

codex alimentarius commission



FOOD AND AGRICULTURE
ORGANIZATION
OF THE UNITED NATIONS

WORLD
HEALTH
ORGANIZATION



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ALINORM 01/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Twenty-fourth Session
Geneva, Switzerland, 2-7 July 2001

REPORT OF THE TWENTY-THIRD SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Budapest, Hungary
26 February – 2 March 2001

Note: This document incorporates Codex Circular Letter CL 2001/5-MAS

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CL 2001/5-MAS
March 2001

TO: - Codex Contact Points
- Interested International Organizations

FROM: - Secretary, Codex Alimentarius Commission, Joint FAO/WHO Food Standards Programme, FAO, 00100 Rome, Italy

SUBJECT: Distribution of the Report of the 23rd Session of the Codex Committee on Methods of Analysis and Sampling (ALINORM 01/23)

A. MATTERS FOR ADOPTION BY THE 24th SESSION OF THE CODEX ALIMENTARIUS COMMISSION

Proposed Amendments to the Procedural Manual

1. General Criteria for the Selection of Methods of Analysis Using the Criteria Approach (para.41, Appendix II - Part 1)
2. Relations between Commodity Committees and General Committees – Methods of Analysis and Sampling (para. 41, Appendix II - Part 1)
3. Guidelines and Working Instructions to Aid the Implementation of the Criteria Approach to the Selection of Methods of Analysis for Codex Purposes (para. 33, Appendix II - Part 2)

Guidelines for Adoption by Reference for Codex Purposes

4. Harmonized IUPAC Guidelines for the Use of Recovery Information on Analytical Measurement (para. 46, Appendix III)

Methods of Analysis and Sampling

5. General Methods of Analysis for Contaminants and for Irradiated Foods (paras. 86 and 106, Appendix IV - Part III)
6. Methods of Analysis and Sampling in Commodity Standards at different steps (paras. 89-99, Appendix IV - Part I and II)

Governments wishing to propose amendments or comments on the above documents should do so in writing in conformity with the Guide to the Consideration of Standards at Step 8 (see Procedural Manual of the Codex Alimentarius Commission) to the Secretary, Joint FAO/WHO Food Standards Programme, FAO, Viale delle Terme di Caracalla, 00100 Rome, Italy **before 15 May 2001.**

B. REQUEST FOR COMMENTS AND INFORMATION

7. Proposed Draft Guidelines on Measurement Uncertainty (para. 63, Appendix V)
8. Criteria for Evaluating Acceptable Methods of Analysis: Dispute Situations (para. 38)

Governments are invited to provide comments and information on current practices for the selection of methods in dispute situations.

Governments and international organizations wishing to submit comments on points 7. and 8. above should do so in writing to the Secretary, Joint FAO/WHO Food Standards Programme, FAO, Viale delle Terme di Caracalla, 00100 Rome, Italy, **before 15 November 2001.**

SUMMARY AND CONCLUSIONS

The summary and conclusions of the 23rd Session of the Codex Committee on Methods of Analysis and Sampling are as follows:

Matters for consideration by the Commission:

The Committee:

- agreed to propose amendments to the Procedural Manual on *General Criteria for the Selection of Methods of Analysis Using the Criteria Approach* and *Relations between Commodity Committees and General Committees – Methods of Analysis and Sampling* (para. 41, Appendix II - Part 1) and the inclusion of *Guidelines and Working Instructions to Aid the Implementation of the Criteria Approach to the Selection of Methods of Analysis for Codex Purposes* (para. 33, Appendix II - Part 2);
- agreed to propose that the Commission adopt by reference for Codex purposes the *Harmonized IUPAC Guidelines for the Use of Recovery Information on Analytical Measurement* with an amendment to Recommendation 1. (para. 46, Appendix III);
- agreed to propose two methods for contaminants and five methods for the detection of irradiated foods for adoption as general Codex methods (paras. 86 and 106, Appendix IV - Part III);
- endorsed a number of methods of analysis and sampling in commodity standards at different steps of the Procedure (paras. 89-99, Appendix IV – Part I and II);
- agreed to initiate new work on Proposed Draft Guidelines on Measurement Uncertainty (para. 63, Appendix V) and on Proposed Draft Guidelines for the Selection of Methods of Analysis directed to governments (para. 34)

Other Matters of Interest to the Commission

The Committee:

- agreed to return the Proposed Draft Guidelines on Sampling to Step 3 for redrafting, circulation and consideration by the next session (para. 24)
- agreed that it should have a general coordinating role as regards the development of methods of analysis for foods derived from biotechnology (para. 12)
- agreed to consider further at its next session the following questions: single-laboratory validation and validation through the use of results from proficiency testing schemes (para. 84) and the relationship between the analytical result, the measurement uncertainty, recovery factors and the specifications in Codex standards (paras. 20 and 64).

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INTRODUCTION

1) The Codex Committee on Methods of Analysis and Sampling held its Twenty-third Session in Budapest, Hungary, from 26 February to 2 March 2001, by courtesy of the Government of Hungary. The Session was chaired by Professor Péter Biacs, Head of Biotechnological Division of the Central Food Research Institute (KÉKI). The Session was attended by 111 delegates and observers representing 40 Member Countries and 17 international organizations. A complete list of participants is given in Appendix I of this report.

OPENING OF THE SESSION

2) The Session was opened and welcomed by Dr. I. Mucsi, Deputy State Secretary of Ministry of Agriculture and Regional Development. Dr. Mucsi emphasized the importance of Codex standards for harmonization of food legislation and for the export of agricultural commodities from Hungary. He informed the delegates of the active participation of Hungary in Codex work and of the reorganization of the Hungarian National Codex Committee. Dr. Mucsi stressed the importance of the work of the Committee on Methods of Analysis and Sampling in ensuring compliance with provisions in Codex standards and wished the delegates all success in their work.

ADOPTION OF THE AGENDA (Agenda Item 1)

3) The Committee adopted the Provisional Agenda as presented in CX/MAS 01/1.

MATTERS REFERRED BY THE CODEX ALIMENTARIUS COMMISSION AND OTHER CODEX COMMITTEES (Agenda Item 2)¹

Sampling Plan for Aflatoxins in Peanuts

4) The Committee recalled that the 23rd Session of the Commission had adopted the maximum level of 15µg/kg for total aflatoxins in peanuts for further processing and adopted the corresponding sampling plan on an interim basis, with the understanding that proposals for revision would be further considered by the Committee on Additives and Contaminants (CCFAC) and the CCMAS. The Proposed Draft Revised Sampling Plan was circulated for comments at Step 3 in the framework of the CCFAC² and forwarded to the CCMAS for consideration.

5) The Observer from the EC informed the Committee that the EC supported the provisions of the revised sampling plan for total aflatoxins in peanuts for further processing.

6) The Delegation of India proposed to amend sections A. Definitions, B. Sampling and C. Sample Preparation in order to reflect that the aggregate sample should be divided into two sub-samples (10 kg each) to be separately homogenized; the sub-samples would be analysed and the acceptance of the lot would be decided by the average of the results of the two final samples.

7) The Delegation of the United States pointed out that the first amendment proposed by the Delegation of India was already covered in section A. Definition - Test Portion indicating that the aggregate sample could be divided into equal size samples. The Delegation also proposed to replace the Horwitz equation with its simplified version in the current text.

8) The Committee agreed that in section C. Sample Preparation, the reference to “All the material received by the laboratory” should be replaced with “All laboratory sample obtained from aggregate sample”, as proposed by the Delegation of India for clarification purposes.

¹ CX/MAS 01/2

² CX/FAC 01/21

9) The Committee was of view that the other amendments proposed by the Delegation of India might entail further changes to the sampling and analysis provisions for aflatoxins, and that the document should remain unchanged. The Committee noted that since the Committee on Food Additives and Contaminants had the main responsibility for the development of the revised sampling plan, and it had not yet been sent for endorsement to the CCMAS, there was no need to discuss the text in detail at this stage. The Committee agreed that no further change should be made and noted that the Delegation of India and interested delegations had the opportunity to provide their comments directly to the CCFAC.

Biotechnology

10) The Committee recalled that the Committee on Food Labelling, while discussing the labelling provisions for foods derived from biotechnology, had asked the CCMAS to consider the methods of analysis for such foods. The first Session of the *Ad hoc* Intergovernmental Task Force on Foods Derived from Biotechnology had also agreed to initiate work on the methods and to ask for proposals in this area through CL 2000/29-FBT/MAS. The Committee noted that the document prepared for the Task Force (CX/FBT 01/8) had been available too late to allow for specific discussion of the methods proposed therein at the present session.

11) Several delegations supported the establishment of methods for foods derived from biotechnology in the CCMAS. The Delegation of the United Kingdom, supported by other delegations, pointed out that, under its terms of reference, the Committee had the possibility to examine specific analysis and sampling questions referred by other committees, when general guidance was required in areas of concern for all Codex committees. Some delegations and observers stressed the need to take into account the work of relevant international organizations, and to ask the Inter Agency Meeting (IAM) to encourage its members to initiate work on methodology in relevant areas even when no specific Codex provisions existed. The Secretariat indicated that international organizations received all Codex working documents and had the opportunity to submit proposals and relevant information in the framework of commodity Committees and general committees, in addition to CCMAS.

12) The Committee agreed that it should exercise a general coordinating role as regards methods for the detection or identification of foods derived from biotechnology and that it was ready to consider the proposals made by the Task Force at its next session, and the proposals which might originate from other Codex Committees in the future. The Committee also agreed that the work of relevant international organizations would be taken into account in the process and invited them to provide relevant information in this area.

Dioxins

13) The Committee noted that the CCFAC had considered a discussion paper on dioxins at its last session and asked the CCMAS to provide information on methods of analysis for dioxins.

14) It was also noted that there was no level for dioxins in foods under consideration in the CCFAC, and that dioxins had not yet been evaluated by JECFA. The Committee was also informed that the methods of analysis for feeds, including the determination of contaminants, would be considered by the *Ad Hoc* Intergovernmental Task Force on Animal Feeding on the basis of the information provided by governments and international organizations³. Some delegations expressed the view that although there were no limits in Codex for dioxins, it would be useful to consider the selection of appropriate methods in the Committee, taking into account the work underway in different international organizations.

15) Several delegations and observers informed the Committee about ongoing work on the analysis and sampling for dioxins in food and feed. The Committee agreed that the Delegation of Germany would collect information on methodology for the determination of dioxins, and invited interested delegations to provide the Delegation with relevant information, in order to prepare a paper for consideration by the next session.

PROPOSED DRAFT GENERAL GUIDELINES ON SAMPLING (Agenda Item 3)⁴

16) The Delegation of France introduced the Proposed Draft Guidelines, prepared in cooperation with Australia, Hungary, Netherlands, United Kingdom, United States, and IDF, as agreed at the last session, and highlighted the objectives, structure and main aspects of the text. The Preamble presented the main issues and stressed the importance of sampling in the framework of Codex, and considered the consequences of differences in sampling procedures in international trade. The situations covered or excluded by the Guidelines were summarized in Table 1 and the relationship with the ISO and ANSI standards was addressed in section 1.4. The Delegation indicated that the Definitions were based on ISO terminology as far as possible, with the addition of new terms where necessary. The guidelines considered the difference types of sampling plans (by attributes or by variable) and discussed the criteria for their selection in different cases.

17) Some delegations, while recognizing the need for a statistical approach, expressed the view that the document was too complex to provide effective guidance to governments in the selection of sampling plans in its present form, and also stressed the need for practical examples to facilitate its application. It was also pointed out that different sampling plans would apply to processed foods and fresh produce.

18) Some delegations proposed that a simplified document should be prepared to provide practical guidance to Codex Committees and governments in the selection of sampling plans. The Delegation of France agreed that simplified recommendations should be prepared but pointed out that the scientific aspects of sampling should be retained in a specific document, in view of the earlier decision to apply a statistical approach.

19) The Committee agreed to establish a Working Group during the session to revise the text in the light of the comments received and facilitate the discussion in the Plenary. The Committee decided to proceed with its work on the Guidelines on the basis of the recommendations of the Working Group included in CRD 7.

20) The Committee agreed to change the structure of the document as follows: the Foreword would be transferred from the Guidelines to another document which would address the use of analytical results: sampling, relationship between the analytical result, the measurement uncertainty, recovery factors and the specification in Codex standards, to be prepared by the United Kingdom (see also para. 64).

21) The sampling guidelines would consist of a new first section on “Basic recommendations for the selection of Codex sampling procedures” with a flow chart to facilitate the selection of sampling plans, and the following sections would correspond to the current draft (from section 2 onwards).

22) The Delegation of France indicated that it had not been possible to incorporate all the detailed comments received, including those of New Zealand, in a revised document at the current session due to lack of time and that the drafting group would be prepared to continue its task in order to complete the revision of the text.

23) The Committee agreed that the Delegation of France would continue its work with the assistance of a drafting group working by electronic mail in order to complete the revision of the text for circulation at Step 3. The Committee also agreed that if required there would be a meeting of the Working Group prior to the next session in order to allow for detailed consideration of the comments and facilitate discussion in the Committee.

Status of the Proposed Draft General Guidelines on Sampling

24) The Committee agreed to return the Proposed Draft to Step 3 for redrafting by the Delegation of France with the assistance of a drafting group⁵, for circulation at Step 3 and consideration by the next session of the Committee.

⁴ CX/MAS 01/3, CX/MAS 01/3-Corrigendum, CX/MAS 01/3-Add.1 (comments of Brazil, Czech Republic, Denmark, Ireland, New Zealand, United States, CX/MAS 01/3-Add. 2 (comments of Cuba, Finland, Norway, ISO), CRD 4 (Argentina), CRD 7 (revised structure of the document proposed by the WG)

⁵ Brazil, Egypt, Finland, Greece, Hungary, Netherlands, New Zealand, Norway, Poland, Sweden, United Kingdom, United States, Uruguay, ISO, OIE, FAO/IAEA, IDF, NMKL.

25) The Committee expressed its appreciation to the Delegation of France and the drafting group for their considerable work in the preparation and revision of this important document.

CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS FOR CODEX PURPOSES (Agenda Item 4)⁶

PROPOSED DRAFT GUIDELINES ON THE APPLICATION OF THE CRITERIA APPROACH (Agenda Item 4a)

26) The Delegation of the United Kingdom introduced the document which had been prepared in cooperation with other countries, following the decision of the last session to develop guidelines for the implementation of the criteria approach. The Delegation stressed the need to provide guidance to Codex Committees and CCMAS to allow more flexibility in the selection of methods on a rational and scientific basis.

27) The Codex Secretariat recalled that the matter was approved as new work in 1996 in general terms and that the Committee might need to clarify the status of the document as Codex Guidelines providing guidance to governments and included in Volume 13, or as instructions for Codex Committees included in the Codex Procedural Manual.

28) The Delegation of Japan while supporting the principles of the criteria approach and its application to all validated methods, excluding defining methods, expressed concern on its implementation at the national level. The Delegation was of the opinion that the Guidelines should be redrafted in the format of Codex guidelines and should provide guidance not only to Codex committees but also to laboratories of Member countries especially on how to use the criteria approach in selecting methods of analysis.

29) Many delegations supported the criteria approach as being more scientific and flexible and as a consequence more easily applicable for Codex purposes. Some delegations were of the opinion that the criteria approach should be limited to Type III methods while other delegations indicated that it could be also applied to Type II methods in order to allow the use of more modern methods. It was pointed out that there was no need for Type II methods from the scientific point of view.

30) The Delegation of the United States, while fully supporting the criteria approach for Type III methods, supported retaining Type II methods especially for dispute situations and for that purpose Type II methods should be selected by the technical experts of the Commodity Committees or CCMAS. If reference methods were no longer available in legal proceedings, differences in results would eventually be interpreted by the legal profession and not on scientific grounds. Type II methods were therefore necessary to provide guidance to laboratories on specific methods and to eliminate method selection bias in dispute situations. The Delegation also pointed out that the simpler form of the Horwitz equation should be used in Appendix I of the document.

31) Some delegations indicated that dispute situations might occur not only because of methods but also due to inadequate sampling and indicated that in order to minimize situations with different results and disputes, it was necessary to rely more on the use of laboratory accreditation systems and proficiency testing, as already recommended by the CCFICS.

32) As a compromise, the Committee agreed that the criteria approach would be applied to Type III methods and that Type II methods would be prescribed by the CCMAS to be used in dispute situations.

Status of the Proposed Draft Guidelines on the Application of the Criteria Approach

33) The Committee noted that the current text provided recommendations to the CCMAS and Codex Committees and agreed that it should therefore be included in the Procedural Manual at the end of the current section on “Principles for the Establishment of the Codex Methods of Analysis”. The Committee agreed to forward the Proposed “Guidelines and Working Instructions to Aid the Implementation of the Criteria

⁶ CX/MAS 01/4, CX/MAS 01/4-Add.1 (comments of Ireland, USA), CX/MAS 01/4-Add.2 (Dispute situations – extracts from reports of the CCMAS 23 and CCFICS 7).

Approach” (Appendix I, CX/MAS 01/4) to the Committee on General Principles for endorsement and to the Commission for adoption and inclusion in the Procedural Manual (see Appendix II, Part 2).

34) The Committee also agreed that the Delegation of the United Kingdom with the assistance of the Delegation of Sweden would redraft the current Guidelines to make them generally applicable to governments, for circulation at Step 3 and consideration at the next session of the Committee.

Dispute situations

35) The Observer from OIV drew the attention of the Committee to the fact that disputes were more likely to originate from differences in legal practices than from analytical problems and stressed the necessity of suitable analytical methods when the criteria approach was not applicable.

36) The Committee was informed about the existence of ISO standards 5725:1994 and 4259:1992 which provide advice in some dispute situations.

37) The Committee supported the general recommendations made by the Committee on Food Import and Export Inspection and Certification Systems⁷ as regards dispute situations and noted that it did not prevent the development of more specific guidance in this area. Several delegations expressed the view that the Committee should address dispute situations from the practical point of view.

38) The Committee agreed that the Delegation of France with the assistance of Australia, Belgium, Brazil, Canada, Denmark, Egypt, Greece, the Netherlands, Norway, South Africa, the United Kingdom and the United States would prepare a discussion paper addressing dispute situations for consideration by the next session. Governments would be requested by Circular Letter to provide information on the current practices in this regard in member countries, in order to facilitate the preparation of the above discussion paper.

CRITERIA FOR THE EVALUATING ACCEPTABLE METHODS OF ANALYSIS FOR CODEX PURPOSES (Agenda Item 4b)⁸

39) The Delegation of Sweden, speaking on behalf of the member states of the EU present at the session, and presenting a common EC position, proposed the following amendments: in the new section on *General Criteria*, to refer to Codex Type II and Type III (deleting the square brackets); to replace “Normal Process” with “Normal Practice” in the *Relations between Commodity Committees and General Committees*; and to clarify the presentation of the sections.

40) In view of the above discussion and the decision to apply criteria only to Type III methods at this stage, the Committee agreed to delete the reference to Type II methods in the new section. The Committee agreed to the editorial amendments proposed for clarification purposes.

41) The Committee agreed to forward the amendments to the *Principles for the Establishment of Methods of Analysis and Relations between Commodity Committees and General Committees – Methods of Analysis and Sampling* to the Committee on General Principles for endorsement and to the 24th Session of the Commission for adoption and inclusion in the Procedural Manual (see Appendix II, Part 1).

CONSIDERATION OF HARMONIZED GUIDELINES FOR THE USE OF RECOVERY INFORMATION ON ANALYTICAL MEASUREMENT (Agenda Item 5⁹)

42) The Delegation of the United Kingdom while introducing the document pointed out the complexity of the matter and indicated that there were differences in the use of recovery factors: for example in the determination

⁷ ALINORM 01/30 para 102.

⁸ CX/MAS 01/5-Add.1, CX/MAS 01/5 (comments of Cuba, Spain and the European Community).

⁹ CX/MAS 01/6, CX/MAS 01/6-Add.1 (comments of Finland and the USA), CX/MAS 01/6-Add.2 (comments of Cuba and Germany).

of aflatoxins recovery factors was often used, while for residues of pesticides and veterinary drugs the use of recovery factors was variable. The Delegation also indicated that differences regarding the use and the application of the recovery factors existed in the legislation of Member Governments and might create problems in trade.

43) Some delegations indicated that generalized use of recovery factors for each and every matrix would place substantial economic burden on food laboratories and would make it impractical, especially in the case of multi-residue analysis, e.g. pesticide residues. They pointed out that the decision to use or not to use the recovery factors should be left to chemical analysts to decide. However in all cases it should be specified whether the results had been corrected or not, and the method used to derive the correction should be provided.

44) Some delegations indicated that the aim of Codex was not to find the “best estimate of the result” while performing chemical analysis but to obtain comparable results, and that could be achieved by giving information on the application of the recovery factors and the methodology used for that purpose.

45) Many delegations recognised the scientific value of the Guidelines, but emphasised the limitations of the universal application of recovery factors in reporting analytical results. Amongst others it was pointed out that recovery rates were affected by different factors, such as processing or extraction and there were several areas where recovery factors were not applicable.

46) The Committee recognized that the acceptance of the first two sentences of Recommendation 1 in the IUPAC Guidelines would create difficulties, as correction of analytical results would then be a general requirement. However, the other recommendations were acceptable. The Committee decided to endorse the IUPAC, ISO and AOAC International *Harmonized Guidelines for the Use of Recovery Information in Analytical Measurement* with the exception of first two sentences of Recommendation 1¹⁰ regarding the general request for correction. It agreed to recommend to the 24th Session of the Commission its adoption by reference for Codex purposes (see Appendix III).

HARMONIZATION OF ANALYTICAL TERMINOLOGY IN ACCORDANCE WITH INTERNATIONAL STANDARDS: MEASUREMENT LIMITS (Agenda Item 6)¹¹

47) The Delegation of the United States introduced the document, which considered the need for the attributes of Limit of Determination and Limits of Detection, following the decision of the last session of the Committee. The Delegation concluded that there was no need to determine these limits on a routine basis once the method had been selected and proposed to consider a definition of the “Lowest Validated Level” as an alternative to measurement limits. It was also noted that methods of analysis must have “direct pertinence to the Codex Standard to which they are directed” and that Codex MRLs were not set at the limit of detection.

48) Some delegations indicated that the limit of detection was included in the general criteria for the selection of methods and differences in terminology in this respect should be addressed. The Delegation of Uruguay proposed to take into account ISO Standard 11843:1999 Capability of Detection. It was also noted that the use of “Lowest Validated Level” instead of limit of determination in relation to method validation was consistent with the use of the term “Lowest Calibrated Level” in relation to analytical quality control.

49) Some delegations and the Observer from COMISA expressed the view that there was no need for additional definitions in this area. It was also noted that the issue of measurement limits was more relevant for residues of pesticides and veterinary drugs, which were not the responsibility of CCMAS.

¹⁰ “Quantitative analytical results should be corrected for recovery unless there are specific reasons for not doing so. Reasons for not estimating or using correction factors include the situations where (a) the analytical method is regarded as empirical, (b) a contractual or statutory limit has been established using uncorrected data, or (c) recoveries are known to be close to unity. However,.....”

¹¹ CX/MAS 01/7

50) The Observer from AOAC indicated that the harmonization of definitions and terminology was included in the future activities of the Inter Agency Meeting (IAM), and that the Committee would be kept informed of further developments in this area.

51) The Committee agreed that there was no need for further consideration of measurement limits at this stage, and that it might consider this issue again at a later date if needed.

MEASUREMENT UNCERTAINTY (Agenda Item 7)

PROGRESS REPORT BY RELEVANT INTERNATIONAL ORGANIZATIONS (Agenda Item 7a)

52) The Delegation of Ireland informed the Committee that the second edition of the EURACHEM/CITAC Guide Quantifying Uncertainty in Analytical Measurement had been published. The changes dealing with the use of method performance data and method validation data in order to determine the components of uncertainty were of particular importance to the Committee. The Delegation pointed out that the examples had been revised and that the Guide also included a new section on traceability. It was noted that a summary was included in document CX/MAS 01/8 and that the complete Guide was available from the EURACHEM website: <http://www.vtt.fi/ket/eurachem>

53) The Delegation of Finland also informed the Committee that the NMKL procedure for the expression of measurement uncertainty was under revision and would be harmonized with the EURACHEM Guide.

RELATIONSHIP BETWEEN THE ANALYTICAL RESULT, THE MEASUREMENT UNCERTAINTY AND THE SPECIFICATION IN CODEX STANDARDS (Agenda Item 7b)¹²

54) The Delegation of the United Kingdom, while presenting the document, highlighted the main issues to be addressed: the terminology, the methodology to estimate measurement uncertainty and its incidence on the interpretation of the result and the assessment of compliance. The Delegation pointed out that the term uncertainty was now widely accepted in the analytical community; that laboratories complying with the principles of laboratory quality did not need a further estimate of the measurement uncertainty according to the ISO component-by-component approach, and that results should be generally reported with the measurement uncertainty. The Committee also noted that the ISO/IEC Guide 25 (endorsed by Codex) had been replaced by ISO/IEC 17025, which referred to measurement uncertainty.

55) The Committee considered the Recommendations presented at the end of the document and agreed that they should be drafted as Guidelines on Measurement Uncertainty intended for governments, to be circulated at Step 3, subject to approval as new work by the Commission. The Committee had an extensive discussion and made a number of amendments and additions to the initial text.

56) The Introduction was reworded for clarification purposes and to reflect the importance of the issue. Some delegations questioned the provision that “food analysis laboratories are required to be in control”. The Delegation of the United Kingdom pointed out that this was a well understood expression to reflect that laboratories applied Codex principles related to the quality of laboratory results.

57) The Delegation of Finland made the following proposals in order to clarify the text; in point 1) the measurement uncertainty should be “estimated” rather than “quantified”; in point 2) it would be preferable to refer first to data available through the use of internal quality control; in point 4) reference to single-laboratory validation could be included.

58) The Delegation of the United States, supported by the Delegation of Thailand, expressed the view that, as measurement uncertainty was a relatively new concept, it might not be well known in some countries, and the Committee should therefore recognize that “reliability” might still be used. The Delegation of Ireland pointed out that “uncertainty” was an accepted term at the international level and that Codex was a reference in

¹² CX/MAS 01/8, CRD 6 (comments of France)

international trade, and proposed that the ISO definition should be used with an appropriate explanation in the text of the Guidelines.

59) The Committee agreed to include the ISO definition of measurement uncertainty in order to clarify the issue of terminology. As a compromise, an explanatory note was added in square brackets after Note 3) to reflect that “measurement reliability” might also be used.

60) The Observer from IOFI indicated that the question of measurement uncertainty was especially important for the analysis of compounds which were present at low concentrations like flavours, as related to statutory provisions or compliance with specifications. The Committee noted that the question of compliance had also been discussed in relation to recovery factors and would need to be addressed from a general perspective (see also para. 64).

61) The Committee agreed to merge Recommendations 3 and 4 as they both referred to the methodology used to evaluate uncertainty. The Delegation of the United States proposed to add an additional sentence to indicate how the overall uncertainty might be determined.

62) The Delegations of Spain and Uruguay pointed out that the term “reliability” had negative implications and should not be used in Spanish. It was therefore important to specify that the expression used should be equivalent to “uncertainty” and had the same meaning. The Committee agreed to refer to an “alternative, equivalent term” and noted that member countries would have the possibility to make comments on the appropriate terminology in different languages. The Delegation of Ireland informed the Committee that the journal Accreditation and Quality Assurance (AQAL) (Volume 1, No. 1, January 1996) included a Glossary of Analytical Terms in which “uncertainty of measurement” was translated into several languages.

63) The Committee agreed to circulate at Step 3 a first draft of the Proposed Draft Guidelines on Measurement Uncertainty for Governments, as attached in Appendix V, subject to approval by the Commission as new work.

64) The Delegation of the United Kingdom proposed to prepare a discussion paper addressing the compliance aspects associated with measurement uncertainty, recovery factors and sampling since these different issues were related, and invited interested delegations to participate in the preparation of the paper. The Committee welcomed this proposal as there was a need for practical guidance to governments and food control laboratories in this area, and agreed to consider this question at the next session (see para. 60).

IN-HOUSE METHOD VALIDATION (Agenda Item 8)¹³

65) The Committee recalled that the last session had agreed to consider the next draft of the IUPAC *Harmonized Guidelines for the In-House Validation of Methods of Analysis* when it became available. It had also agreed that the Delegation of the Netherlands would prepare a discussion paper on the characteristics of in-house validated methods for consideration by the next session.

66) The Committee agreed that it was preferable to use the term “single-laboratory validation” instead of “in-house validation” as proposed by the Delegation of the Czech Republic.

67) The Delegation of the Netherlands recalled that this question was essentially relevant for the determination of residues of pesticides and veterinary drugs, as relatively few collaboratively tested methods were available, and proposed the following recommendations for consideration by the Committee: to recognize the use of single laboratory validation for Codex purposes; to adopt criteria for in-house validated methods for Codex purposes; and to adopt the guidelines prepared by FAO/IAEA/AOAC for this purpose.

68) The Delegation of the United Kingdom indicated that the IUPAC Guidelines provided guidance on in-house validation but that it should not generally replace collaborative testing, and pointed out that two separate issues should be considered by the Committee: in-house validation, and the use of proficiency testing. The

Delegation also drew the attention of the Committee to the examples presented in Annexes II and III as reflecting the practical experience of the United Kingdom in this area.

IUPAC Guidelines

69) The Committee noted that the Guidelines were not yet published in their final form by IUPAC, and therefore it was not possible to propose their adoption by reference at the present session.

70) The Observer from AOAC indicated that AOAC did not harmonize yet with the approach of the IUPAC Guidelines as they did not provide practical guidance on how to proceed with single laboratory validation. This view was supported by some delegations. The Delegation of the United Kingdom pointed out that the IUPAC Guidelines were intended as an overarching document rather than a detailed manual for food laboratories, and invited governments and international organizations to provide their comments to IUPAC.

71) The Delegation of Ireland informed the Committee that the EURACHEM Guide "*Fitness for purpose of analytical methods. A Laboratory Guide to method validation and related topics*" could provide useful guidance for food laboratories on single-laboratory validation. The Committee noted that NMKL had also established a procedure on in-house method validation and that NMKL had recently published guidelines on how to use certified reference materials: NMKL Procedure No. 9 (2001) *Evaluation of Results Derived from the Analysis of Certified Reference Materials*.

72) The Delegation of the United States proposed to reword the end of the second paragraph in section 4.4 *Method and Laboratory Effects* to the effect that "the Horwitz function deviates at concentrations lower than about 120 ppb".

Requirements for single-laboratory validation

73) The Committee had an exchange of views on the criteria proposed in the working document (page 2).

74) The Delegation of Germany, supported by other delegations and observers, expressed the view that the use of single-laboratory validation should not be restricted to multi-residue methods and that this recommendation should be revised (paragraph 8a). Some delegations pointed out that this problem was more general as laboratories had to develop new methods to detect substances which posed a hazard to health for food control purposes, such as dioxins or contaminants in contact material.

75) The Delegation of Germany also pointed out that the recommendations might be in contradiction with the criteria approach already approved by the Committee, especially as in the future certain methods might be presented only as criteria. The Committee agreed that these points should be addressed further in the revision of the "Requirements" section.

Relationship with the CCPR and CCRVDF

76) The Representative of FAO/IAEA informed the Committee that the lectures presented at the AOAC/FAO/IAEA/IUPAC International Workshop on Principles and Practices of Method Validation (4-6 November 1999) had been published in scientific literature¹⁴, including the Guidelines for Single Laboratory Validation of Analytical Methods for Trace-Level Concentrations of Organic Chemicals, which were subsequently finalized by the FAO/IAEA/AOAC Consultation (8-11 November 1999).

77) The Representative drew the attention of the Committee to the need for a harmonized approach to single/laboratory validation in the areas of veterinary drug residues and pesticide residues and stressed the

¹⁴ Principles and Practices of Method Validation, Royal Society of Chemistry, Cambridge, 2000.

importance of the work of FAO/IAEA in this area. The Delegation of the Netherlands proposed to forward the Guidelines to the CCPR and CCRVDF.

78) The Delegation of Sweden recalled that under part (g) of its terms of reference the Committee could make general recommendations to other committees on procedures and protocols, such as the IUPAC Guidelines, even when those committees were responsible for the development of their own methods.

79) The Secretariat confirmed that the CCMAS could address general recommendations on methodology to all other Committees, and that would apply to the IUPAC Guidelines. However it was not within its mandate to consider and make recommendations on a document covering exclusively chemical residues, such as the FAO/IAEA/AOAC Guidelines mentioned above.

80) The Secretariat also recalled that the usual practice for UN Agencies was to distribute the reports of expert consultations to Codex Committees when the recommendations put forward by these consultations required specific consideration by the Committees. This had been the case for example with the 1997 FAO/IAEA *Consultation on Validation of Analytical Methods for Food Control*¹⁵ and was standard practice for FAO/WHO Consultations. According to the usual procedure, it was expected that the next sessions of the CCPR and the CCRVDF would have the opportunity to consider the reports of both the AOAC/FAO/IUPAC/IAEA Workshop and the FAO/IAEA/AOAC Consultation including the Guidelines, when distributed by FAO/IAEA.

81) The Committee was informed that the last session of the CCRVDF had noted the general results the AOAC/FAO/IUPAC/IAEA Workshop and the FAO/IAEA/AOAC Consultation¹⁶, and the CCPR had noted the results of the FAO/IAEA/AOAC Consultation¹⁷ but the relevant reports and guidelines had not been published at the time. The Committees had already agreed that they would take into account the relevant recommendations made in these meetings while developing criteria for the assessment of suitable analytical methods.

82) The Committee took note that the Workshop and the Consultation had made a number of recommendations concerning single-laboratory validation, including the Guidelines, and invited CCPR and CCRVDF to consider them further, in order to ensure a harmonized approach throughout Codex for single laboratory validation.

Further action

83) The Committee generally recognized that single-laboratory validation could be used for Codex purposes and agreed that the next session would consider the inclusion of a specific text in the Procedural Manual to that effect.

84) The Committee agreed that it would consider the published version of the IUPAC *Harmonized Guidelines for the In-House Validation of Methods of Analysis* at its next session, with a view to adopting them by reference. The Delegation of the Netherlands, with the assistance of interested countries, would revise the paper on Requirements for Single-Laboratory Validation in the light of the discussion held at the present session. In addition, the Delegation of the United Kingdom would prepare a paper on the validation of methods through the use of results from proficiency testing schemes as a separate issue.

¹⁵ Food and Nutrition Paper No. 68, Rome 1998

¹⁶ ALINORM 01/313, para 99-101

¹⁷ ALINORM 01/24, paras. 152–153

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS (Agenda Item 9)¹⁸

85) The report of the *ad hoc* Working Group on Endorsement of Methods of Analysis (CRD 1) was presented by its chairperson Dr. William Horwitz (USA). Dr. Gregory Diachenko served as rapporteur. The following countries and international organizations participated in the Working Group: Brazil, Finland, Germany, Hungary, Japan, Malaysia, the United Kingdom, the United States, AOAC International, CEN, EC, IDF, ISO and NMKL.

86) The Committee agreed to replace the Codex General Method for copper (AOAC 971.20) by the NMKL Method 139 (1991) for lead, cadmium, copper, iron and zinc in foods by AAS after dry ashing which is also identical to AOAC 999.11 (as Type II method). The Committee also agreed to endorse the NMKL Method 161 (1998), which uses AAS following microwave digestion, and the identical method AOAC 991.10 as a Type III Codex General Method for lead, cadmium, zinc, copper and iron in foods.

87) The Committee concluded that the NMKL method proposed by the Committee on Food Additives and Contaminants for the analysis of Ochratoxin A in cereals and cereal products appeared to work well for the commodities specified at a level of >2ng/g. The Committee also felt that it would not be procedurally correct to endorse a method before relevant Codex provisions had been established. It was also pointed out that other methods were currently being validated for Ochratoxin A, and that CCFAC might also consider them.

88) The Committee was informed that the reference to ICUMSA GS 2/3-5 to measure invert sugar in soft sugars and brown sugar was not correct and that the correct reference was ICUMSA GS 1/3/7-3.

89) The Committee noted the written comments of CEFS regarding the difference in the results of determination of colour values for sugar with a colour higher than 60 IU using different ICUMSA methods. However the ICUMSA GS 2/3-10 (1998) method was retained on the basis of the recommendation of the Commodity Committee.

90) The Committee decided to delete the methods for determination of arsenic and lead in the sugar standards as there were no provisions for those contaminants there.

91) The Committee changed the status from “endorsed” to “temporarily endorsed” for several methods in the honey standard, due to lack of information on collaborative studies and to be consistent with earlier decisions.

92) The Committee agreed to request clarification of the Committee on Sugars regarding the availability of the specific reagent for the Phadebas method.

93) The Committee accepted the information of the Delegation of Norway regarding the availability of the collaborative studies and endorsed the WEFTA method for determination of salt in Salted Fish of the *Gadidae* Family as a Type II method, with the understanding that the result will be calculated on the basis of the chloride content. The Committee also agreed that the method of determination of histamine in Salted Atlantic Herring (AOAC 977.13) was applicable to all fish species where histamine was a concern and endorsed the method for “fish and fishery products”, as this would apply to all relevant standards.

94) As there were separate provisions for moisture and for solids in different cheese standards, the Committee decided to include separate entries for these provisions.

95) The Committee decided to delete the reference to a deviation of $\pm 2^{\circ}$ C in Whey Cheese standard as the precise information for that value had been provided in the standard, and the endorsement document included only the principle of the method.

96) The Committee deleted the reference to unpublished ISO standard for the determination of *Streptococcus thermophilus* in Fermented Milks (yoghurt) and decided not to endorse methods for protein and dry matter in the Standard for Unripened Cheese including Fresh Cheese as these provisions were not specified in the standard.

¹⁸ CX/MAS 01/10, CX/MAS 01/10-Add.1 CRD 1 (report of the *ad hoc* Working Group).

97) The Committee did not endorse the methods for the determination of fat content in the draft Standard for Cocoa Powders and requested the CCCPC to provide evidence of collaborative study validation and information regarding the purpose and the type of method. The Committee did not endorse the method for the determination of copper in the Proposed Draft Standard for Chocolate and Chocolate Products and requested the Committee to consider adopting one of the Codex general methods for copper that to determine the percentage of fat in these products.

98) The reference to the Committee on Processed Fruits and Vegetables was included in the section on Aqueous Coconut Products as the draft standard (initiated by the CCASIA) was currently under consideration in that Committee. The Committee also agreed to request the CCPFV to consider its earlier questions concerning the draft Standard for Pickles while finalizing the Standard.

Endorsement of Methods of Sampling Provisions

99) The Committee endorsed the sampling provisions in several standards for milk products. The Committee also endorsed sampling provisions in the Draft Standard for Wheat Protein Products and amended the reference to the ISO standard.

Methods for Detection of Irradiated Foods

100) The Delegation of Sweden, speaking on behalf of the Member States of the EU participating at the present Session and presenting a common EC position, introduced CRD 3 and CRD 3 Addendum and recalled that the Codex General Standard for the Labelling of Prepackaged Foods required mandatory labelling of irradiated foods and it was therefore necessary to establish methods for control purposes. The Delegation pointed out that with the assistance of FAO/IAEA Joint Division of Nuclear Techniques in Agriculture, a number of methods were elaborated and validated and that those methods were subsequently standardized by CEN. The Delegation proposed to consider and endorse five methods for the detection of different irradiated foods as presented in CRD 3.

101) The Observer from AOAC pointed out that the Committee on Food Additives and Contaminants had not proposed any methods for the detection of irradiation of foods, and indicated that methods are proposed by commodity committees well in advance of the meeting in order to be considered by the Working Group on the endorsement of methods under the CCMAS. Some delegations and the Secretariat recalled that the Codex provision concerned was the requirement for the labelling of irradiated foods in the General Standard for the Labelling of Prepackaged Foods, that it was under the terms of reference of the Committee to consider general methods and that this was not an endorsement of methods proposed by commodity committees.

102) The Delegation of the United States indicated that despite numerous references there was no bibliography provided for the methods and that the usual procedure for the Committee was to consider such a matter through the Working Group.

103) Following the request of the Delegation of Australia regarding false positive or negative results, the Observer from the EC indicated that methods provided a very high percentage of correctly identifiable samples which in some cases reached even 100%. It was pointed out that these methods were currently used in practice in some countries with a significant success and were thoroughly validated.

104) The Delegation of the United Kingdom urged the Committee to take a more proactive position on such important issues and supported the consideration and endorsement of the proposed methods at the current session. This view was supported by some other delegations and the Representative from FAO/IAEA.

105) The Committee had an extensive debate on the typing of proposed methods. Some delegations indicated that these methods could be attributed to Type I as they provide only an estimate of positive or negative results while other delegations pointed out that methods could be differentiated between Type II and Type III.

106) The Committee decided to endorse the proposed methods and concluded that the method EN 1785:1996 *for detection of irradiated foods containing fat on the basis of GC/MS analysis of 2-alkylcyclobutanones* should be endorsed as Type III and the remaining methods were specified as Type II (see Appendix IV).

REPORT OF AN INTER-AGENCY MEETING ON METHODS OF ANALYSIS (Agenda Item 10)¹⁹

107) The draft report of the 14th Interagency Meeting was presented by the Observer of AOAC International. The Committee was informed that 20 international organizations attended the meeting and noted that the meeting appreciated very much the chairmanship of Dr Roger Wood (UK).

108) The Committee was informed and that three new organizations (American Oil Chemists' Society, National Food Processors Association and AOAC Research Institute) were accepted as new members. The Observer pointed out that several matters might be of direct relevance to the CCMAS and other Codex Committees, such as: harmonization of analytical terminology in accordance with international standards, quality assurance in food analysis, policies and practices regarding the use of proprietary techniques, assessment of the standard method of analysis - effects of proficiency testing etc.

109) The Observer drew the attention of the Committee to the list of actions and indicated that the draft report, when finalized, would be placed on the AOAC webpage at: www.aoac.org.

110) The Committee noted the report of the 14th IAM and expressed its appreciation to the IAM for providing technical support to the work of Committee in many fields.

OTHER BUSINESS AND FUTURE WORK (Agenda Item 11)

111) The Committee noted that its future work would consist of the following matters:

- Proposed Draft General Guidelines on Sampling
- Proposed Draft Guidelines on Measurement Uncertainty
- Consideration of Guidelines for the Use of Criteria Approach including consideration of dispute situations
- Single-Laboratory Validation
- Validation of methods using the results of proficiency testing
- The use of analytical results: sampling, relationship between the analytical results, the measurement uncertainty, recovery factors and the specifications in Codex Standards
- Methods of Analysis for the detection and identification of GMOs
- Methods of Analysis for the determination of dioxins
- Endorsement of Methods in Codex Standards

DATE AND PLACE OF THE NEXT SESSION (Agenda Item 12)

112) The Committee was informed that the next session of the Committee was tentatively scheduled to be held in Budapest in November 2002. The exact date and venue would be determined between the Host government and the Codex Secretariat, subject to the approval of the CAC.

¹⁹ CRD 2 (Draft Report of the 14th Interagency Meeting (IAM-14)).

SUMMARY STATUS OF WORK

Subject Matter	Step	Action by	Document Reference in ALINORM 01/23
Proposed amendments to the Procedural Manual: <ul style="list-style-type: none"> - Criteria for the Selection of Methods of Analysis Using the Criteria Approach - Relations between Commodity Committees and General Committees - Guidelines and Working Instructions to Aid the Implementation of the Criteria Approach 		CCGP Governments 24 th CAC	paras. 33 and 41 Appendix II
IUPAC Guidelines for the Use of Recovery Information in Analytical Measurement (for adoption by reference)	(*)	Governments 24 th CAC	para. 46 Appendix III
Endorsement of methods of analysis and sampling, including general methods		Governments 24 th CAC	paras. 86 to 106 Appendix IV
Proposed Draft General Guidelines on Sampling	3	France Governments 24 th CCMAS	para. 24
Proposed Draft Guidelines on Measurement Uncertainty	1/2/3	24 th CAC Governments 24 th CCMAS	para. 63 Appendix V
Proposed Draft Guidelines for evaluating acceptable methods of analysis	1/2/3	24 th CAC United Kingdom 24 th CCMAS	para. 34
Validation of methods <ul style="list-style-type: none"> - Single Laboratory Validation - Use of Proficiency Testing Schemes 		Netherlands IUPAC United Kingdom	para. 84

(*) Equivalent to Step 8

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PROPOSED AMENDMENTS TO THE PROCEDURAL MANUAL**PART 1 – AMENDMENTS TO CURRENT SECTIONS****1. PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS¹**

Addition of a new subsection at the end of *General Criteria for the Selection of Methods of Analysis* as follows:

General Criteria for the Selection of Methods of Analysis using the Criteria Approach

In the case of Codex Type III methods, method criteria may be identified and values quantified for incorporation into the appropriate Codex commodity standard. Method criteria which are developed will include the criteria in section Methods of Analysis, paragraph (c) above together with other appropriate criteria, e.g., recovery factors.”

2. RELATIONS BETWEEN COMMODITY COMMITTEES AND GENERAL COMMITTEES - METHODS OF ANALYSIS AND SAMPLING²

Addition of new paragraphs at the end of “*Normal Practice*” section as follows:

“The Codex Committee on Methods of Analysis and Sampling will assess the actual analytical performance of the method which has been determined in its validation. This will take account of the appropriate precision characteristics obtained in collaborative trials which may have been carried out on the method together with results from other development work carried out during the course of the method development. The set of criteria that are developed will form part of the report of the endorsement by the Codex Committee on Methods of Analysis and Sampling and will be inserted in the appropriate Codex Commodity Standard.

In addition, the Codex Committee on Methods of Analysis and Sampling will identify numeric values for the criteria for which it would wish such methods to comply.”

¹ Procedural Manual, 11th Edition, pages 72-73

² Procedural Manual, 11th Edition, page 96

PART 2 – ADDITION OF A NEW SECTION IN THE *PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS*

PROPOSED GUIDELINES AND WORKING INSTRUCTIONS TO AID THE IMPLEMENTATION OF THE CRITERIA APPROACH TO THE SELECTION OF METHODS OF ANALYSIS FOR CODEX PURPOSES

(for inclusion at the end of the *Principles for the Establishment of Codex Methods of Analysis*)

INTRODUCTION AND BACKGROUND

The Codex Alimentarius Commission (CAC) has historically endorsed specific methods of analysis for Codex purposes. These methods of analysis have to comply with the quality criteria given in the Codex Procedural Manual. However, the Commission has recently adopted the “criteria approach” (*aka* performance based approach) for the acceptance of methods of analysis for Codex purposes in some situations. This approach allows the endorsement of method criteria by the Commission rather than only the adoption of identified methods of analysis.

These Guidelines outline Working Instructions on how and when this new approach should be employed by Codex Commodity Committees when recommending methods of analysis for endorsement by the Codex Committee on Methods of Analysis and sampling, and their final acceptance by the Commission.

PRESENT SYSTEM

The present procedure for the adoption of methods of analysis within the Codex System requires Codex Committee on Methods of Analysis and Sampling (CCMAS) to consider and endorse methods of analysis proposed by Commodity Committees in the elaboration of their Codex Standards. In addition CCMAS may propose methods of analysis of general applicability (e.g. for trace elements). Methods of analysis proposed by Commodity Committees or by CCMAS may be Codex Type I, II, III or IV procedures; these types are defined in the Guidelines on Codex Methods of Analysis and Sampling given in the Codex Procedural Manual. These Guidelines recognise that there are, in essence, 2 sorts of methods of analysis, i.e.

- defining or empirical procedures, where the analytical result is method dependent (e.g. the determination of “fat” in a food), and
- the determination of a discrete chemical entity where the analytical result is not, in principle, method dependent (sometimes known as rational methods).

In addition, for specific methods of analysis, preference should be given to methods of analysis the reliability of which have been established in respect of the following criteria, selected as appropriate:

- specificity
- accuracy
- precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories)
- limit of detection
- sensitivity
- practicability and applicability under normal laboratory conditions
- other criteria which may be selected as required.

and, in addition,

- the method selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use,
- all proposed methods of analysis must have direct pertinence to the Codex Standard to which they are directed.
- methods of analysis which are applicable uniformly to various groups of commodities should be given preference over methods which apply only to individual commodities.

- official methods of analysis elaborated by international organisations occupying themselves with a food or group of foods should be preferred.

THE NEW APPROACH

The new approach will only apply to the determination of specific chemical analytes (i.e. Type III methods). It will not apply to Type I defining methods of analysis: however, it should be noted, that most of the empirical methods (i.e. the Type I methods) required by the Codex Alimentarius Commission have already been adopted by the Commission. Specific empirical methods already adopted by the Commission remain attached to the appropriate standard. They need not be reviewed unless the Standard itself is reviewed. Then the Codex Commodity Committee will still have to recommend a single Type I method which will be assessed by the Codex Committee on Methods of Analysis and Sampling on its own merits.

When a Codex Commodity Committee has developed a standard and the method of analysis to be attached to it, the Committee shall decide whether the method also to be developed is a Type I empirical procedure, a Type II method, a Type III rational procedure or a Type IV procedure. The Codex Commodity Committee will then proceed along the following lines:

Type I Methods

In the present system this is a method which determines a value that can only be arrived at in terms of the method *per se* and serves by definition as the only method for establishing the accepted value of the item measure.

The procedure for Type I methods remains as at present, i.e. specific methods are attached to the Commodity Standard and then considered for endorsement by the CCMAS. As type I methods are empirical, i.e. the analytical result is intimately linked to the method used to obtain that result, it is not appropriate to separate the specification and the method to determine the specification.

The Commodity Committee will select the appropriate Type I method as at present. It will be required to meet the existing criteria as given above. It will be sent to CCMAS for consideration and endorsement. There will be no change to the present system.

The number of Type I methods to be endorsed by CCMAS should decline in future as the number of specific commodity linked specifications without methods attached declines. Internationally, there is a tendency to consider that safety aspects of food have a greater importance than compositional/commodity aspects. Codex is following that tendency, and thus the majority of methods from “active” Codex Committees will be concerned with an identifiable, discreet chemical substance (i.e. be Type II or III methods).

Type II and III Methods

Type II: Reference Method: Type II methods are retained.

Type III: Alternative Approved Method: In the present system this is a method which meets the criteria required by the Codex Committee on Methods of Analysis and Sampling for methods that may be used for control, inspection or regulatory purposes.

The Codex Commodity Committee may continue to propose an appropriate method of analysis for the chemical entity being determined or develop a set of criteria to which a method used for the determination must comply. It is expected that the Codex Commodity Committee will find it easier to recommend a specific method and request CCMAS to “convert” that method into appropriate criteria. The criteria would then be endorsed by CCMAS and will form part of the Codex Commodity Standard replacing the recommended method of analysis. If the Codex Commodity Committee which is to develop the criteria itself rather than allowing the endorsement working party of CCMAS to do so, then it should follow instructions given for the development of specific criteria as outlined below. These criteria must be approved/recommended for the determination in question.

However, the primary responsibility for supplying methods of analysis and criteria resides with the Commodity Committee. If the Commodity Committee fails to provide a method of analysis or criteria

despite numerous requests, then CCMAS may supply an appropriate method and “convert” that method into appropriate criteria.

When CCMAS endorses, or recommends, a Type II or III method it is considering the applicability of the method in a given situation. On occasions a number of methods for the same determination are considered for endorsement by CCMAS: one of these will be selected, on often arbitrary grounds, as the Type II method, the rest being treated as Type III methods.

In future any method capable of being shown that it meets the given analytical characteristics will be “approved” for use for Codex purposes as a Type III method.

The minimum “approved” Codex analytical characteristics will include the following numeric criteria as well as the general criteria for methods laid down in the Codex Procedural Manual:

- precision (within and between laboratories, but generated from collaborative trial data rather than measurement uncertainty considerations)
- recovery
- selectivity (interference effects etc.)
- applicability (matrix, concentration range and preference given to 'general' methods)
- detection/determination limits if appropriate for the determination being considered
- linearity

CCMAS will generate the data corresponding to the above criteria. CCMAS has defined the terms to be used for each of the characteristics to be evaluated. These are given in Annex I. It will be necessary for a laboratory to demonstrate that whatever method it uses, its application conforms to the laboratory quality standards adopted by the Codex Alimentarius Commission.

Much of the data that will be required by CCMAS should be submitted to the Committee by the Codex Commodity Committees as result of the adoption of the Checklist of information required to evaluate methods of analysis submitted to the Codex Committee on Methods of Analysis and Sampling for endorsement.

In practice it must be appreciated that such information rarely, if ever, is provided by the Commodity Committees.

Type IV Methods

In the present system this is a method which has been used traditionally or else has been recently introduced but for which the validation criteria required for acceptance by the Codex Committee on Methods of Analysis and Sampling have not yet been determined.

Type IV methods will be considered as at present, i.e. they will be “noted” by CCMAS but not formally endorsed. Type IV methods are candidate Type I, II and III methods.

Type IV methods will continue to be treated in their own right as tentative procedures. It will not be possible to convert them to criteria as their precision characteristics would be unknown: Type IV methods have not been subjected to a collaborative trial.

CONVERSION OF SPECIFIC METHODS OF ANALYSIS TO METHOD CRITERIA BY THE CCMAS

The CCMAS endorses specific methods of analysis which are sent to it by Codex Commodity Committees. It also recommends adoption of certain Codex general methods of analysis which are not linked to a specific quality standard. The CCMAS will take the information that should be supplied by the Codex Committee seeking endorsement of the method and convert it into suitable generalised analytical characteristics. The CCMAS will convert to criteria those Type II and III methods which are sent to it for endorsement.

Information on the following criteria will be required to enable the conversion to be undertaken:

- accuracy
- applicability (matrix, concentration range and preference given to 'general' methods)
- detection limit
- determination limit
- precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories), but generated from collaborative trial data rather than measurement uncertainty considerations
- recovery
- selectivity
- sensitivity
- linearity

These terms are defined in Annex I, as are other terms of importance. Comments on each of the terms, if appropriate, together with suggested acceptable numeric values is also included in the Annex.

The CCMAS will assess the actual analytical performance of the method which has been determined in its validation. This will take account of the appropriate precision characteristics obtained in collaborative trials which may have been carried out on the method together with results from other development work carried out during the course of the method development. The set of criteria that are developed will form part of the report of the CCMAS and will be inserted in the appropriate Codex Commodity Standard.

In addition, the CCMAS will identify numeric values for the criteria for which it would wish such methods to comply, i.e. it will be pro-active as well as reactive.

ACCEPTABILITY OF THE VALUES USED

The definitions required to implement the Instructions are given in the Codex Procedural Manual as supplemented by the comments given in Annex II.

RETROSPECTIVE ACTION

There are a large number of methods already adopted by Codex. These will be left as at present and, if the criteria approach is adopted, then only methods which are still to be elaborated in Codex Standards or endorsed by CCMAS be displayed as criteria, except in cases where a multiplicity of methods are considered for endorsement as Type III methods by CCMAS, e.g. for trace element determinations.

ANNEX I: ANALYTICAL TERMINOLOGY FOR CODEX USE AND INFORMATION OF ACCEPTABLE NUMERIC VALUES

Information on Analytical Terminology for Codex Use is given in the CAC Procedural Manual. Where the terminology is to be amended or expanded, this is indicated below. Information on the terms which may be used in the elaboration of the criteria are given below:

TERMINOLOGY

The following terms are defined in the Procedural Manual:

- Accuracy
- Applicability
- Precision
- Selectivity
- Sensitivity

OTHER TERMS TO BE USED IN THE CRITERIA APPROACH

Detection Limit

The detection limit is conventionally defined as field blank + 3σ , where σ is the standard deviation of the field blank value signal (IUPAC definition).

However, an alternative definition which overcomes most of the objections to the above approach (i.e. the high variability at the limit of measurement can never be overcome) is to base it on the rounded value of the reproducibility relative standard deviation when it goes out of control (where $3\sigma_R = 100\%$; $\sigma_R = 33\%$, rounded to 50% because of the high variability). Such a value is directly related to the analyte and to the measurement system and is not based on the local measurement system.

Determination limit

As for detection limit except that 6σ or 10σ is required rather than 3σ .

However, an alternative definition that corresponds to that proposed for the detection limit is to use $\sigma_R = 25\%$. This value does not differ much from that assigned to the detection limit because the upper limit of the detection limit merges indistinguishably into the lower limit of the determination limit.

Recovery

Proportion of the amount of analyte present or added to the test material which is extracted and presented for measurement.

Selectivity

Selectivity is the extent to which a method can determine particular analyte(s) in mixtures or matrices without interferences from other components.

Selectivity is the recommended term in analytical chemistry to express the extent to which a particular method can determine analyte(s) in the presence of interferences from other components. Selectivity can be graded. The use of the term specificity for the same concept is to be discouraged as this often leads to confusion.

Linearity

The ability of a method of analysis, within a certain range, to provide an instrumental response or results proportional to the quantity of analyte to be determined in the laboratory sample. This proportionality is expressed by an a priori defined mathematical expression. The linearity limits are the experimental limits of concentrations between which a linear calibration model can be applied with a known confidence level (generally taken to be equal to 1%).

ASSESSMENT OF THE ACCEPTABILITY OF THE PRECISION CHARACTERISTICS OF A METHOD OF ANALYSIS

The calculated repeatability and reproducibility values can be compared with existing methods and a comparison made. If these are satisfactory then the method can be used as a validated method. If there is no method with which to compare the precision parameters then theoretical repeatability and reproducibility values can be calculated from the Horwitz equation.

Horwitz trumpet and equation is $RSD_R = 2C^{-0.1505}$

The values derived from this equation are:

Concentration ratio	RSD _R		Concentration ratio	RSD _R
1 (100%)	2		10 ⁻⁵	11
10 ⁻¹	2.8		10 ⁻⁶ (ppm)	16
10 ⁻² (1%)	4		10 ⁻⁷	23
10 ⁻³	5.6		10 ⁻⁸	32
10 ⁻⁴	8		10 ⁻⁹ (ppb)	45

Horwitz has derived the equation after studying the results from many (~3,000) collaborative trials. Although it represents the average RSD_R values and is an approximation of the possible precision that can be achieved, the data points from “acceptable” collaborative trials lie within a range of one half to twice the values derived from the equation. This idealised smoothed curve is found to be independent of the analyte, method, matrix, laboratory and time (state of the art). In general the values taken from this curve are indicative of the precision that is achievable and acceptable of an analytical method by different laboratories. Its use provides a satisfactory and simple means of assessing method precision acceptability.

It may be conveniently demonstrated for any particular method/concentration combination by calculating the HORRAT values, i.e.

The HORRAT value for reproducibility = RSD_R (observed)/RSD_R (predicted)

The HORRAT value for repeatability is calculated similarly using the observed RSD_r in the numerator and assuming the predicted RSD_r = 0.66 RSD_R, i.e.

HORRAT_r = RSD_r (observed)/RSD_r (predicted)

VALUES LESS THAN 120 µG/KG

It should be noted that the equation has been recalculated in the light of recent collaborative trials. This has now been published by Thompson¹, who recommends that for values less than 120 µg/kg, the a constant value for the relative standard deviation of 22% is used. However, for many purposes, e.g. mycotoxins and pesticide residues, the original form is still applicable in many cases.

REFERENCE

1. “Recent Trends in Inter-Laboratory Precision at ppb and sub-ppb Concentrations in Relation to Fitness for Purpose Criteria in Proficiency Testing”, M. Thompson, *Analyst*, 2000, **125**, 385-386.

HARMONIZED IUPAC GUIDELINES FOR THE USE OF RECOVERY INFORMATION IN ANALYTICAL MEASUREMENT

(Recommended to the Commission for adoption by reference)

The *Harmonized IUPAC Guidelines for the Use of Recovery Information in Analytical Measurement* are recommended for adoption for Codex purposes by the 24th Session of the Commission with the exception of the first two sentences of Recommendation 1³.

Recommendation 1 of the IUPAC Guidelines should therefore read as follows:

It is of over-riding importance that all data, when reported, should (a) be clearly identified as to whether or not a recovery correction has been applied and (b) if a recovery correction has been applied, the amount of the correction and the method by which it was derived should be included with the report. This will promote direct comparability of data sets. Correction functions should be established on the basis of appropriate statistical considerations, documented, archived and available to the client.

Reference

Harmonised Guidelines for the Use of Recovery Information in Analytical Measurement (Pure Appl. Chem., Vol. 71, pp. 337 – 348, 1999)

³ *“Quantitative analytical results should be corrected for recovery unless there are specific reasons for not doing so. Reasons for not estimating or using correction factors include the situations where (a) the analytical method is regarded as empirical, (b) a contractual or statutory limit has been established using uncorrected data, or (c) recoveries are known to be close to unity. However,.....”*

STATUS OF ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING

This Appendix consists of three sections:

Part I Methods of Analysis for Commodity Standards

Part II. Methods of Sampling for Commodity Standards

Part III. General Methods of Analysis

PART I . METHODS OF ANALYSIS FOR COMMODITY STANDARDS

A. CODEX COMMITTEE ON SUGARS

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Sugars (white sugar, plantation or mill white sugar, soft white sugar, soft brown sugar, powdered sugar, powdered dextrose, raw cane sugar)	Sulphur dioxide	ICUMSA GS 2-33 (1994)	Rosaniline procedure		III	E
Sugars (powdered sugar)	Polarisation	ICUMSA (1994) GS 2/3-1	Polarimetry	Corrected reference to the method	II	E
Sugars (fructose)	Conductivity ash	ICUMSA GS 2/3-17 (1994)	Conductimetry		I	E
Sugars (soft sugars, brown sugar)	Invert sugar	ICUMSA GS 1/3/7-3	Titrimetry (Lane & Eymon)		I	E
Sugars (plantation or mill white sugar)	Invert sugar	ICUMSA GS 2-6 (1998)	Titrimetry		I	E

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Sugars (soft white sugar, powdered sugar, plantation or mill white sugar)	Colour	ICUMSA GS 2/3-10 (1998)	Photometry	Corrected reference to the method. It was noted that the Commodity Committee recommended this method even though some members of the trade objected.	I	E
Honey	Sample preparation	AOAC 920.180			I	
Honey	Moisture content	AOAC 969.38B	Refractometry	Both methods cited are identical	I	E
Honey	Fructose and Glucose (sum of both)	MAFF validated method V21				
Honey		Harmonised method of the EHC, Apidologie, Special Issue 28, 1997, Chapter 1.7..2	HPLC	The Commodity Committee is requested to verify that a collaborative study has been performed on this method.	II	TE
Honey	Sucrose content	Harmonised method of the EHC, Apidologie, Special Issue 28, 1997, Chapter 1.7.2	HPLC	The Commodity Committee is requested to verify that a collaborative study has been performed on this method.	II	TE
Honey	Sugars added: for sugar profile	AOAC 998.18	Carbon isotope ratio mass spectrometry	AOACI has replaced method 991.41 with AOAC 998.18. It was also noted that at the last session method AOAC 978.17 was endorsed for honey (sugars added) and has been replaced with AOAC 998.12.	I	E
Honey	Water-insoluble solids content	MAFF validated method V22, J A Public Analyst 1992, 28(4) 189-193??	Gravimetry		I	E
Honey	Electrical conductivity	Harmonised method of the EHC, Apidologie, Special Issue 28, 1997, Chapter 1.2		The Commodity Committee is requested to verify that a collaborative study has been performed on this method.	I	TE
Honey	Acidity	MAFF Validated method V19, J A Public Analyst 1992, 28(4) 171-175.	Titrimetry		I	E

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Honey	Diastase activity	Phadebas – Harmonised method of the EHC	Enzyme	The Commodity Committee is requested to verify that the reagents for the method are available, and a collaborative study has been performed on this method and to provide a method reference.	III	TE
Honey	Hydroxymethylfurfural	Harmonised method of the EHC	HPLC	The Commodity Committee is requested to verify that a collaborative study has been performed on this method and to provide a method reference.	III	TE
B. Codex Committee on Fish and Fishery Products						
1. Methods referred back to CCCFFP						
Quick Frozen Fish Sticks (fish fingers) and Fish Portions-Breaded and in Batter(except for certain fish species with soft flesh)	Proportion of fish fillet and minced fish	WEFTA Method	Gravimetry	The Commodity Committee is requested to provide the reference to the published method.	I	E
Quick Frozen Fish Sticks (fish fingers) and Fish Portions-Breaded and in Batter	Proportion of fish flesh in fish sticks (fish core)	AOAC 996.15 (with an adjustment factor of 2% for raw breaded and batter-dipped products; 4% for precooked products)	Gravimetry	AOAC 996.15 is a modified method of AOAC 971.13 which was endorsed previously..	I	E
Salted Fish of the <i>Gadidae</i> Family	Salt	WEFTA Method	Titrimetry (Mohr)	The Commodity Committee is requested to provide method reference.	II	E
			Salt determined as chloride expressed as sodium chloride			

2. Methods proposed in standards under elaboration

Fish and Fishery Products

Histamine

AOAC 977.13

Fluorimetry

II

E

C. CODEX COMMITTEE ON MILK AND MILK PRODUCTS

1. Methods of analysis referred back to CCMMMP (adopted standards)

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	Type	Status
Cheese	Solids	IDF Standard 4A:1982	Gravimetry, drying at 102 °C	The Working Group was interested in obtaining information on the difference in results between the previous method and this newly adopted method.	I	E
		ISO 5534:1985				
Cheese	Moisture	IDF Standard 4A:1982	Gravimetry, drying at 102 °C	The Working Group was interested in obtaining information on the difference in results between the previous method and this newly adopted method.	I	E
		ISO 5534:1985				
Sweetened Condensed Milks	Solids	IDF Standard 15B:1991	Gravimetry, drying at 102 °C	The Working Group was interested in obtaining information on the difference in results between the previous method and this newly adopted method.	I	E
		ISO 6734:1989				
Milkfat Products	Peroxide value (expressed as milliequivalents of oxygen/kg fat)	AOAC 965.33	Titrimetry		I	E
Evaporated Milks	Protein	AOAC 945.48H	Kjeldahl, titrimetry		I	E
Sweetened and Condensed Milks		AOAC 991.20 – IDF 20B:1993				
Milkfat Products	Copper	IDF Standard 76A:1980/ISO 5738:1980/AOAC 960.40	Photometry, diethylidithiocarbamate	The Commodity Committee was encouraged to consider adopting one of the general methods for copper.	II	E
Butter						

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	Type	Status
Whey Cheese	Dry matter (for denomination)	IDF Standard 58:1970 (confirmed 1993) ISO 2920:1974	Gravimetry, drying at 88 °C	.	I	E
Whey Powders	Lactose	IDF 79B:1991	Enzymatic method: glucose moiety (method A), galactose moiety (method B)		II	E

2. Methods of analysis proposed for standards under elaboration (advanced to Step 5 or 8)

Cheeses, individual	Dry matter (specified in individual standards)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	The Working Group was interested in obtaining information on the difference in results between the previous method and this newly adopted method	I	E
Cheeses, individual	Milkfat in dry matter	IDF Standard 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid-Bondzynski-Ratslaff)		I	E
Creams, Whipped Creams and Fermented Creams	Milk solids-not-fat	IDF Standard 80:1977 ISO 3727:1977 AOAC 920.116	Gravimetry		I	E
Creams, Whipped Creams and Fermented Creams	Milk solid-not-fat	IDF Standard 11A:1986	Gravimetry	The Commodity Committee should review to assure that this method is appropriate since the cited method is for milk solids in butter not cream.		NE

Cream	Milkfat	IDF Standard 16C:1987 ISO 2450:1999 AOAC 995.19	Gravimetry	I	E
Creams Lowered in Milkfat Content	Milkfat	IDF Standard 16C:1987 ISO 2450:1999 AOAC 995.19	Gravimetry	I	E
Edible Casein Products	Casein in protein	IDF Standard 29:1964	Titrimetry, Kjeldahl	I	E
Fermented Milks	Lactic acid	IDF Standard 150:1991 ISO 11869:1997	Potentiometry	I	NE
Fermented Milks	Lactic acid	AOAC 937.05	Spectrophotometric (for lactate acid in milk & milk products)		NE
Fermented Milks	Protein	IDF Standard 20B:1993 AOAC 991.20-23	Titrimetry (Kjeldahl)	I	E
Fermented Milks	Dairy starter cultures of lactic acid bacteria (LAB)	IDF Standard 149A:1997 (Annex A)	Colony count at 25 °C, 30 °C, 37 °C & 45 °C according to the starter organism in question	I	TE
Fermented Milks (Yoghurt)	<i>Streptococcus thermophilus</i> & <i>Lactobacillus delbrueckii</i> subsp. <i>Bulgarius</i>	IDF Standard 117B:1997	Colony count at 37°C	I	TE
	>= 10 ⁷ cfu/g				The Commodity Committee should provide information on whether a collaborative study has been performed and the type of the method.

Fermented Milks (Yoghurt)	<i>Streptococcus thermophilus</i> ^{CS} <i>Lactobacillus delbrueckii</i> subsp.:bulgaricus ≥= 10 ⁷ cfu/g	IDF Standard 146A:1998	Test for identification	The Commodity Committee should provide information on whether a collaborative study has been performed and the type of the method.	I	TE
Milk Products obtained from Fermented Milks Heat-Treated after Fermentation	Protein	IDF Standard 20B:1993 ISO 8968 Part I AOAC 991.20-23	Titrimetry (Kjeldahl)		I	E
Unripened Cheese Including Fresh Cheese ⁴	Dry matter [not decided (unripened/ fresh cheese)] ≥= 35 % (cream cheese)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C			NE
Unripened Cheese Including Fresh Cheese ¹	Dry matter [not decided (unripened/ fresh cheese)] ≥= 35 % (m/m), < Restricted by the MMFB	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C			NE
Unripened Cheese Including Fresh Cheese ¹	Dry matter [not decided (unripened/ fresh cheese)] ≥= 35 % (cream cheese)	AOAC 926.08	Gravimetry, vacuum oven			NE
Unripened Cheese Including Fresh Cheese ¹	Protein	IDF Standard 20B:1993 AOAC 991.20-23 ISO 8968 Part I	Titrimetry, Kjeldahl		I	E

⁴ The CCMMP decided to remove these provisions at their last session therefore method endorsement is unnecessary

D. CODEX COMMITTEE ON COCOA PRODUCTS AND CHOCOLATE

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Cocoa (Cacao) Mass, Cocoa/Chocolate (liquor) and Cocoa Cake	Fat content	AOAC 963.15 or IOCCC 14 (1972)	Sohxlet extraction		I	E
Cocoa powders (cocoas) and dry mixtures of cocoa and sugars	Cocoa butter	IOCCC 37 (1990) and IOCCC 14 (1972)	Total fat and total sterol content by GLC	The Commodity Committee is requested to provide evidence of collaborative study validation and information regarding the purpose and type of the method.		NE
Chocolate and Chocolate Products	Copper	NMKL 139 AOAC 971.20	Atomic absorption Spectrophotometry	The Commodity Committee should consider adopting one of the Codex general methods for copper that will handle the % fat in their product.		NE

E. CODEX COORDINATING COMMITTEE FOR ASIA AND CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

Aqueous Coconut Products	Total solids	AOAC 925.23A (IDF-ISO-AOAC method)	Gravimetry	AOAC 925.23kA has been repealed and the Commodity Committee should provide information on validation of the cited method for this application.	I	NE
Aqueous Coconut Products	Total Fats	AOAC 945.48G	Gravimetry (Röse-Gottlieb method)	The Commodity Committee should provide information on validation of the cited method for this application.	I	NE

F. CODEX COMMITTEE ON SOUPS AND BROTHS

Soups and Broths	Creatinine	AHBP Method 2/5 (2000)	HPLC	II	E
Soups and Broths	Creatinine	AOAC 920.115	Colorimetry	NE	Based on the recommendation of the Commodity Committee this method should be deleted.
Soups and Broths	Total Nitrogen	AOAC 928.08	Kjeldahl	II	E
Soups and Broths	Sodium Chloride	AHBP No. 2/4	Potentiometric titration	II	E

G. CODEX COMMITTEE ON VEGETABLE PROTEINS

Wheat Protein Products	Vital wheat gluten and devalitized wheat gluten		AOAC 979.09 (Wheat protein in grain N x 5.7)	Kjeldahl	I	E
Wheat Protein Products	Solubilized wheat protein		AOAC 920.87 (wheat protein in flour N x5.7)	Kjeldahl	I	E

H. COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Guidelines for Nutrition Labelling	Saturated fat	AOAC 996.06	Gas liquid chromatography		II	E

I. CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

Pickles	Benzoic acid	ISO 5518:1978	Spectrophotometry		IV	TE
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The Commodity Committee is requested to review more modern methods such as the liquid chromatographic method IFU 63 (1995) or the gas chromatographic method NMKL 103 (1984)/AOAC 983.16 which has been endorsed as ^a Type II Codex general method.

Sampling of Cheese in Brine

(Amendment at Step 3 of the Accelerated Procedure)

Amend Section 8.1 of the Codex Standard for Cheeses in Brine (CODEX STAN 208-1999) concerning sampling as follows (struck-out text to be deleted):

8.1 *Sampling*

According to IDF Standard 50C:1995/ISO 707:1997/AOAC 933.12. - Endorsed

Special requirements for cheese in brine: A representative piece of cheese is placed on a cloth or on a sheet of ~~non~~-absorbent paper for 5 to 10 min. A slice of 2-3 cm is cut off and sent to the laboratory in a sealed insulated box for analysis.

B. COMMITTEE ON VEGETABLE PROTEINS

Draft Standard for Wheat Protein Products

Sampling: ISO 13690: 1999 - *Endorsed*

PART III GENERAL CODEX METHODS

1. Methods for contaminants

All foods	Lead, cadmium, copper, iron and zinc	NMKL 139 (1991) AOAC 999.11	AAS after dry ashing	Type II	II	E
All foods	Lead, cadmium, copper, iron and zinc	NMKL 161 (1998) AOAC 991.10	AAS after microwave digestion	Type III	III	E

2. Methods for the detection of irradiated foods

Food containing fat	detection of irradiated food containing fat	EN 1784:1996	Gas chromatographic analysis of hydrocarbons		II	E
Food containing fat	detection of irradiated food containing fat	EN 1785:1996	Gas chromatographic/ spectrophotometric analysis of 2/alkylcyclobutanones		III	E
Food containing bone	detection of irradiated food containing bones	EN 1786:1996	ESR spectroscopy		II	E
Food containing cellulose	detection of irradiated food containing cellulose	EN 1787:2000	ESR spectroscopy		II	E
Food from which silicate minerals can be isolated	detection of irradiated food from which silicate minerals can be isolated	EN 1788:1996	Thermoluminescence		II	E

PROPOSED DRAFT GUIDELINES ON MEASUREMENT UNCERTAINTY
(At Step 3 of the Codex Procedure)⁵

Introduction

It is important that analysts are aware of the uncertainty associated with each analytical result and estimates that uncertainty. The measurement uncertainty may be derived by a number of procedures. Food analysis laboratories are required, for Codex purposes, to be in control, use collaboratively tested methods when available, and verify their application before taking them into routine use. Such laboratories therefore have available to them a range of analytical data which can be used to estimate their measurement uncertainty.

Terminology

The accepted definition for Measurement Uncertainty¹ is:

"Parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand.

NOTES:

1. The parameter may be, for example, a standard deviation (or a given multiple of it), or the half-width of an interval having a stated level of confidence.
2. Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of results of a series of measurements and can be characterised by experimental standard deviations. The other components, which can also be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information.
3. It is understood that the result of a measurement is the best estimate of the value of a measurand, and that all components of uncertainty, including those arising from systematic effects, such as components associated with corrections and reference standards, contribute to the dispersion.

[It is recognised that the term "measurement uncertainty" is the most widely used term by International Organisations and Accreditation Agencies. However The Codex Committee on Methods of Analysis and Sampling has commented on a number of occasions that the term "Measurement Uncertainty" has some negative associations in a legal context and so has noted that an alternative, equivalent, term, "measurement reliability", may be used.]

Recommendations

The following recommendations are made to governments:

1. For Codex purposes the term "measurement uncertainty" [or "measurement reliability"] shall be used.
2. The measurement uncertainty [or "measurement reliability"] associated with all analytical results is to be estimated and must, on request, be made available to the user (customer) of the results.
3. The measurement uncertainty [or "measurement reliability"] of an analytical result may be estimated in a number of procedures, notably those described by ISO¹ and EURACHEM². These documents recommend procedures based on a component-by-component approach, method validation data, internal

⁵ Subject to approval as new work by the Commission.

quality control data and proficiency test data. The need to undertake an estimation of the measurement uncertainty [or "measurement reliability"] using the ISO component-by-component approach is not necessary if the other forms of data are available and used to estimate the uncertainty [or reliability]. In many cases the overall uncertainty may be determined by an inter-laboratory (collaborative) study by a number of laboratories and a number of matrices by the IUPAC/ISO/AOAC INTERNATIONAL³ or by the ISO 5725 Protocols⁴.

References

1. "Guide to the Expression of Uncertainty in Measurement", ISO, Geneva, 1993.
2. EURACHEM/CITAC Guide Quantifying Uncertainty In Analytical Measurement (Second Edition), EURACHEM Secretariat, BAM, Berlin, 2000. This is available as a free download from <http://www.vtt.fi/ket/eurachem>
3. "Protocol for the Design, Conduct and Interpretation of Method Performance Studies", ed. W. Horwitz, *Pure Appl. Chem.*, 1995, 67, 331-343.
4. "Precision of Test Methods", Geneva, 1994, ISO 5725, Previous editions were issued in 1981 and 1986.