestos which were to be phased out and be replaced

by safer chemicals and reagents. It was noted that this could have implications for the validity of previous reliability studies.

- (g) Some delegations indicated their preference to express heat/energy in kilojoules rather than in kilocalories. The Committee was informed that the Codex Committee on Nutrition and Foods for Special Dietary Uses had been considering the use of the two units.

62. The methods of analysis considered and endorsed by the Committee are given in Appendix IV, together with detailed notes on some of the methods.

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

63. The Committee had before it document CX/MAS 94/11 prepared by the Delegation of Canada and written comments from the Russian Federation and the IDF. The Committee was informed that the Codex Alimentarius Commission at its 20th Session indicated that this work was of high priority and expressed the hope that a suitable final text would be available for consideration at Step 8 by the 21st Session of the Commission. During the introduction of the document, the Delegation of Canada proposed that the Committee recommend five of the ten methods discussed at the last session taking into consideration comments received on these methods: Methods 2, 4, 5, 6 and 8. These methods were not identical and there was no single method that was applicable to all foods. Nevertheless, with these five methods, eight different elements could be determined in a wide variety of food although there might be exceptions such as milk products as pointed out by IDF.

64. The Committee was informed that the CCMAS generally endorsed methods of analysis proposed by other Codex bodies. However, since these five methods had been considered by the CCMAS itself, it was proposed that they be recommended as general Codex methods of analysis of contaminants for Codex purposes. Should these methods be recommended to be used in case of dispute, Type classification would be necessary. It was suggested that these methods be included in the list of Codex general methods.

65. The Delegation of the Russian Federation asked the Committee to endorse the method submitted by Russia. This method had not been included in the recommendation, however, due to the lack of collaborative study and its not being a complete method.

66. The Delegation of Hungary welcomed the document and discussed the situation of trace element analysis in Hungary. Methods using the least steps and least chemicals in the sample preparation are preferred in order to prevent the sample both from contamination and losses of the elements. Inter-laboratory trial in Hungary had discovered importance of analytical expertise. For this reason, necessity in giving close attention to proficiency testing was reinstated.

67. The Committee noted that at its 18th Session a working document containing about 700 references on multi-element, multi-food, and multi-element/multi-food analytical methods had been presented by the Delegation of Canada and many comments on the document were received since. A number of delegations expressed their support for publication of this useful document and some organizations showed interest in publishing it, noting that it would require updating. The Committee agreed to request FAO and other international organizations to seek possibility of publishing an exhaustive list of methods.

Status of General Methods of Analysis for Contaminants in Codex Standards

68. The Committee decided to recommend these five methods for adoption by the Commission at Step 8 as Codex General Methods. These methods are listed in Appendix III of this report.

DEVELOPMENT OF OBJECTIVE CRITERIA FOR ASSESSING THE COMPETENCE OF TESTING LABORATORIES INVOLVED IN THE IMPORT AND EXPORT CONTROL OF FOODS (Agenda Item 15)

69. The Delegation of Finland introduced document CX/MAS 94/13, which had been prepared on the basis of discussions which had taken place during the 20th Session of the Commission in July 1993 in regard to systems for the import and export control of foods. It was noted that whereas the CCFICS had been given responsibility for developing guidelines on import and export inspection and certification systems in general, the CCMAS was responsible for developing similar guidance for testing laboratories.

70. The document discussed aspects of laboratory accreditation, but stressed that competence could not be assessed by accreditation alone. The paper proposed that the criteria established in ISO/IEC Guide 25:1990 "General Requirements for the Competence of Calibration and Testing Laboratories" would be suitable for assessing the competence of laboratories involved in import and export control of foods if supplemented by additional criteria, especially:

- participation in schemes for proficiency testing; and
- insistence on the use of analytical methods which have been fully validated in interlaboratory method performance studies, wherever possible.

71. Delegations generally welcomed the paper, considering it to be very timely and urgently required for use in international trade. It was noted that many governments were considering alternative ways of obtaining the analytical services required in relation to certification of foods moving in international trade, partly to contain costs and to achieve maximum efficiencies. The use of accredited laboratories outside the traditional government framework was one way of achieving this, but that there had to be means for assessing the competence of such laboratories.

72. Some delegations referred to the work being undertaken by the CCFICS on mutual recognition of inspection and certification systems including certification of laboratory results, but stressed that some national legal systems would continue to provide for the inspection of imported food in conformity with national legislation. The Observer of the EEC stated that the European Community would not be in a position to accept opinions on the measures under discussion, in relation to laboratory accreditation and proficiency testing except in so far as these are consistent with the existing Community Legislation.

73. A question was raised as to the use of ISO/IEC 25:1990 within the framework of quality systems operating in conformity to ISO 9000 documents. It was suggested that certification of food consignments in conformity with ISO 9000 would automatically include procedures for assuring the quality of laboratory results. It was pointed out, however, that testing laboratories could operate under ISO 9000 quality schemes as part of the manufacturing process or as part of a certification system but still be required to demonstrate technical competence. It was noted that accreditation according to ISO 25 did not have the same objective as certification under ISO 9000.

74. The Committee recognized that the accreditation of laboratories was not, in itself, a matter for the CAC. The question arose as to whether the CAC should establish objective criteria itself or whether to recommend criteria or Protocols developed by other international organizations competent in the field. It was agreed that the subject matter fell within the broad framework of the CAC's responsibilities and,

even though it was not specifically included within the Committee's Terms of Reference, the CCMAS was the appropriate forum for further discussion of the subject.

75. The Committee agreed that the paper should be considered as a basis for recommendations to governments in this area. It agreed to request governments and interested international organizations for comments and that the paper should be referred to the CCFICS for comment. The paper is attached to this report as Appendix VII.

FUTURE WORK (Agenda Item 16)

76. The Committee noted that with the conclusion of several substantive items on its current agenda, the following matters would be scheduled for discussion at its next Session:

- Consideration of the IUPAC/ISO/AOAC Harmonized Protocol for the Quality Control of (Chemical) Analytical Data (Step 7);
- Consideration of Proposed Draft General Guidelines on Sampling (Step 4);
- Consideration of Objective Criteria for Assessing the Competence of Testing Laboratories Involved in Import and Export Control of Foods (Step 4);
- Criteria for Evaluating Acceptable Methods of Analysis for Codex Purposes and Classification of Such Methods;
- Report of the Inter-Agency Meeting on Methods of Analysis and Sampling; and
- Endorsement of Methods of Analysis and Sampling for Codex Purposes.

77. The Committee agreed to propose that the following new work be undertaken by the Commission:

- Harmonization of analytical terminology in accordance with international standards;
- Development of uniform criteria for the reporting of test results especially when the provision or specification to be tested was not identical to the analyte; and
- Harmonization of reporting of tested results corrected for recovery factors.

78. In the latter case the Committee noted that it was normal analytical procedure to apply corrections for recovery factors but that this was not applied uniformly across Codex methods of analysis. In particular it was noted that the results of pesticide residue analyses were not corrected for recovery. The Committee welcomed the offer of the Representative of IUPAC to prepare a report for the Committee's consideration.

79. The Committee noted that AOAC International and other organizations were considering the implication of prohibitions on the use of certain toxic or environmentally detrimental materials or reagents used in previously evaluated and endorsed methods of analysis. It was noted that this might have implications for the Committee's future work.

80. The Committee was informed that the Commission had requested all Codex Committees to consider their medium-term objectives as a standing agenda item (paras 75-79, ALINORM 93/40), and that a report on the Committee's current status of work (see Annex 1) should be made to the Executive Committee on a regular basis, to be reviewed in light of the medium-term objectives.

OTHER BUSINESS (Agenda Item 17)

81. There was no other business.

DATE AND PLACE OF THE NEXT SESSION (Agenda Item 18)

82. The Committee was informed that its 20th Session was tentatively scheduled to be held in Budapest in the first week of November 1995, the exact date to be determined between the Hungarian and the Codex Secretariats.

SUMMARY STATUS OF WORK

SUBJECT	ACTION TO BE TAKEN BY	DOCUMENT REFERENCE ALINORM 95/23
Approval of Amendments to the Checklist of Information Required and Guidelines to Evaluate Methods of Analysis	41st Session, Executive Committee	Appendix II, para. 9
Adoption of the Revised Protocol for Collaborative Studies	21st Session CAC	Appendix V
Adoption of Proposed Revised Protocol for Collaborative Studies	IUPAC, Codex Secretariat, Executive Committee, 21st Session CAC	para. 38 and Appendix V
Adoption of Proposed Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories	21st Session CAC	paras 44-47
Adoption of 5 General Methods of Analysis for Contaminants in Codex Standards at Step 8 as Codex General Methods	21st Session CAC	Appendix III
Proposed Draft Codex Guidelines on Sampling	Codex Secretariat, Governments and 20th Session CCMAS	paras 21-28
Criteria for Evaluating Acceptable Methods of Analysis for Codex Purposes	UK, Canada, Codex Secretariat and 20th Session CCMAS	paras 29-33 and CX/MAS 94/4
Endorsement of Codex Methods and Their Classification	20th Session CCMAS	paras 55-58
Development of Objective Criteria for Assessing the Competence of Testing Laboratories Involved in the Import and Export Control of Foods	Governments and 20th Session CCMAS	Appendix VII, paras 69-75

SUBJECT	ACTION TO BE TAKEN BY	DOCUMENT REFERENCE ALINORM 95/23
Harmonization of Analytical Terminology in Accordance with International Standards	20th Session CCMAS	para. 77
Uniform Criteria for the Reporting of Test Results Especially when the Provision or Specification to be Tested Was Not Identical to the Analyte	20th Session CCMAS	para. 77
Application and Reporting of Test Results Corrected for Recovery Factors	20th Session CCMAS	para. 77

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**AMENDMENTS TO THE CHECKLIST OF INFORMATION AND PRECISION
REQUIREMENTS REQUIRED TO EVALUATE METHODS OF ANALYSIS SUBMITTED
TO THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING FOR
ENDORSEMENT**

(Submitted to the Executive Committee for Adoption ¹)

1. Amend the second paragraph on page 26 of the Report of the 18th Session of the Committee (ALINORM 93/23) to read as follows:

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.~~ (Rest of the paragraph unchanged)

2. Amend the first paragraph on page 29 of the Report of the 18th Session of the Committee (ALINORM 93/23) to read as follows:

It has been shown that when the precision of the 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 when considered over many orders of magnitude. (Rest of the paragraph unchanged)

(Deletions are ~~struck out~~, additions are underlined.)

¹ See the Report of the 20th Session of the Codex Alimentarius Commission, ALINORM 93/40, paragraphs 263-264.

**DRAFT CODEX GENERAL METHODS OF ANALYSIS FOR CONTAMINANTS
(At Step 8 of the Procedure)**

The following methods are recommended as general methods for the analysis of metal contaminants in foods. These methods cover eight elemental contaminants in a wide variety of foods, however no single method covers all foods nor all elements. The choice of method will depend on the required analysis.

1. Lead and Cadmium in Food

Principle: Anodic stripping voltammetry

References:

R.J. Gajan, S.G. Capar, C.A. Subjoc and M. Sanders - *Determination of Lead and Cadmium in Foods by Anodic Stripping Voltammetry: I. Development of Method.* J. Assoc. Off. Anal. Chem. 65, 970-977 (1982).

S.G. Capar, R.J. Gajan, E. Madzsar, R.H. Albert, M. Sanders and J. Zyren - *Determination of Lead and Cadmium in Foods by Anodic Stripping Voltammetry: II. Collaborative Study.* J. Assoc. Off. Anal. Chem. 65, 978-986 (1982).

Association of Official Analytical Chemists. *Cadmium and Lead in Food. Anodic Stripping Voltammetric Method*, Official Final Action, 1988. AOAC Official Methods of Analysis, 1, (92.23) 239-241, AOAC Arlington, VA, (1990).

Applications:

The method appears excellent for screening and survey work. The method appears reasonable for regulatory applications provided the poorer precision caused by the application of standard additions can be tolerated, the test for thallium is incorporated as a safety measure, and that matrix effects are determined to determine baseline error.

2. Copper, Iron and Nickel in Edible Oils and Fats

Principle: Direct graphite furnace atomic absorption spectrometry

References:

P.W. Hendrikse, F.J. Slikkerveer, J. Zaalberg and A. Hautfenne - *Determination of Copper, Iron and Nickel on Oils and Fats by Direct Graphite Furnace Atomic Absorption Spectrometric Method.* Pure and Applied Chemistry, 60 (6) 893-900 (1988).

Association of Official Analytical Chemists. *Copper, Iron and Nickel on Oils and Fats by Direct Graphite Furnace Atomic Absorption Spectrometric Method*, Official Final Action, 1990 method. AOAC Official Methods of Analysis, 15th ed., 1st Supplement, (990.05) 7-8. AOAC, Arlington, VA (1990).

Applications:

The method appears excellent for screening, survey and regulatory work.

3. Lead in Edible Oils and Fats

Principle: Direct graphite furnace atomic absorption spectrometry

Reference:

P.W. Hendrikse, F.J. Slikkerveer, A. Folkersma and A. Dieffenbacher - *Determination of Lead in Oils and Fats by Direct Graphite Furnace Atomic Absorption Spectrometric Method*. Pure and Applied Chemistry, **63** (8) 1183-1190 (1991).

Applications:

The method appears excellent for screening, survey and regulatory work.

4. Tin in Canned Foods

Principle: Atomic absorption spectrometry

References:

R.W. Dabeka, A.D. McKenzie and R.H. Albert - *Atomic Absorption Spectrophotometric Determination of Tin in Canned Foods Using Nitric Acid-Hydrochloric Acid Digestion and Nitrous Oxide Acetylene Flame*, J. Assoc. Off. Anal. Chem. **68** (2) 209-213 (1985).

Association of Official Analytical Chemists. *Tin in Canned Foods. Atomic Absorption Spectrophotometric Method*, Official Final Action, 1988. AOAC Official Methods of Analysis, 1, (985.16) 270-217, AOAC Arlington, VA, (1990).

Applications:

The method appears satisfactory for both surveys and regulatory purposes related to higher tin levels in all types of foods. It is not sufficiently sensitive for natural tin levels in uncanned foods.

5. Multiple Elements in Foodstuffs

Reference:

Metals. Determination by Atomic Absorption Spectrophotometry in Foodstuffs, Nordic Committee on Food Analysis. No. 139, 1991.

Applications:

Without changes, the method as it applies to Zn, Fe and higher levels of Cu appears satisfactory for screening, surveys and regulatory applications. The method may be used for both screening and routine survey work for Pb, Cd, Cr and Ni. If poorer precision is no problem, the method appears acceptable for regulatory purposes for Ni.

The method can be used for regulatory purposes for Cr, Pb and Cd provided matrix effects are determined and provided Cr, Pb and Cd losses can be tolerated during dry ashing, i.e., a negative bias, is acceptable. Estimates from the collaborative study of the detection limits obtained for lead and cadmium are the best achieved to date for these elements.

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

Annex 1: Methods of Analysis Considered by the CCMAS for Endorsement (CX/MAS 94/10)

Annex 2: Methods of Analysis for Special Foods Considered by the CCMAS for Endorsement

Annex 3: Methods of Analysis Considered by the CCMAS for Endorsement (CX/MAS 94/12)

Notes on the Annexes.

*** Endorsed by the Nineteenth Session of the CCMAS**

METHODS OF ANALYSIS CONSIDERED BY CCMAS FOR ENDORSEMENT (CX/MAS 94/10)

ANNEX I

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
138	Bouillons and consommés 117-1981	Sodium chloride < 12.5 g/L	AIIBP Method No 2/4	Volhard titrimetry	III	TE *
536	Canned corned beef 88-1991	Lead 1 mg/kg	AOAC (1990) 972.25	Atomic absorption	II	E *
539	Canned corned beef 88-1991	Nitrite, potassium and/or sodium Salts 50 mg/kg	ISO 2918:1975	Colorimetry	IV	E *
558	Canned corned beef 88-1991	Nitrite, potassium and/or sodium salts 50 mg/kg	AOAC (1990) 973.31	Colorimetry	II	E *
538	Canned corned beef 88-1991	Tin (Products in other containers) 50 mg/kg	AOAC (1990) 985.16	Atomic absorption	II	E *
537	Canned corned beef 88-1991	Tin (products in tinplate containers) 200 mg/kg	AOAC (1990) 985.16	Atomic absorption	II	E *
554	Cooked cured chopped meat 98-1991	Fat 25-30 %	ISO 1443:1973	Extraction/gravimetry	I	E *
556	Cooked cured chopped meat 98-1991	Lead 0.5mg/kg	AOAC (1990) 972.25	Atomic absorption	II	E *
555	Cooked cured chopped meat 98-1991	Nitrite 125-200 mg/kg	ISO 2918:1975	Colorimetry	IV	E *
559	Cooked cured chopped meat 98-1991	Nitrite 125-200 mg/kg	AOAC (1990) 973.31	Colorimetry	II	E *
557	Cooked cured chopped meat 98-1991	Tin 50-200 mg/kg	AOAC (1990) 985.16	Atomic absorption	II	E *

METHODS OF ANALYSIS CONSIDERED BY CCMAS FOR ENDORSEMENT (CX/MAS 94/10)

ANNEX I

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
546	Cooked cured ham 96-1991	Fat	ISO 1443:1973	Extraction/gravimetry	I	E *
547	Cooked cured ham 96-1991	Lead 0.5 mg/kg	AOAC (1990) 972.25	Atomic absorption	II	E *
545	Cooked cured ham 96-1991	Nitrite 125-200 mg/kg	ISO 2918:1975	Colorimetry	IV	E *
560	Cooked cured ham 96-1991	Nitrite 125-200 mg/kg	AOAC (1990) 973.31	Colorimetry	II	E *
544	Cooked cured ham 96-1991	Protein (conversion factor 6.25) (Calculation of protein on fat free basis) 16.5-18.0%	ISO 937:1978	Kjeldahl digestion	II	E *
548	Cooked cured ham 96-1991	Tin 50-200 mg/kg	AOAC (1990) 985.16	Atomic absorption	II	E *
550	Cooked cured pork shoulder 97-1991	Fat	ISO 1443:1973	Extraction/gravimetry	I	E *
552	Cooked cured pork shoulder 97-1991	Lead 0.5 mg/kg	AOAC (1990) 972.25	Atomic absorption	II	E *
551	Cooked cured pork shoulder 97-1991	Nitrite 125-200 mg/kg	ISO 2918:1975	Colorimetry	IV	E *
561	Cooked cured pork shoulder 97-1991	Nitrite 125-200 mg/kg	AOAC (1990) 973.31	Colorimetry	II	E *
549	Cooked cured pork shoulder 97-1991	Protein (Calculation of protein on fat free basis) 16.0-17.5%	ISO 937:1978	Kjeldahl digestion	II	E *

METHODS OF ANALYSIS CONSIDERED BY CCMAS FOR ENDORSEMENT (CX/MAS 94/10)

ANNEX I

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
553	Cooked cured pork shoulder 97-1991	Tin 50-200mg/kg	AOAC (1990) 985.16	Atomic absorption	II	E *
267	Fruit juices 990	Ethanol < 2 - 5 g/kg	TBD proposed IFJU Method No 53, 1983, AOAC method or Determination of ethanol in fruit juices (in press)	Enzymatic or gas chromatography	II	
509	Grated desiccated coconut 177-1991	Granularity Extra fine, fine and medium	ISO 2591-1:1988 Test Sieving According to British Standard Mesh Nominal Test Sieves: BS410-1986	Sieving	I	TE
540	Luncheon meat 89-1991	Fat < 35% in products with binder < 30% in products without binder	ISO 1443:1973	Extraction/gravimetry	I	E *
542	Luncheon meat 89-1991	Lead 0.5mg/kg	AOAC (1990) 972.25	Atomic absorption	II	E *
562	Luncheon meat 89-1991	Nitrite, potassium and/or sodium salt 125-200 mg/kg	AOAC (1990) 973.31	Colorimetry	II	E *
541	Luncheon meat 89-1991	Nitrite, potassium and/or sodium salts 125-200 mg/kg	ISO 2918:1975	Colorimetry	IV	E *
543	Luncheon meat 89-1991	Tin 50-200mg/kg	AOAC (1990) 985.16	Atomic absorption	II	E *
489	Natural mineral waters 108-1981	Total cyanide not more than 0.01 mg/l, calculated as CN-	ISO 6703-1:1984	Titrimetry	II	TE

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type Status
577	Foods with low sodium content (including salt substitutes) 53-1981	Ammonium (salt substitutes)	TBD		
576	Foods with low sodium content (including salt substitutes) 53-1981	Calcium and magnesium (salt substitute)	TBD		
255	Foods with low sodium content (including salt substitutes) 053-1981	Choline (salt substitutes) < 3 % (m/m)	TBD		
578	Foods with low sodium content (including salt substitutes) 53-1981	Phosphorus (salt substitutes)	TBD		
579	Gluten-free foods 118-1981	Gluten	TBD		
583	Guidelines for nutrition labelling CAC/GL 2-1985	Organic acids	TBD		
580	Guidelines for nutrition labelling CAC/GL 2-1985	Polyunsaturated fat	TBD		
581	Guidelines for nutrition labelling CAC/GL 2-1985	saturated fat	TBD		
582	Guidelines for nutrition labelling CAC/GL 2-1985	Sugars	TBD		
573	Infant formula and follow-up formula 72-1981 & 156-1987	Biotin 1.5 µg/ 100 kcal	TBD		
575	Infant formula and follow-up formula 72-1981 & 156-1987	Carbohydrate (direct)	TBD		

METHODS OF ANALYSIS FOR FOODS FOR SPECIAL DIETARY USES (ALINORM 93/26, Appendix IV)

ANNEX II

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
574	Infant formula and follow-up formula 72-1981 & 156-1987	Choline 7 mg/100 kcal	TBD			
563	Infant formula and follow-up formula 72-1981 & 156-1987	Iodine (milk based formula) 5 µg/100 kcal 150 µg/100 g	AOAC 992.24	Ion-selective potentiometry	II	E *
565	Infant formula and follow-up formula 72-1981 & 156-1987	Pantothenic acid >300µg/100kcal	AOAC 992.07	Microbiological turbidimetry	II	E *
566	Infant formula and follow-up formula 72-1981 & 156-1987	Pantothenic acid >300 µg/kcal	The Analyst 89 (1964):1, 3-6, ibid. 232 US Dept Agr., Agr. Handbook 97 (1965)	Microbioassay	IV	E *
531	Infant formula and follow-up formula 72-1981 & 156-1987	Total dietary fiber	AOAC (1990) 991.43	Enzymatic digestion/ gravimetry	I	E
569	Infant formula and follow-up formula 72-1981 & 156-1981	Vitamin A 75-225 µg/100 kcal [400-1200 µg/100 g]	AOAC 974.29	Colorimetry	IV	E *
568	Infant formula and follow-up formula 72-1981 & 156-1987	Vitamin A (retinol isomers) 75-225 µg/100 kcal [400-1200 µg/100 g]	AOAC 992.04	Liquid chromatography	II	E *
567	Infant formula and follow-up formula 72-1981 & 156-1987	Vitamin A (retinol) 75-225 µg/100 kcal [400-1200 µg/100 g]	AOAC 992.06	Liquid chromatography	II	E *
572	Infant formula and follow-up formula 72-1981 & 156-1987	Vitamin K1 >4 µg/100kcal	AOAC 992.27	Liquid chromatography	II	E *

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
564	Special foods 980	Loss on drying (milk based)	AOAC 925.23 IDF 21B (1987) ISO 6731 (1989)	Gravimetry	I	E *
585	Special foods 980	Vitamin D (D2) [40-100 i.u./100 kcal]	TBD			
570	Special foods 980	Vitamin D (D3, milk based infant formula) [40-100 i.u./kcal]	AOAC 992.26	Liquid chromatography	II	E *
243	Special foods 980	Vitamin E > 0.7 i.u./100 kcal	AOAC (1990) 971.30	Colorimetry	IV	E
571	Special foods 980	Vitamin E (milk based infant formula)	AOAC 992.03	Liquid chromatography	II	E *

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
140	Bouillons and consommés 117-1981	Tin < 150 mg/kg	AOAC 985.16	Atomic absorption	II	E*
199	Cocoa butter confectionery 147-1985	Sugars < 55 %	AOAC 980.13	Liquid chromatography	II	E*
178	Cocoa powders (cocoa) and dry cocoa-sugar mixtures 105-1981	Ash insoluble in HCl in cocoa nib, cocoa mass and cocoa press cake No limit	AOAC 972.15	Gravimetry	I	E*
181	Cocoa powders (cocoa) and dry cocoa-sugar mixtures 105-1981	Cocoa powder > 20 - 25 %	TBD			
182	Cocoa powders (cocoa) and dry cocoa-sugar mixtures 105-1981	Fat reduced cocoa powder 8 - 20 % (m/m)	TBD			
183	Cocoa powders (cocoa) and dry cocoa-sugar mixtures 105-1981	Sugars	AOAC 980.13	Liquid chromatography	II	E*
191	Composite and filled chocolate 142-1983	Percentage of chocolate in composite chocolate > 40 %	TBD			
435	Durum wheat semolina and durum wheat flour 178-1991	Particle size	Recommended: A.Menger; W.Seibel. Untersuchung von Teigwarenrohstoffen Durchführung der Sieb analyse bei Geteidegries und-dunst. Arbeitsgemeinschaft Getreideforschung-ev Merkblatt N0 57, 2 Auf (1992)	Sieving	I	TE*
508	Edible cassava flour 176-1991	Total cyanide < 10 mg/kg	TBD			

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
361	Edible ices 137-1981	Foreign fat in milk fat	TBD			
255	Foods with low-sodium content (including salt substitutes) 053-1981	Choline < 3 % (m/m)	TBD			
268	Fruit juices 990	Honey No limit	TBD			
269	Fruit juices 990	Minimum content of fruit ingredient	TBD			
380	Gari (African regional standard) 151-1985	Acidity as lactic acid 0.6 - 1 % (m/m)	TBD			
379	Gari (African regional standard) 151-1985	Cyanogenic glucosides and hydrocyanic acid < 2 mg/kg as HCN	TBD			
258	Gluten-free foods 118-1981	Nitrogen No limit	TBD			
529	Guideline levels for mercury in fish 2007	methyl mercury 0.5 mg/kg (all fish except predatory fish) 1 mg/kg (predatory fish)	AOAC 988.11	Atomic absorption	II	E *
50	Pickled cucumbers 115-1981	Drained weight 53 - 57 %	AOAC 968.30	Gravimetry	I	E *
118	Powdered dextrose (Icing dextrose) 054-1981	Reducing sugar > 99.5 %	TBD Proposed AOAC 923.09	Volumetry		
117	Powdered dextrose (Icing dextrose) 054-1981	Starch < 5 %	TBD Proposed AOAC925.50	Gravimetry		

METHODS OF ANALYSIS REQUIRED BY CODEX TO BE DEVELOPED AND/OR VALIDATED (CX/MAS 94/12)

ANNEX III

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
120	Powdered dextrose (Icing dextrose) 054-1981	Sulphated ash < 0.25 % (m/m)	TBD Proposed AOAC 900.02	Gravimetry		
119	Powdered dextrose (Icing dextrose) 054-1981	Total solids > 90.0 %, > 98.0 %	TBD Proposed AOAC 925.45	Gravimetry		
80	Powdered sugar (Icing sugar) 005-1981	Anti-caking agents < 1.5 %	TBD			
78	Powdered sugar (Icing sugar) 005-1981	Colour < 60 ICUMSA units	TBD			
76	Powdered sugar (Icing sugar) 005-1981	Conductivity ash < 0.04 %	TBD			
75	Powdered sugar (Icing sugar) 005-1981	Invert sugar < 0.04 %	TBD Proposed AOAC 923.09	Volumetry		
74	Powdered sugar (Icing sugar) 005-1981	Polarization > 99.7 degrees S	TBD Proposed AOAC 925.46	Polarimetry		
73	Powdered sugar (Icing sugar) 005-1981	Starch < 5 %	TBD Proposed AOAC 925.50	Gravimetry		
151	Quick frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh 165-1989	Net contents of products covered by glaze	AOAC 963.18	Gravimetry	I	E*
159	Quick frozen fish sticks (fish fingers) and fish portions - Breaded or in batter 166-1989	Fish/mince proportions	AOAC 988.09	Gravimetry	I	E*
213	Quick frozen french fried potatoes 114-1981	Free fatty acid content in fat or oil < 1.5 % (m/m)	TBD			

Serial No.	Commodity Standard No.	Provision Level	Method	Principle	Type	Status
212	Quick frozen french fried potatoes 114-1981	Moisture < 76 % (m/m)	AOAC 984.25	Gravimetry/convection oven	I	E *
208	Quick frozen fruits and vegetables: Whole kernel corn and Corn-on-the-cob 132-1981	Alcohol insoluble solids	TBD			
242	Special foods 980	Biotin > 1.5 ug/100 kcal	TBD			
226	Special foods 980	Choline > 7 mg/100 kcal	TBD			
250	Special foods 980	Iodine > 5 ug/100 kcal	TBD			
241	Special foods 980	Vitamin K1 > 4 ug/100 kcal	TBD			
350	Specified animal or mixed animal and vegetable fat products 158-1987	Lead < 0.1 mg/kg	AOAC 994.02 IUPAC 2.623 ISO 12193	Atomic absorption/graphite furnace	II	E *
336	Specified vegetable fat products 157-1987	Lead < 0.1 mg/kg	AOAC 994.02 IUPAC 2.632 ISO 12193	Atomic absorption/graphite furnace	II	E *
443	Wheat gluten 163-1987	Heavy metals	TBD Proposed AOAC 986.15	Atomic absorption		

NOTES ON THE ANNEXES

Methods of Analysis Considered by the CCMAS for Endorsement in the Document CX/MAS 94/10 (Annex 1)

- 138 The Working Group agreed to retain the status of the method for lack of additional information.
- 536 The Working Group replaced method AOAC (1990) 934.07 with a general method for metals, i.e., AOAC (1990) 972.25. The replacement method was endorsed as a Type II method.
- 539 The Working Group classified the ISO method as Type IV as no collaborative studies have been performed. The WG however considered another method AOAC (1990) 973.31 which is equivalent in principle to the ISO method and endorsed it as Type II.
- 555 Same as for 539.
- 554 The Working Group endorsed the method as Type I because it is a traditional method with no intention to be collaboratively studied.
- 556 Same as for 536.
- 544 The Working Group agreed that if the protein conversion factor is specified in the method then the method is classified as a Type I method, but if in the commodity standard independent of the method then it should be classified as a Type II method, or Type IV if no collaborative studies were performed.
- On the basis of this position, the ISO method was classified as Type II.
- The Working Group requested the Codex Secretariat to take the above position into consideration in the review of Volume 13 in order to assure consistency in classification.
- 545 Same as for 539.
- 546 Same as for 554. The Working Group moved the comment in brackets i.e. (Calculation of proteins on fat free basis) to 544.
- 547 Same as for 536.
- 553 The Secretariat corrected the commodity to read Cooked Cured Pork Shoulder.
- 549 Same as for 544. The factor of 6.25 for the conversion of nitrogen to proteins was in the commodity standard. The error in the ISO Standard reflected in the Annex was corrected i.e. ISO 1443:1973 was amended to read ISO 937:1978.
- 550 Same as for 554.
- 551 Same as for 539.
- 561 Same as for 539.
- 552 Same as for 536.

267 The Working Group was informed of three methods:

- AOAC (1990) 983.13 Method for wine but tested in fruit juices;
- an enzymic procedure by the Journal of the Association of Public Analysts which is collaboratively studied and is in press; and
- an IFJU Method No. 53, 1983, that would be published by CEN in 1995.

On the basis of this information the Working Group decided to postpone consideration of the method to be endorsed until the next meeting to allow for the publication and evaluation of these three methods.

509 The Working Group maintained the status because ISO could not advise on the equivalence of the ISO and BSI references to test sieves. The Secretariat was requested to contact the Commodity Committee and BSI.

540 Same as for 554.

541 Same as for 539.

542 Same as for 536.

562 Same as for 539.

489 The Working Group reiterated its earlier stand on this method, i.e. "Information from ISO indicates that the ISO method is applicable at levels as low as 10 microgram per litre but samples with such a low concentration of cyanide cannot be tested in a collaborative study for rapid decomposition of cyanide in the samples.

The Working Group therefore postponed action and indicated that another potentiometric method was being collaboratively studied. The method would be provided to the Codex Secretariat by ISO/AFNOR.

Methods of Analysis for Special Foods Considered by the CCMAS for Endorsement (Annex 2)

General Comment:

The Working Group decided not to consider those provisions in the Annex the status of which are stated as "To be developed".

531 Dietary fiber - The Working Group endorsed the AOAC method as a Type I method.

563 Iodine - The Working Group endorsed the AOAC method which has only been studied for Infant formula and Follow-up formula as Type II.

564 Loss on drying - The Working Group endorsed AOAC (1990) 925.23 as a Type I method for milk based products only. The Working Group also recommended that the Commodity Committee consider the general method AOAC (1990) 934.01 (for loss on drying) and identical methods citations are ISO 6731:1989 and IDF Standard 21B:1987.

Pantothenic acid - The Working Group considered the two methods AOAC (1990) 992.07 and The Analyst 89 (1964) (1) reference as Types II and IV respectively, i.e.

565 AOAC (1990) 992.27, - Type II, and

566 The Analyst 89 (1964) (1); 3-6; *ibid*, 232 - Type IV.

569 Vitamin A - The Working Group deleted method AOAC (1990) 941.15 as it has already been endorsed and it was replaced with AOAC (1990) 974.29 (colorimetric), and classified this method as a Type IV.

The methods AOAC (1990) 992.06 Retinol and AOAC (1990) 992.04 Retinol-isomers were endorsed as Type II methods.

567 AOAC (1990) 992.06, and

568 AOAC (1990) 992.04

570 Vitamin D - While the status of the method for Vitamin D₂ is "to be developed", that for Vitamin D₃, i.e. AOAC (1990) 992.26 was endorsed as a Type II method.

571 Vitamin E - The Working Group classified method AOAC (1990) 971.30 as Type IV and endorsed AOAC (1990) 992.03 for milk-based infant formula as Type II.

581-584 Methods were to be developed for these provisions by the Commodity Committee.

Methods of Analysis Considered by the CCMAS for Endorsement (CX/MAS 94/12) (Annex 3)

140 AOAC (1990) 980.19 is a surplus method and the Working Group proposed the endorsement of another method AOAC (1990) 985.16 as a Type II method.

The Secretariat was requested to inform the Chairman of the respective Commodity Committee of the changes made.

178 The Working Group endorsed the method as Type I but requested the Secretariat to determine if a method is required when no limits are prescribed in the standard.

435 The Working Group temporarily endorsed the method as Type I. The WG recommended that the Commodity Committee may wish to consider other methods such as ICC Standard 127 and another one which was developed by AFNOR. The Representative of AFNOR was to provide the number of the method to the Secretariat for the information of the CCMAS.

529 The Working Group believed that the Commission had decided to redefine this provision as Methyl mercury. This being so the WG endorsed method AOAC (1990) 988.11 as a Type II method.

117-120
73-78
80 ICUMSA representative indicated that the respective Codex Committee has reviewed the standards for various sugars and had incorporated individual standards for sugars into more general standards. The reviewed standards were circulated (CL 1993/14) and the Commodity Committee is reviewing the comments received.

The Working Group decided to postpone the consideration of these methods and await further development from the Commodity Committee.

159 The Working Group endorsed method AOAC (1990) 988.09 as Type I. If the standard is designed to determine the percentage of fish and percentage of fish mince.

If the standard specifies the percentage of fish and fish mince as a proportion of the total breaded product then the Working Group endorses AOAC 971.13 as Type I. The Secretariat was asked to clarify the specification or refer it back to the Commodity Committee for a clearer recommendation.

208 The Delegation of the United Kingdom informed that the proposed methods AOAC (1990) 971.29 and ISO 8129/1 were not applicable. The WG did not endorse the methods.

350 The Working Group proposed that the Commodity Committee may wish to consider other methods such as IUPAC 2.623, AOAC (1990) 994.02 and ISO 12193. If the Commodity Committee agrees the new methods were endorsed as Type II methods.

336 Same as 350.

443 The Working Group requested the Secretariat to explore the requirements under heavy metals which will determine if this method or sulfide precipitation method is more applicable to the standard.

**RECOMMENDED PROTOCOLS FOR THE DESIGN, CONDUCT AND INTERPRETATION
OF COLLABORATIVE STUDIES AND PROFICIENCY TESTING OF (CHEMICAL)
ANALYTICAL LABORATORIES
(At Step 8 of the Procedure)**

The following protocols are recommended for the design, conduct and interpretation of collaborative studies and for the proficiency testing of (chemical) analytical laboratories for Codex purposes:

Protocol for the Design, Conduct and Interpretation of Collaborative Studies.
International Union of Pure and Applied Chemistry, Pure and Appl. Chem. (in preparation)¹.

Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories.
Pure and Appl. Chem. 65 (9) 2123-2144 (1993) and J. AOAC Intl. 76 (4) 926-940 (1993).

¹ This Protocol has been recommended by the Committee subject to its final adoption by IUPAC. IUPAC has been invited to confirm to the Executive Committee that no substantive changes have been made to the Protocol in the final stages of adoption.

REPORT OF THE TENTH INTER-AGENCY MEETING (IAM)
Budapest, 18 March 1994

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OPENING OF THE MEETING

1. The Tenth Inter-Agency Meeting was opened by Mr. Sándor Vass, Director of International Relations at the Hungarian Office for Standardization (MSZH) . After welcoming the Representatives of the various International Organizations (See Annex 1) , Mr. Vass said that MSZH was pleased to host the IAM, which also demonstrated the involvement of his Office in international work. He also stressed the interest of Hungary in food industry, research and technology. Mr. Vass was particularly pleased to note that the Hungarian Government had recently issued a decree on national standardization. In conformity with most industrialized countries, Hungarian National Standards are now no longer mandatory but voluntary.

ELECTION OF CHAIRMAN

2. Upon the proposal of Mr. Vass, Mr. G. Castan (Technical Advisor to AFNOR) was elected Chairman. Mr. Castan thanked the IAM for the renewed confidence and MSZH for hosting the IAM. He was also pleased to note the recent development from mandatory to voluntary standardization in Hungary.

ADOPTION OF THE AGENDA

3. The Agenda was adopted without amendment. The Chairman drew particular attention to the item dealing with a Harmonized Protocol on Internal Laboratory Quality Control which had been added to the agenda upon the request by AOAC International.

REVIEW OF MEMBERSHIP TO THE IAM

4. The Secretary was pleased to note that most of the Member Organizations of the IAM had regularly attended the IAM or submitted written activity reports. However, as the International Society of Dietetic including all Infant and Young Children Food Industries (ISDI) had neither attended the IAM nor made any written contribution so far, it was decided to delete this organization from the list of Members.

5. The IAM also noted that the International Federation of Fruit Juice Producers (IFJU), the International Office for Cocoa, Chocolate and Sugar Confectionery (IOCCC) and the International Union of Microbiological Societies (IUMS) had not attended the IAM over the past six years and not submitted any activity reports. Therefore, the Secretary was invited to write to these organizations in order to ascertain whether or not they wish to remain Members of the IAM.

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

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

- The report of the Ninth IAM has been circulated as Appendix IV to the Report of the 18th Session of the Codex Committee on Methods of Analysis and Sampling (ALINORM 93/12).
- The Codex Secretariat had presented a list of methods for Codex Standards which were to be considered by the CCMAS Working Group on Endorsement of Methods on 19 March 1994.
- The Codex Secretariat had prepared a pre-publication of Volume 13 of the Codex Alimentarius. Copies were made available to interested Representatives or Organizations attending the IAM for comment and updating.

- The Codex Secretariat has prepared a list of provisions contained in Codex Standards and Guidelines which need the establishment of a method of analysis to be developed. This list had been circulated to the IAM. (It is the Annex to the Working Paper CX/MAS 94/12).

Exchange of Information on Collaborative Studies

7. The Representative of AOAC International said that information on collaborative studies planned or carried out by various organizations was regularly published in "The Referee". She invited interested organizations to submit all relevant information in the format agreed by the IAM and circulated by the Secretary in February 1989 (Annex 2).

8. The Representative of AOAC International also drew attention to the "Montreal Protocol" that had been adopted in September 1993. The purpose of this Protocol was to prohibit the use of solvents and substances depleting the ozone layer.

Status of Joint Work by AOAC International, IUPAC and ISO towards a Harmonized Protocol on the Presentation and Organization of Proficiency Testing Schemes

9. The Representative of AOAC International gave an account of the history and the current status of work on the Harmonized Protocol for Collaborative Studies which had been carried out by AOAC/IUPAC/ISO (Annex 3). It was noted that the Protocol had also been adopted by the EU under the Food Control Directive.

10. In response to a query by the Representative of ISO, The Representative of AOAC International confirmed that several experts from ISO/TC 69 "Applications of Statistical Methods" had participated in the elaboration of the Protocol. The IAM also noted that Working Group 5 of Sub-committee ISO/TC 69/SC 6 "Measurement Methods and Results" was currently preparing a draft on decision limit, detection limit, and capacity of detection that was in line with the Harmonized Protocol (ISO/WD 11843, Parts 1 and 2).

Harmonized Protocol on Internal Laboratory Quality Control

11. The Representative of AOAC International informed the IAM that the Third Draft of the Harmonized Protocol developed by AOAC/IUPAC/ISO had been circulated for comment. He added that the Protocol would also be circulated to the Nineteenth Session of CCMAS as a conference room document, and that the final meeting of the Working Group that had prepared the Protocol was planned in May 1994.

12. During the ensuing discussion, the question arose whether or not the present Terms of Reference of the IAM included matters relating to quality control in laboratories dealing with chemical analysis. As no agreement could be reached it was decided to await the outcome of discussions at the Session of CCMAS which might result in a mandate to the IAM to deal with this subject. Should this be the case, this topic would be included in the Agenda of the next IAM.

13. The Representative of ISO, noting that an item dealing with interpretation of results of collaborative studies was on the Agenda of CCMAS, drew attention to the fact that ISO 5725:1986 was currently being revised to take into account the problems that arose during the application of the first edition. She pointed out that the future second edition of this Standard had been approved by all National Bodies of ISO and, consequently represented a real international harmonization. As the Harmonized Protocol prepared by AOAC/IUPAC/ISO was based on the first edition of ISO 5725, however, the Representative of ISO was

concerned that it was significantly different from the new edition of the ISO Standard. For example, she mentioned that the limits of outliers in the new edition of ISO 5725:1986 and the Harmonized Protocol were different. In this context, the IAM noted that EUROCHEM was currently examining both ISO 5725:1986 and the future edition of the Standard. The Representative of AOAC International offered to make available copies of the EUROCHEM document to interested organizations.

14. The Representative of NMKL, supported by several other Representatives, said that the number of laboratories participating in a collaborative study was more important than the treatment of outliers. She also mentioned that, contrary to the Summary of the Report of the 18th Session of CCMAS, the Harmonized Protocol for Interlaboratory Collaborative Studies had not been approved but would rather be circulated for comment and further discussion.

METHODS OF ANALYSIS AND SAMPLING REQUIRED BY THE CAC

15. The Chairman invited the representatives of organizations present to report on activities relevant to the IAM and also drew attention to a few written activity reports submitted by AOAC International, EOQ, IAEA, FAO, ICC, IDF, ISO, IUPAC, NMKL and OIV.

Methods of Sampling

16. The Representative of the Codex Secretariat informed the IAM that a FAO Technical Consultation on Sampling Plans for Aflatoxin Analysis in Peanuts and Corn was held in Rome from 3 to 6 May 1993. The Consultation was convened at the recommendation of the Codex Committee on Food Additives and Contaminants to provide member countries with guidance on sampling procedures of aflatoxin contaminated peanut and corn commodities. The Consultation emphasized the importance of aflatoxin contamination and its effects on international trade. It also recognized the crucial importance of sample size on the acceptance or rejection of an aflatoxin contaminated lot. A sampling plan for peanuts and corn was developed and evaluated, based on two sample sizes, thin layer chromatography (as analytical method) and five guidelines levels of 5, 10, 15, 20 and 30 u/kg which reflect the levels most commonly found in national food legislation. The Consultation also discussed practical issues associated with the application of sampling and sample preparation procedures.

17. The Representative of the Codex Secretariat added that, following on the Commission's endorsement of the elaboration of Draft Codex Guidelines on Sampling, the Codex Secretariat wished to inform the IAM that the draft of the Guidelines prepared by a consultant would be considered at the forthcoming session of the CCMAS.

Pesticide Residues and Residues of Veterinary Drugs

18. The Representative of the Codex Secretariat, referring to Volume 2 of the Codex Alimentarius, informed the IAM that this Volume contained in addition to MRLPs:

4. Analysis of Pesticide Residues
 - 4.1 Portion of commodities to which Codex MRLPs apply and which is analyzed;
 - 4.2 Guidelines on Good Laboratory Practice in pesticide residue analysis;
 - 4.3 Recommended methods of analysis for pesticide residues.

Supplement 1 to Volume 2 was also published and it contains (in addition to MRLPs) Guidelines on Good Laboratory Practice in pesticide residue analysis.

19. The IAM was also informed that Volume 3 of the Codex Alimentarius had been published which contains (in addition to MRLVDs) Guidelines for the Establishment of Regulatory Programmes for the Control of Veterinary Drug Residues in Foods. These Guidelines include sampling for the control of veterinary drug residues in:

- i) Meat and poultry products;
- ii) Milk, dairy products, eggs and fish;
- iii) Honey.

The CCPR Working Group on Methods of Analysis and the CCRVDF Working Group on Methods of Analysis and Sampling are actively discussing methods of analysis and sampling.

20. The IAM agreed that whilst these were no specific requests to the IAM or the CCMAS, it would be available to provide advice on any type of methods required by the Codex.

Methods of Analysis for Nutritional Food Labelling

21. The Representative of AOAC International offered to make available to interested organizations a complete list of methods that are still required. It was noted that at present there was no need for action by the IAM in this area.

Food Additives and Contaminants

22. The Representative of NMKL, referring to the recent meeting of CCFAC, informed the IAM that the Canadian Member of the Codex was currently updating a list of methods for the determination of food additives in foods. Accordingly, International Organizations were invited to provide information on suitable validated methods. The Representative of AOAC International circulated copies of the request of the CCFAC and pointed out that any information on methods for food additives, validated or not, should be transmitted to Canada before 1 July 1994.

Microbiology

23. The IAM noted the information in the written reports submitted by NMKL and ISO. It also considered the question of validation of microbiological methods and test kits. The Representative of ISO informed the IAM of relevant activities of the European Committee for Standardization (CEN), which will shortly lead to adoption of four Standards developed by ISO/TC 34/SC 9 "Microbiology" as European Standards.

Nutrition and Foods for Special Dietary Uses

24. The Representative of the Codex Secretariat introduced a list of methods for infant formula required by the Codex and invited Organizations participating in the IAM to submit suitable methods to the Codex Secretariat. The Representative of AOAC International mentioned that the former method for the determination of gliadins in gluten free foods had not been accepted by AOAC and that a new method was therefore required.

Methods of Analysis for Irradiated Foods

25. The Representative of the Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture gave an overview of the activities of his Organization. A comprehensive report on these activities was

presented at the IAM. He emphasized that the same quality and hygiene standards can be applied to conventional foods and irradiated foods and that identification tests (Yes/No tests) were required for irradiated food rather than quantitative methods.

26. The IAM noted that various organizations, including CEN and AOAC International were involved in the development of methods for irradiated foods. It also noted that the work of CEN in this area was mandated by DG III of the EC.

Methods for the Detection and Determination of Radionuclides in Foods

27. Whilst there was no need for action by the IAM in this area, the Representative of the Joint FAO/IAEA Division offered to make available copies of the relevant FAO/IAEA studies, if necessary.

Cocoa Products and Chocolate

28. The IAM noted that the respective Codex Committee had adjourned sine die and that at present these were no requests to the IAM.

Milk and Milk Products and Edible Ices

29. The Representative of IDF gave an account of the current tripartite programme of work on Methods of Sampling and Analysis for Milk and Milk Products and presented a document on this subject. The IAM also noted the relevant information in the activity report submitted by AOAC International. The Representative of the Codex Secretariat informed the IAM that the Codex Committee on Milk and Milk Products is now the body responsible for this area in the Codex structure and that the Codex Committee in Edible Ices had adjourned sine die. Attention was also drawn to the work of CEN on milk and milk products which had established a new technical committee in this field the secretariat of which is held by The Netherlands.

Fats and Oils

30. The Representative of IUPAC introduced the current programme of work of IUPAC Commission VI.3 "Oils, Fats and Derivatives". The IAM reiterated its earlier request to the Codex Secretariat that references to equivalent methods prepared by AOAC International, IUPAC and ISO be included in Codex documents on fats and oils. It also noted the CEN work on methods of analysis for fats, oils and oilseeds, which is mainly based on the Standards developed by ISO/TC 34/SC 11 "Animal and Vegetable Fats and Oils".

Fruit Juices

31. The IAM was informed that ten European Standards will shortly be published by CEN which are based on IFJU methods. The Representative of AOAC International added that her Organization had an active Working Group on Methods for Fruit Juices and also drew attention to the activity report submitted by AOAC International.

Processed Fruits and Vegetables

32. No information had been received from International Organizations. The IAM was informed that the respective Codex Committee had adjourned sine die and that there were no specific request for methods of analysis.

Processed Meat and Poultry Products

33. The respective Codex Committee had adjourned sine die and there were no specific requests for methods of analysis. The relevant work of ISO/TC 34/SC 6 "Meat and Meat Products" was noted.

Fish and Fishery Products

34. The Representative of AOAC International referred to relevant information in the activity report and said that CCFAC was currently considering methyl mercury in fish and fishery products.

Sugars

35. The Representative of ICUMSA informed the IAM that his Organization had published a new book of methods of sugar analysis that are written in ISO format.

Starch Hydrolysis Products

36. The Representative of ISO informed the IAM that CEN was in the course of adopting several Standards developed by ISO/TC 93 "Starch, Derivatives and By-Products" and that the European professional associations were contributing to the CEN work in this area.

Cereals, Pulses and Legumes

37. The IAM noted the work of ICC and ISO/TC 34/SC 4 "Cereals and Pulses". The Representative of ISO added that the method for the determination of impurities in cereals (ISO 11050:1993) required by the CCMAS, had been published in 1993. The Representative of ICC, referring to the activity report which was presented, mentioned the collaboration that existed between his Organization, ISO, CEN and ISTA. He also drew attention to the forthcoming ICC events in the Netherlands and in China and the ICC Congress in 1995.

Mineral Waters

38. The Secretary informed the IAM that some of the methods developed by ISO/TC 147 "Water Quality" were of interest to the Codex and that he had provided relevant information to the Codex Secretariat.

Wines and Spirits

39. The Representative of OIV gave an account of the activities of her Organization during the period 1993/1994. She noted that a collection of methods for alcoholic beverages and aromatic fractions of beverages had been published in January 1993. The methods in this collection are based on the Official French Methods of Analysis for Alcohol and Spirits issued in 1973 which had been revised by OIV experts. They had been published under the responsibility of Professor Alain Bertrand, who is the Secretary of the OIV Sub-Committee on Analytical Methods. The Representative of OIV added that an overview of pesticide residues in fruits, especially in grapes and the authorized limits in different countries had been published in 1994, both in the English and French languages. This overview permits one to compare authorized limits for more than 700 pesticides in 36 countries. She also noted that publication of the English version of a collection of international methods of analysis for wines and musts is planned for October 1994. Furthermore, a working document on the adoption of the Harmonized Protocol on Collaborative Studies proposed by AOAC/IUPAC/ISO is in preparation within OIV. Another document dealing with the analysis

of wine vinegars will be presented at the meeting of the Sub-Committee on Methods of Analysis in March 1994 in Paris.

40. The Representative of the Codex Secretariat indicated that, in view of the importance of international trade in wine and alcoholic beverages and the implementation of the GATT Agreement, the Codex may eventually be interested in methods developed by OIV.

OTHER ACTIVITIES OF INTEREST TO THE IAM

41. The Representative of AOAC International drew attention to the Montreal Protocol mentioned earlier at the IAM and said that substances such as carbon tetrachloride and asbestos should no longer be used in the laboratory. However, it had to be ascertained that comparative tests carried out with methods that use alternative substances give the same results.

42. The IAM was pleased to note that DG III of the EU was apparently in the course of changing its philosophy concerning the implementation of standardized methods in its Directives. Whilst it had been common practice in the EC so far to include the full text of methods in the Directives there are now developments towards inclusion of simple references to the relevant methods..

EXCHANGE OF VIEWS ON PROPRIETARY LABORATORY TECHNIQUES VERSUS TRADITIONAL METHODOLOGY

43. The Representative of AOAC International introduced a document on this subject which had been prepared by a Member of his Organization. The object of this document was to establish internationally agreed general principles of validation of proprietary technology based laboratory methods and to provide a design for validation for third-party performance verification.

44. The Representative of IDF distributed a document on the same subject and informed the IAM of the policy of his Organization which allows the publication of IDF Standards that are based on proprietary laboratory techniques.

45. The Representative of ISO mentioned a similar work item of ISO/TC 34/SC 9 "Microbiology" and a current project called MICROVAL, which is supported by EU within the framework of EUREKA. The object of this project is to establish a European validation mechanism acceptable to all interested European parties for the validation of alternative microbiological methods (mainly test kits) against a (official) reference method. With regard to the document presented by AOAC International, she noted that no reference at all was made to the organization of the laboratory itself.

46. The Representative of AOAC International informed the IAM of the relevant activities of the Research Institute of her Organization. Supported by the Representative of NMKL, the Representative pointed out that the approach used by AOAC International was to check the manufacturer's claims. The approach by MICROVAL was that different routine methods were validated against one single reference method which needs to be precisely defined. The Research Institute of AOAC International usually approved test kits for a period of one year with the option of annual renewal, since experience had shown that their composition and performance were varying.

47. As the IAM was unable at this stage to agree on one of the two different approaches being considered, it was suggested that a scientific conference or symposium be organized on this topic, with participation from all interested organizations. The IAM also noted that AOAC International was in the course of preparing a Conference on Proprietary Laboratory Techniques and that CCMAS would be

considering this topic at its forthcoming session. The IAM therefore agreed to await the outcome of the discussions of CCMAS and to pursue consideration of proprietary laboratory techniques at the next IAM. The Chairman invited all interested organizations and in particular AOAC International, CEN, IUPAC, ISO and NMKL, to prepare a document on the possibilities of the different approaches to the validation of proprietary laboratory techniques for the next IAM.

OWNERSHIP RIGHT FOR METHODS AND COPYRIGHT

48. In the absence of a document and in view of the current discussions on questions of copyright between ISO and the American National Standards Institute (ANSI), it was decided to defer consideration of this topic to the next IAM. The Representative of AOAC International was invited to prepare a document on this issue for the next IAM.

ANY OTHER BUSINESS

49. The Representative of EOQ invited interested organizations and individuals to attend the 9th World Congress on Food Science and Technology, to be held in Budapest from 30 July to 4 August 1995 and distributed copies of the programme of the Congress. Drawing particular attention to the Session on Food Safety and Quality Management, he said that some of the topics to be discussed at this Session were of interest to Member Organization of the IAM. He would therefore appreciate any contribution AOAC International or other organizations might wish to make to the Congress.

50. The Representative of the Joint FAO/IAEA Division informed the IAM that, during the period from 20 to 24 June 1994, the Department of Agriculture of Northern Ireland together with FAO/IAEA will be organizing two events in Belfast which are of interest to Member Organization of the IAM. He offered to make available the relevant documents on request.

DATE OF THE NEXT MEETING

51. As the IAM is closely associated with the Sessions of CCMAS, it was agreed to await the decision of CCMAS concerning the dates of its next Session.

52. Before closing the Tenth IAM, the Chairman thanked the participants for their contributions, the Hungarian hosts for the arrangements made and the hospitality, the Interpreter and the Secretary.

53. On behalf of all participants, Ms. Lauwaars (AOAC International) thanked the Chairman for his able conduct of the IAM.

10th INTER-AGENCY MEETING

18 March 1994, Budapest

List of Participants

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RECOMMENDATIONS OF THE INTER-AGENCY MEETING REGARDING COOPERATION IN THE EXCHANGE OF INFORMATION

In an effort to promote cooperation between International Organizations aimed at responding to the needs of the Codex Alimentarius Commission for the development of suitable methods of analysis and sampling of food products, the Inter-Agency Meeting agreed that:

- (a) There is a need for a more extensive exchange of information and a greater degree of cooperation between the bodies, in relation to the work they are about to undertake.
- (b) Exchange of information between Organizations concerning work on the development and validation of methods is the responsibility of the Secretariats of the Organizations.
- (c) There is a need, therefore, to exchange limits of work in hand, with up-dating information as appropriate.
- (d) Exchange of invitations between bodies to send observers to each others' meetings is necessary. The observers should be properly briefed on the matters of interest and other relevant questions and should be expected to report back to their sponsors. The names of observers should always be sent in advance to the organizers. Registration fee for participation in working sessions should be waived.
- (e) Advance notice should be given of items of work to be undertaken, inter-laboratory and other studies planned with invitations to participate where appropriate. Reports on results achieved should be issued. Advance notices should include a minimum amount of information such as indicated as follows in case of inter-laboratory collaborative studies.

Suggested Standard Information for Announcement of Inter-Laboratory Collaborative Studies

The announcement should mention the name of the method (reference when published) and include all information necessary for potential participants including the following:

- analyte: criterion and parameter to be determined;
- matrix: the product or products on which the determination is to be performed;
- principle of method: including treatment of test portion (digestion, extraction), separation (purification, e.g. chromatography) and quantification (e.g. NMR, spectrometry, detector system);
- organizing body:
- contact point: name of person and address (telephone, telex, telefax);

- schedule: expected commencement of study (month, year)
and expected date of report;
- participation: specification whether participation is open or restricted.

The announcement should be as brief as possible and should be made in good time before the year of the study. Organizations should communicate news of studies at least once each year.

- (f) Organizations should send announcements to the European Representative of AOAC International at the following address:

Mrs. Margareet Lauwaars
European Representative of
AOAC International
P.O. Box 153
NL-6720 AD BENNEKOM
The Netherlands

The European Representative of AOAC International will subsequently circulate these announcements to all interested Organizations, indicating the name of the member of the Inter-Agency Meeting to whose attention the announcements should be brought.

- (g) Where circumstances so require, participation by specialists in each other's working groups, groups of experts, etc. should be made possible.
- (h) A comprehensive list of methods of analysis and sampling required by the Codex Alimentarius Commission should be prepared by the Codex Secretariat and kept up-to-date. The criteria included in such a list requiring the development and/or validation of methods of analysis and sampling should be appropriate expert groups within the Codex system in order to see whether the methods are really required and to indicate the exact purpose which the method are intended to serve.

TENTH INTER-AGENCY MEETING

Status of IUPAC/ISO/AOAC Harmonization Activities

IUPAC/ISO/AOAC International Harmonization Symposiums and Workshops have been held as follows, producing the indicated harmonized documents:

- 1978 - Representatives of various organizations met in London in 1978.
- 1981 - The First International Symposium on the Harmonization of Collaborative Analytical Studies was held in Helsinki, Finland.
- 1984 - The Second International Symposium, Harmonization of Collaborative Analytical Studies was held in Washington, DC. Material was produced at this symposium for use in the publication of later documents.
- 1987 - An IUPAC Workshop on Harmonization of Collaborative Analytical Studies was held in Geneva, producing material leading to the production of "Guidelines for Collaborative Study Procedure to Validate Characteristics of Methods of Analysis". This document was adopted by AOAC and is used as AOAC Guidelines in conducting collaborative studies.
- 1989 - The Third International Symposium on the Harmonization of Quality Assurance Systems in Chemical Analysis was held in Washington, DC. This workshop produced a document - "Harmonized Protocols for the Adoption of Standardized Analytical Methods and for the Presentation of their Performance Characteristics", published in *Pure and Applied Chemistry*, Vol. 62, 1990. This Protocol has not been adopted by AOAC.
- 1991 - The Fourth International Symposium on Harmonization of Quality Assurance Systems in Chemical Analysis was held in Geneva, Switzerland. This Symposium produced a draft of "International Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories".
- 1992 - A workshop to finalize the "International Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories" was held in Delft, The Netherlands. This document has been endorsed by AOAC and published in *AOAC International*, (76) 703-944 (July/August 1993).
- 1993 - The Fifth International Symposium on the Harmonization of International Quality Assurance Schemes for Analytical Laboratories was held in Washington, DC. A draft document "International Harmonized Protocol for the Quality Control of (Chemical) Analytical Laboratories" was produced.
- 1994 - The next meeting, a workshop, will be held in Delft, The Netherlands, May 9-10, 1994, to finalize the document "International Harmonized Protocol for the Quality Control of (Chemical) Analytical Laboratories" drafted at the Washington Symposium in 1993.
- The Sixth International Symposium on the Harmonization of "Total Quality Management in Analytical Laboratories" will be held in Melbourne, Australia, in 1995.

**DEVELOPMENT OF OBJECTIVE CRITERIA FOR ASSESSING THE
COMPETENCE OF TESTING LABORATORIES INVOLVED IN THE
OFFICIAL IMPORT AND EXPORT CONTROL OF FOODS**

Summary

This paper deals with objective criteria for assessing the competence of testing laboratories involved in the official import and export control of foods. The internationally recognized form of assessment is accreditation of the laboratory for specific tests or specific types of tests. Accreditation is a systematic, practical, and often the simplest means of establishing the conformity of an organization to international requirements based on relevant laws, GATT and customer requirements. Accreditation may be defined as: Formal recognition that a testing laboratory is competent to carry out specific tests or specific types of tests.

It is suggested that the ISO/IEC Guide 25:1990 "General requirements for the competence of calibration and testing laboratories" should form the basis for the objective criteria to be developed by this Committee. These criteria should be supplemented with requirements that laboratories participate in proficiency testing schemes, and whenever possible use validated methods of analysis.

Introduction

Most sophisticated products require testing for compliance with specifications and safety regulations before release into many markets, and trade in many simpler commodities and products also requires supporting technical information. Test documentation has become an essential element in this trade. Food intended for human consumption certainly falls into the category "sophisticated products".

In order to avoid barriers to trade and unnecessary duplication of laboratory tests, mutual recognition of laboratory results should be regarded as an important means of facilitating international trade in food products. In the interests both of human health and of legal security it is necessary to develop objective criteria for assessing the competence of testing, i.e. third party approval of laboratories involved in the official control of foods. When criteria have been established, it will become necessary to take measures to introduce suitable systems of quality standards in laboratories involved in the official import and export control of foods.

Lack of acceptance of laboratory test data across national borders is claimed to be a significant barrier to trade. A number of international agreements, such as the GATT Standards Code, the OECD Code of Good Laboratory Practice and the EC and EFTA Policies on Testing and Certification have been developed in efforts to overcome this particular problem and related matters.

The GATT Standards Code requires that standards, testing and certification are not to be used as trade barriers and that testing conducted in the country of export should be accepted in the country of import. Clearly, such acceptance will only be possible if the body responsible for accepting the

goods can have confidence that the laboratories conducting the testing are competent. Therefore, some form of assessment of competence is seen as essential.

The GATT Standards Code is currently being reviewed within the so called "Uruguay Round". Proposals have been made to insert into the code new articles that relate directly to the accreditation of testing laboratories.

In December 1989 the Council of the European Communities adopted a resolution on a Global Approach to conformity assessment, in which acceptance data across national borders were dealt with both within the EC and between the EC and third countries. The Global Approach promotes, among its guiding principles, the setting up of laboratory accreditation systems in each of the European Community countries.

If agreements and policies are to be effective, it is essential that tests made in other countries should be reliable, so that unnecessary duplication of tests can be avoided. Therefore, it is necessary to know the status and competence of testing laboratories which provide data for international trade.

It is important to recognize that confidence in technical matters cannot be imposed by legislation or treaty but must be developed from mutual respect and understanding at all levels amongst the parties concerned.

Mechanisms for Acceptance of Test Data

The existing mechanism by which test data are accepted across borders can be based on:

- * acceptance of test data without question;
- * approval of the laboratory by the acceptance body or customer;
- * approval of the laboratory through evaluation or recommendation by a third party in either country;
- * mutual recognition agreements between laboratories;
- * mutual recognition agreements between laboratory accreditation organizations in both countries.

Each of these arrangements can be shown to work satisfactorily in certain circumstances. It is a common experience, however, that satisfactory agreements on acceptance of test data are almost always underpinned by regular inter-comparisons and technical discussions at the laboratory level.

Quality Standards for Food Laboratories

For a laboratory to produce consistently reliable data it must implement a programme of quality assurance procedures. Analytical methods must be thoroughly validated before use, e.g. by subjecting them to an interlaboratory study conforming to a recognized protocol. These methods must be well documented, the staff adequately trained in their use and control charts should be established to ensure proper statistical control. Accreditation of the laboratory indicates that it applies sound quality assurance principles.

When surveying the current standards containing requirements for the performance of testing laboratories, several standards can be identified. The standards generally have the same objective, to ensure that results reported from a laboratory complying with the standard can be relied upon, i.e. are of a sufficient quality. Four basic terms are required to express the quality of test results: accuracy, precision, repeatability and reproducibility. These terms are explained e.g. in the standard ISO 8402: 1986 "Quality - Vocabulary". When discussing confidence in test results a major concern is assuring a high degree of reproducibility. Ideally, all test methods should produce accurate results with a high degree of precision and should be described in such a way as to ensure high levels of repeatability and reproducibility. Unfortunately, test methods possessing all these characteristics are rare and considerable effort is required to improve the working procedures to prepare relevant test methods. In terms of international trade, problems with reproducibility lead to the most serious disputes.

The OECD Code of Good Laboratory Practice (GLP) is mainly concerned with the testing of chemicals and is best applicable in research work. The GLP rules are not easily applicable to the needs for quality assurance required in laboratories performing tests on a routine basis.

There are a number of regional standards setting down general quality requirements for testing laboratories, e.g. the European EN 45001 standard "General Criteria for the Operation of Testing Laboratories", the National Association of Testing Authorities Australia (NATA) standard "Chemical Testing - Requirements for Registration" and the American National Standard ANSI/ASQC Q2-1991 "Quality Management and Quality System Elements for Laboratories - Guidelines".

The most important and widely accepted international quality standard for testing laboratories is the ISO/IEC Guide 25:1990 "General requirements for the competence of calibration and testing laboratories". In ISO/IEC Guide 25, attention has been paid to the activities of both calibration and testing laboratories and account is taken of other requirements for laboratory competence, such as those laid down in the aforementioned OECD Code of GLP and the ISO 9000 series of quality assurance standards.

It is suggested that ISO/IEC Guide 25 should form the basis of objective criteria for assessing the competence of testing laboratories involved in the official import and export control of foods to be elaborated by this Committee.

ISO/IEC Guide 25: 1990 is available from the ISO Central Secretariat in Geneva and from national standardization institutes.

The ISO/IEC Guide is intended for all kinds of testing laboratories and is therefore rather general in nature. Experience shows that analytical food laboratories are in need of guidance in the interpretation of the standards containing general quality requirements. It may therefore prove necessary to indicate where such guidelines are available, e.g. documents published by the U.K. accreditation body NAMAS (NIS 31 "Accreditation for Microbiological Testing" and NIS 45 "Accreditation for Chemical Laboratories") and an interpretation guide published in 1993 by a joint working group of WELAC (Western European Laboratory Accreditation Cooperation) and Eurachem "Accreditation for Chemical Laboratories: Guidance on the interpretation of the EN 45000 series of Standards and ISO/IEC Guide 25". Since such guidelines are closely related to laboratory practices, the Committee may wish to include the discussion of such interpretative texts in its future work.

Participation in proficiency testing schemes, i.e. determination of laboratory testing performance by means of interlaboratory comparisons, is currently becoming an integral part of many accreditation body requirements. Participation in proficiency testing schemes provides laboratories with a means of objectively assessing, and demonstrating, the reliability of the data they produce. Although there are several types of schemes, they all share a common feature of comparing test results obtained by one laboratory with those obtained by other testing laboratory. At its 20th Session in July 1993 the Commission included in this Committee's terms of reference the elaboration of procedures, protocols, guidelines and related texts for the assessment of food laboratory proficiency, as well as quality assurance systems for laboratories. An available, international proficiency testing harmonized protocol is currently discussed by this Committee.

A provision for the participation in proficiency testing schemes is included in European Council Directive 93/99/EEC on the subject of additional measures concerning the official control of foodstuffs to ensure that all Member States of the EC follow the same procedures - i.e. proficiency testing must be carried out even if the accreditation agency does not formally require it. The EC Directive also requires Member States to ensure that the methods of analysis used by the laboratories involved in the official control of foods have whenever possible been validated in interlaboratory studies conducted in accordance with an internationally recognized protocol, e.g. the standard ISO 5725:1986.

It is suggested that criteria for assessing the competence of testing laboratories involved in the official import and export control of foods are not only based on the requirements of the ISO/IEC Guide 25: 1990, but include:

- * **participation in schemes for proficiency testing;**
- * **the use of analytical methods which have been fully validated in inter-laboratory method performance studies.**

The proficiency testing schemes in which competent laboratories should be requested to participate should be arranged in accordance with recommendations to be issued by this Committee.

In deciding which analytical methods should be regarded as having been fully validated in interlaboratory method performance studies, the recommendation of this Committee should be followed. In November 1992 this Committee approved draft guidelines on the criteria it follows when considering the performance of methods of analysis. These criteria include the requirement that the organization of the study in which the performance characteristics of the analysis methods was assessed, and the statistical analysis of the results obtained, should be carried out according to the principles outlined in the IUPAC 1987 Harmonized Protocol, i.e. "Protocol for the Design, Conduct and Interpretation of Collaborative Studies".

National Accreditation Systems

There is a growing realization that the utilization of national laboratory accreditation systems operating in accordance with national practices defined in various ILAC, ISO/IEC and CEN/CENELEC documents provides an efficient mechanism to assure a sufficient degree of reproducibility of test results and yields a high level of assurance. The mechanisms generally involve some degree of evaluation of the laboratory. Use of the laboratory accreditation option effectively delegates separate evaluations to

a single national body which is regarded as competent to carry out that function. The difficulty is to have all parties agree that these national bodies are in fact competent.

As conceived, if laboratory accreditation bodies operate at a comparable level, if that level meets internationally agreed conditions and if certain safeguards are applied, then assessment by one national body should meet the needs of a second national body. Acceptance of foreign test data then depends on the degree of recognition accorded to the national accreditation body in its own country.

* * *

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