codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

JOINT OFFICE: Viale delle Terme di Caracalla 00153 ROME Tel: 39 06 57051 www.codexalimentarius.net Email: codex@fao.org Facsimile: 39 06 5705 4593

ALINORM 09/32/23

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

Thirty-second Session
Rome, Italy, 29 June- 4 July 2009

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

Balatonalmádi, Hungary
9 - 13 March 2009

Note: This document incorporates Codex Circular Letter CL 2009/12-MAS

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CX 4/50.2 **CL 2009/12-MAS**
April 2009

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 30th Session of the Codex Committee on Methods of Analysis and Sampling (ALINORM 09/32/23)**

A. MATTERS FOR ADOPTION BY THE 32nd SESSION OF THE CODEX ALIMENTARIUS COMMISSION

Draft Guidelines at Step 8

1. Draft Guidelines for Settling Disputes on Analytical (Test) Results (para. 25, Appendix II)

2. Draft Guidelines on Analytical Terminology (para. 43, Appendix III)

Methods of Analysis and Sampling

3. Methods of Analysis in Codex Standards at different steps (paras. 46-82, Appendix IV)

Proposed Amendments to the Procedural Manual

4. Proposed Amendment to the *Working Instructions for the Implementation of the Criteria Approach in Codex* (para. 92, Appendix V)

5. Proposed Amendment to the *General Criteria for the Selection of Methods of Analysis* (consequential amendment on terminology) (para. 44, Appendix VI)

Governments wishing to propose amendments or comments on items 1 to 5 above 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 above address **before 30 May 2009.**

B. REQUEST FOR COMMENTS AND INFORMATION

6. Methods of Analysis for Certain Substances in Natural Mineral Waters (para. 8)

Information is requested on methods of analysis and sampling for the substances mentioned in Section 3.2 of the *Standard for Natural Mineral Waters*, especially Sections 3.2.17 to 3.2.20: surface active agents, PCBs, mineral oil, and polynuclear aromatic hydrocarbons (para. 8).

Governments and international organizations wishing to submit information and comments should do so in writing to the above address, with a copy to the Codex Contact Point of Hungary, Hungarian Food Safety Office, H-1097 Gyáli út 2-6. Budapest Hungary, Fax: +36 1 387 94 00, e-mail: HU_CodexCP@mebih.gov.hu **before 15 October 2009**.

SUMMARY AND CONCLUSIONS

The summary and conclusions of the 30th Session of the Codex Committee on Methods of Analysis and Sampling are as follows:

Matters for adoption by the 32nd Session of the Commission:

The Committee:

- advanced to Step 8 the Draft Guidelines for Settling Disputes on Analytical (Test) Results (para. 25, Appendix II);
- advanced to Step 8 the Draft Guidelines on Analytical Terminology (para. 43, Appendix III)
- endorsed or updated the status of several methods of analysis in Codex standards (paras. 46-82, Appendix IV);
- agreed to propose an amendment to the *Working Instructions for the Implementation of the Criteria Approach in Codex* in the Procedural Manual (para. 92, Appendix V)
- agreed to propose a consequential amendment on terminology to the *General Criteria for the Selection of Methods of Analysis* (para. 44, Appendix VI);
- agreed to discontinue work on the Draft Guidelines for Evaluating Acceptable Methods of Analysis (para.18).

Other Matters of Interest to the Commission

The Committee:

- agreed to return to Step 2/3 the Proposed Draft Guidelines on Criteria for Methods for the Detection and Identification of Foods Derived from Biotechnology (para. 108);
- agreed to return to Step 2/3 the Proposed Draft Revised Guidelines on Measurement Uncertainty (para. 121);
- agreed to consider further at its next session guidance on uncertainty of sampling (para. 108); and the methods of analysis for natural mineral waters (para. 8).

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LIST OF APPENDICES

INTRODUCTION

1) The Codex Committee on Methods of Analysis and Sampling held its Thirtieth Session in Balatonalmádi, Hungary, from 9 to 13 March 2009, by courtesy of the Government of Hungary. The Session was chaired by Professor Árpád Ambrus, Deputy Director General, Hungarian Food Safety Office. Dr Béla Kovacs, Professor, University of Debrecen, acted as the Vice-Chairperson. The Session was attended by 140 delegates and observers representing 48 Member Countries, one Member Organisation (EC) and 9 international organizations.

OPENING OF THE SESSION

2) The Session was opened by Dr Zoltán Gyaraky, Head of Department, Ministry of Agriculture and Rural Development, who recalled the strong support of Hungary for the work of Codex and stressed the importance of international standards for food safety and quality in a globalised environment. He highlighted the recent changes in food legislation and food control in Hungary and the importance of food production and processing for its economy. Dr Gyaraky pointed out that the present session had a very full agenda addressing various issues arising from other Codex committees and general questions such as measurement and sampling uncertainty. He noted that the increasing number of delegates participating in the Committee throughout the years reflected the relevance and importance of the work on methods of analysis and sampling, and wished delegates all success in their work.

ADOPTION OF THE AGENDA (Agenda Item 1)[1]

3) The Committee agreed with several proposals of some delegations as follows:

- To establish an in-session working group, working in English, in order to facilitate the discussion of Agenda Item 3(b): Draft Guidelines for Setting disputes over Analytical (Test) results, to consider the comments received and prepare a revised version for consideration by the plenary session.
- To change the order of the agenda and to discuss Agenda Item 6 (Guidelines on Establishing Methods Criteria for the Identification of Relevant Analytical Methods (Conversion of Methods for Trace Elements into Criteria)) before Item 3(a) (Draft Guidelines for Evaluating Acceptable Methods of Analysis); and Agenda Item 7 (Proposed Draft Guidelines on Criteria for Methods for the Detection and identification of Foods Derived from Biotechnology) following Item 3.
- To discuss a new subject, Defining Method(s) for the Analysis of Melamine in Food and Feeds (CRD 15), under Agenda Item 13 'Other Business and Future Work', as proposed by the Delegation of Nigeria.
- To consider the updating of references in many Codex documents, together with Agenda Item 5, Endorsement of Methods of Analysis Provisions in Codex Standards, if relevant for the methods under consideration for endorsement, and otherwise to discuss the proposal under Agenda Item 13 'Other Business and Future Work', as proposed by the Delegation of Brazil.

4) The Committee adopted the Provisional Agenda as its Agenda for the Session with these amendments.

5) The Delegation of the European Community presented CRD 3 on the division of competence between the European Community and its Member States according to Rule of Procedure II.5 of the Rules of Procedure of the Codex Alimentarius Commission.

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

6) The Committee noted that matters arising from the Commission were for information purposes only or would be discussed in more detail under the relevant Agenda Items. Observations made are summarized as follows:

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1 CX/MAS 09/30/1, CRD 15 (comments of Nigeria)

2 CX/MAS 09/30/2, CRD 12 (comments of European Community),

Standard for Food Grade Salt (CODEX STAN 150-1984)

7) The Delegation of the European Community, noting that, in the Codex Standard for Food Grade Salt, there were four reference to document CX/MAS 1-1987, which was not a Codex text hence was not easily accessible for the users of the Standards, suggested that all reference to CX/MAS 1-1987 be replaced with a reference to the General Guidelines on sampling (CAC/GL 50-2004). The Committee agreed to check whether the definitions related to the sampling of food grade salt were available in other Codex documents. After some discussion the Committee agreed to replace the current references with the reference to the General Guidelines on Sampling.

Standard on Natural Mineral Waters (CODEX STAN 108-1981)

8) The Committee noted that the issue on the determination of PCBs would be discussed under Item 11. The Committee agreed that the Secretariat would prepare a Circular Letter to ask members to provide information on methods and sampling currently used by members and views on the need for development of appropriate methods, for discussion at its next session.

Aflatoxin Sampling Plan for Almonds, Hazelnuts and Pistachio

9) The Delegation of the European Community, referring to its written comment in CRD 12 regarding paragraph 10 of the Aflatoxin Sampling Plan for Almonds, Hazelnuts and Pistachio as incorporated in Codex General Standard for Contaminants and Toxins in Foods (GSCTF) (CODEX STAN 193-1995), proposed to replace the text ”dry grind with vertical cutter mixer type mill and a 50 g test portion” to ”such that each laboratory sample shall be finely ground and mixed thoroughly using a process that has been demonstrated to provide the lowest sample preparation variance.” and amend the text for Decision Rule to read “If the aflatoxin test result corrected for recovery is less than or equal to 15ng/g total aflatoxin, taking into account the measurement uncertainty, then accept the lot”. Those proposals were also applied to amend the similar texts for Aflatoxin for ready-to-eat treenuts. One error was corrected, replacing RSD_r with RSD_R on the last row of recommended values in Table 2.

10) The Delegation of Iran asked for clarification of the above addition on measurement uncertainty and wondered whether this might significantly impact on the Aflatoxin Sampling Plan and the maximum levels for aflatoxins in Almonds, Hazelnuts and Pistachio in the GSCTF. The Delegation noted that Annex I of the Aflatoxin Sampling Plan addressed analytical variance only.

11) After some discussion, the Committee agreed to refer back to the Committee on Contaminants in Foods the consideration of the above proposed amendments and to ask whether the Use of Analytical Results: Sampling Plans, Relationship between the Analytical Results, the Measurement uncertainty, Recovery Factors and Provisions in Codex Standard[3] had been duly taken into account in the Aflatoxin Sampling Plan.

Standard for Sugars: Method for Determination of Colour in Plantation and Mill White Sugar

12) The Committee noted that this matter would be considered under Agenda Item 5.

CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS (Agenda Item 3)

DRAFT GUIDELINES FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS (Agenda Item 3a)[4]

13) The Committee recalled that the Draft Guidelines had been redrafted twice by a working group led by New Zealand, the results of which were considered at its 28th and 29th Sessions, and that the Draft Guidelines had been retained at Step 7 pending the publication of scientific papers reflecting the approach proposed by New Zealand for the evaluation of acceptable methods.

14) The Delegation of New Zealand informed the Committee that the paper on ”Allowing for imprecision in experimental estimates of measurement uncertainty”, which applied the concept of tolerance intervals to the evaluation of test methods, had not been accepted for publication; however this should not delay the work of the Committee in the development of Guidelines based on well established techniques. The Delegation pointed out that the primary requirement of an analytical method is its fitness for purpose and that

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3 Procedural Manual of the Codex Alimentarius Commission.

4 CX/MAS 09/30/03, CRD12 (comments of the European Community)

any decision on the performance of a method should be based on how the performance of the method affects the assessment of conformity, and proposed to develop principles for compliance assessment and a procedure to assess fitness for purpose. The Delegation highlighted the limitations of the current criteria under consideration, which could result in the rejection of suitable methods or acceptance of unsuitable methods, and proposed to proceed with the Draft Guidelines taking into account the work on the criteria in CX/MAS 09/30/7 and the draft pepared for the 28th Session in CX/MAS 07/28/3. The Delegation proposed that the Committee should note the apparent risks of the current criteria and the need for further work including the development of principles for compliance assessment of foods; the revision of the Codex Guidelines for method performance studies; the revision of the Working Instructions for the Implementation of the Criteria Approach; and the revision of the Guidelines mentioned above.

15) Several delegations expressed the view that the Committee should not proceed with the development of the guidelines as the paper presented at the session was not structured in the form of guidelines but a discussion paper.

16) The Committee recalled that work on the evaluation of methods had proceeded on the basis of a document prepared in earlier sessions by the United Kingdom including the conventional approach, which was used as a basis for the Draft Guidelines, and the fitness for purpose approach, on which the Committee had already decided not to proceed further. It was also noted that the fitness for purpose approach could be considered for Type IV methods, and that the criteria approach was applicable for Type II and III methods.

17) One delegation proposed that the work carried out so far should not be lost and that this item and Item 6 on the criteria should be merged or considered jointly in order to avoid duplication. Some other delegations expressed the view that work on the criteria should proceed separately and that new work on the revision of other existing Codex texts could be put forward in the future if needed. In view of the above discusssion, the Committee recognised that there was no support to proceed with the development of Draft Guidlines at this stage.

Status of the Draft Guidelines for Evaluating Acceptable Methods of Analysis

18) The Commttee agreed to propose to the 32nd Session of the Commision to discontinue work on the Draft Guidelines.

DRAFT GUIDELINES FOR SETTLING DISPUTES OVER ANALYTICAL (TEST) RESULTS (Agenda Item 3b)[5]

19) The Committee recalled that its last session had had an extensive discussion and made significant changes and agreed to return the Draft Guidelines to Step 6 for further comments and consideration at Step 7, with a view to its finalization at the present session.

20) The Committee considered the document in CRD 22 prepared by the in-session physical working group, held during the present session to make the text simple and more precise, reflecting the comments submitted. The Delegation of the Netherlands, speaking as Chair of the in-session physical working group, pointed out that the Guidelines address how to deal with disputes related to test results, but do not address questions of sampling and that the text in Section 3 was amended to allow flexibility in solving disputes. It was also noted that these guidelines should be applied in the situation where both importing and exporting countries were in agreement to use them.

21) Observation and amendments made are summarized as follows:

Section 2: Prerequisites/assumptions

22) In the third bullet, it was agreed to add a footnote to state that "at least one representative sample" might refer to a set of samples when more than one sample was involved, noting that throughout the Guidelines, the word sample could refer to more than one sample. With regard to reserve samples, although they were primarily required to be split into three essentially identical parts for confirmatory analysis, it was agreed to add a footnote to allow flexibility to split the sample into only two identical parts.

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[5] CL 2008/7-MAS, ALINORM 08/31/23 Appendix IV), CX/MAS 09/30/4 (comments of Brazil, Cuba, European Community, New Zealand), CRD 5 (comments of Kenya), CRD 22 (Report of the in-session physical working group)

Section 4: Analysing reserve sample

23) The Committee noted that the physical working group had removed a reference to "two laboratories" in point 2 to allow having two results from one laboratory. The Delegation of Brazil, referring to its written comments in CX/MAS 09/30/4, proposed to amend the second paragraph to give an option allowing comparison of results from two different samples. This proposal was not accepted as it was noted that comparison of results from different samples would not allow to solve disputes and in this case measurement uncertainty was not applicable.

Annex

24) The term "laboratories" was replaced with "results" in the first sentence. It was agreed to include the following text: "in case as set of samples is involved a different formulation for the critical difference should be used" to address cases where results from more than one sample are compared. A request for insertion of one example regarding measurement uncertainties was not accepted as it was noted that a reference to measurement uncertainties existing elsewhere in Codex documents might help understanding this issue.

Status of the Draft Guidelines for Setting Disputes Over Analytical (Test) Results

25) The Committee agreed to forward the Draft Guidelines as amended above, with some minor editorial changes, to the 32nd Session of the Commission for adoption at Step 8.

26) The Delegation of Brazil expressed its reservation to the decision mentioned in para. 23, first sentence, as the decision to eliminate the mention of two laboratories created practical implications in laboratories and needed to be better evaluated.

DRAFT GUIDELINES ON ANALYTICAL TERMINOLOGY (Agenda Item 4)[6]

27) The Committee recalled that the Draft Guidelines had been adopted at Step 5 by the 31st Session of the Commission and circulated for comments at Step 6. The Committee considered the document section by section and made a number of amendments and comments.

28) The Delegation of Brazil proposed to replace the definition of Bias with the VIM definition as it was clearer and more practical to apply, taking into account that the true value was not known. Other delegations pointed out that the note on Bias specified that "in practice the accepted reference value is substituted for the true value", that the expectation was also clarified in the notes and therefore the definition should be retained as the reference to the true value was clearly explained.

29) The Delegation of the United States also recalled that the terminology had been revised on the following basis: the definitions in the Procedural Manual were retained where possible, as in the case of bias, the revision had integrated the definitions from ISO Standard 3534-2 and, when these were not available, the VIM definitions.

30) After some further discussion, the Committee agreed to retain the current definition of Bias with an additional sentence indicating that "in practice the true value may be substituted with the conventional quantity value" and to delete the note referring to the "accepted reference value". The definition of the Conventional Quantity Value, as included in the VIM, was also added to the guidelines.

31) The Delegation of New Zealand pointed out that the consequences of introducing the Critical Value should be considered carefully in terms of the acceptability of methods of analysis and proposed to delete the notes relating to the estimation as they were not soundly based from the statistical point of view. The Delegation also indicated that the quantity Lc used in the equation for Limit of Detection was not the Lc used under Critical Value and proposed to review the notation in order to avoid confusion.

32) The Committee was informed that the notations used in the definitions might be reconsidered in the framework of ISO TC 69 in the future and agreed that the current notations should be retained in the Limit of Detection at this stage.

33) The Committee discussed whether the Critical Value could be retained as a separate definition or integrated into the Limit of Detection as it was used only in the framework of that definition. After some

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[6] ALJNORM 08/ 31/23, Appendix V, CL 2008/28-MAS, CX/MAS 09/30/5 (comments of Australia, Brazil, Cuba, Iran, Japan, Kenya, New Zealand, Portugal, United States), CRD12 (comments of the European Community), CRD 13 (comments of Chile), CRD 16 (comments of the Republic of Korea)

discussion, the Committee agreed to add a note clarifying that "the Critical Value is important to determine the Limit of Detection" and to retain the definition of Critical Value in view of its importance to define the Limit of Detection.

34) The Committee agreed to retain the abbreviations LOD and LOQ for Limit of Detection and Limit of Quantification as they were widely used, instead of L_D and L_Q.

35) In the HorRat definition, the Committee agreed to insert the actual value of the predicted relative standard deviation (22%) in the last sentence regarding concentrations less than 0.12 mg/kg.

36) In the definition of Recovery, it was agreed to delete the reference to the extraction as the recovery was applicable to the analytical procedure as a whole

37) The Committee agreed to insert a note to the effect that the Repeatability (Reproducibility) Relative Standard Deviation is also known as Coefficient of Variation, as this term is also commonly used.

38) The Committee agreed to replace the definition of Trueness by the VIM definition which was more precise.

39) The Committee agreed to insert the following new definitions: Analyte, as used for the purpose of defining good laboratory practice in pesticide residue analysis[7]; Assay[8] (ISO definition); Run[9], using the ISO definition with a note concerning qualitative run; Measurement Method (VIM definition); and Outliers (ISO definition).

40) The Committee also made several editorial corrections for clarification purposes, to ensure consistency throughout the text, or to update references.

41) The Committee welcomed the proposal of the Delegations of Chile and Cuba for Spanish speaking countries to review the document, in order to ensure that the adequate terminology was used in Spanish. It was also agreed that the Delegation of the United States, the Delegation of Chile and the Codex secretariat would work together to ensure that the symbols and acronyms used for the definitions were preserved in translation.

42) The Committee noted that after the guidelines were finalised, some Codex documents would need to be reviewed to ensure that they were consistent with the revised terminology.

Status of the Draft Guidelines on Analytical Terminology

43) The Committee agreed to advance the Draft Guidelines, as amended at the present session, to Step 8 for adoption by the 32nd Session of the Codex Alimentarius Commission (see Appendix III). It was also agreed that on adoption of the Guidelines, the section on Analytical Terminology in the Procedural Manual would be deleted, as initially agreed when new work on the Guidelines was approved.

Consequential Amendment

44) The Committee noted that a reference to one of the definitions recommended for deletion, "specificity", appeared in the Procedural Manual under *General Criteria for the Selection of Methods of Analysis*, section (b) (i) and agreed that it should be replaced with "selectivity". The Committee agreed to forward this proposed amendment to the Committee on General Principles for endorsement and to the 32nd Session of the Commission for adoption (see Appendix VI).

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS (Agenda Item 5)[10]

45) The report of the *ad hoc* Working Group on Endorsement of Methods of Analysis was presented by its Chair, Dr Roger Wood (United Kingdom). The Committee considered the methods proposed for endorsement and in addition to editorial changes made the following amendments and recommendations.

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7 Guidelines on Good Laboratory Practice in Residue Analiysis (CAC/GL 40-1993)

8 The reference will be provided

9 The reference will be provided

10 CX/MAS 09/30/5, CRD 1 (Report of the Working Group on Endorsement of Methods of Analysis and Sampling), CRD4 (comments of the Republic of Korea), CRD 10 (comments of Thailand)

Committee on Nutrition and Foods for Special Dietary Uses

Standard for Infant Formula and Formulae for Special Medical Purposes Intended for Infants

46) The Committee considered the methods in the above standard, taking into account the replies provided by the Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU) to the questions from the 28th and 29th CCMAS sessions.

47) The Committee agreed to ask the CCNFSDU to clarify the reference to calories only in the provision and calculation of energy and to consider the establishment of the relevant conversion factors for kilojoules, and the method was endorsed as Type I. Some of the methods required for the calculation of calories were listed under the determination of total carbohydrates.

48) For total fat determination, as the Rőse Gottlieb method is applicable only when the formula are completely soluble in ammonia, while the Weinbull-Berntrop method is used when products are not completely soluble in ammonia, the conditions of use were clarified in the Table.

49) As the same method was used both for the determination of trans fatty acids and for fatty acids it was agreed to retain only one entry for fatty acids (including trans fatty acids).

50) The Committee agreed that, although it had not been validated for infant formula, the method for fatty acids had been validated for a broad range of matrices, and therefore was applicable to infant formula. As similar situations occur with several methods, the Committee agreed that general methods can be recommended for individual foods, taking into account the other matrices on which they have been validated.

51) It was noted that the AOCS method applied to the determination of total fatty acid content and had been optimized for trans fatty acids, and that it was already adopted as a Type II method for the purposes of nutrition labelling (saturated fat). The AOAC and AOCS methods were endorsed as Type III.

52) The methods for phospholipids was endorsed as Type III, in view of the above discussion on the applicability of general methods to individual foods

53) All methods used in the calculation of total carbohydrates were listed under a single entry as Type I, consistently with current practice.

54) The AOAC 934.01 method was deleted as it was applicable to animal feeds and the AOAC 925.23 method was replaced with AOAC 990.19 and AOAC 990.20. It was also confirmed that the IDF|ISO method was applicable both to liquid and dried products.

55) For vitamin A the applicability of the methods in relation to the content of Vitamin A was clarified and all methods proposed were endorsed as listed in the Table.

56) For vitamin D, the NMKL and CEN methods were endorsed as Type II, the AOAC methods as Type III, and the forms of Vitamin D measured were specified.

57) The CEN method for vitamin E was endorsed as Type II as it can determine all individual tocopherol congeners and the AOAC method as Type III.

58) For vitamin K, the AOAC and CEN methods were listed together as Type II as they are identical. AOAC 992.27 was deleted as it can detect only trans-K_1 and the standard provides no qualification on the form of vitamin K, therefore the method should detect both cis- and trans- K vitamins.

59) As regards thiamine, AOAC 942.23 was deleted in view of the significant spectral interference and was also deleted from the methods for "special foods". Although such interference also exists with AOAC 986.27, it was retained as Type III due to its current use, with a note that care should be taken in the application of the method due to spectral interference. The CEN method was endorsed as Type II.

60) The Committee noted that the AOAC method for riboflavin was subject to spectral interference but was easier to use than the HPLC CEN method, and retained it as Type III with the same note as in the case of thiamine, and the CEN method was endorsed as Type II.

61) The Committee recalled its earlier recommendation to review microbioassay methods and replace them with more modern methods. The AOAC method for niacin using microbioassay was retained as Type III as it was still used. As the prEN 15652:2007 method using HPLC had not yet been published, it was endorsed as Type II for inclusion after its final publication, to be expected in July 2009.

62) For Vitamin B_6, the AOAC and CEN microbioassay methods were endorsed as Type III. The committee agreed to ask the CCNFSDU whether these microbioassay methods should be retained in view of the earlier recommendation to replace them with more modern methods. As the AOAC 2004.07 and EN 14164:2008 methods are identical, they were listed together as Type II, and the other CEN method as Type III.

63) The Committee noted that AOAC 986.23 for Vitamin B_{12} was validated for infant formula and that no other methods were currently available for the determination of vitamins B_{12}, and therefore endorsed it as Type II.

64) For folic acid, the AOAC and CEN microbioassay methods, which are identical, were listed as Type II because no other validated methods are currently available. As the optical biosensor immunoassay and HPLC methods are still in the phase of collaborative study in AOAC they were listed as Type IV, with the understanding that they could be reconsidered after completion of the studies.

65) The Committee noted the clarification from the CCNFSDU on the expression of vitamin C with the AOAC 985.33 method. As it determines only L(+) ascorbic acid and not the total of ascorbic acid and dehydroascorbic acid, as specified in the standard, it was agreed to delete this method and to retain the CEN method as Type II. Although it was pointed out that the AOAC method is used for quality control purposes, the Committee recalled that the methods selected in Codex standards were intended for use by governments to control compliance, while other methods could be used for quality control.

66) As regards iron, the Committee discussed the approach to be taken when specific methods have been developed for individual foods, in addition to general methods. After some discussion it was agreed to include only the specific methods, which were endorsed as proposed by the CCNFSDU, since the general method was already listed for all foods, and to insert a note indicating that Codex general methods was also available. It was agreed to endorse the methods for iron proposed by the CCNFSDU as Type III.

67) The Committee noted that the IDF|ISO method for calcium was already listed as a Type II method for the determination of potassium and sodium in special foods and it was endorsed as Type II, with the AOAC method as Type III.

68) For chloride, the AOAC 986.26 method was endorsed as Type III since a general method already applied to special foods as Type II.

69) The Committee noted that the methods proposed for selenium had not been validated specifically for infant formula but agreed that they could be used as they had been validated on a wide range of matrices. The CEN method was endorsed as Type II, and the two AOAC methods as Type III.

70) For chromium, the Committee agreed to endorse the EN 14082:2003 method as Type II and the two other methods proposed as Type III. For molybdenum the CEN method was endorsed as Type II and the AOAC method as Type III.

71) As regards the general question on the criteria for the selection of appropriate Type II methods, the Committee informed the CCNFSDU that the methods had been selected on the basis of analytical characteristics, precision, sensitivity, limit of detection, and the scope of the validation of each individual method, which had allowed to assign a Type on a consistent basis to all the methods put forward by the CCNFSDU, and that this was the general approach followed for the typing of methods. The Committee also agreed to encourage Codex committees to follow the criteria approach as an alternative to the selection of specific methods.

Committee on Processed Fruits and Vegetables

Draft Standard for Jams and Jellies

72) The Committee agreed to revoke the methods for calcium and mineral impurities as no provisions exist in the Draft Standard.

Draft Standard for Certain Canned Vegetables

73) The Committee agreed to seek clarification from the Committee on Processed Fruits and Vegetables (CCPFV) as to whether the ISO 762:1982 method currently listed for canned palmito should be retained, and endorsed the AOAC 971.33 method as Type I. All other methods were endorsed or revoked as proposed by the CCPFV.

Standard for Aqueous Coconut Products

74) The Committee deleted the Bligh-Dyer and AOAC 983.23 methods for total fats because they used chloroform as a solvent.

75) The Committee considered the information provided by the Delegation of Thailand in CRD 10 on the validation studies carried out on ISO 1211:1999 for total fats and ISO 6731:1989 for total solids in coconut milk and agreed to endorse both methods as Type I. The AOAC 963.15 method for total fats was therefore deleted as only one Type I method could be retained. The Committee noted that it would be useful if the validation studies were published in order to provide relevant information for users, as these methods were originally developed for milk products.

Sampling Plans

76) The Committee noted that there was no indication of the purposes of the sampling plans in the Draft Standard for Certain Canned Vegetables and the Draft Standard for Jams and Jellies and asked the CCPFV to clarify to which provisions in the standards the sampling plans applied.

FAO/WHO Coordinating Committee for Asia

Draft Standard for Gochujang

77) The last session of the Committee had endorsed the AOAC method for the determination of capsaicin and temporarily endorsed the method proposed in the Draft Standard for Gochujang as Type IV as it was not fully validated.

78) The Committee considered the validation studies carried out by the Republic of Korea for the determination of capsaicin (CRD 4) and endorsed the method as Type IV, as the inter-laboratory study had not been completed.

79) The last session of the Committee had temporarily endorsed the method for the determination of crude protein since the scope of AOAC 984.13 had not been extended to gochujang and the AOAC 934.01 method for moisture as clarification was needed on the range of temperature for drying. In view of the information provided by the Republic of Korea on the validation of these methods in collaborative studies, they were endorsed as Type I.

Proposed Draft Standard for Fermented Soybean Paste

Proposed Draft Standard for Edible Sago Flour

80) The Committee endorsed the methods as proposed by the CCASIA with some editorial corrections.

Committee on Sugars

81) The Committee recalled that its last session had considered the method for the determination of colour in plantation and mill white sugar and had deferred a decision pending consideration of this question by ICUMSA. The Committee noted the reply from ICUMSA that Method GS2/3-9 had Accepted Status and Method GS9/1/2/3-8 Official Status in the ICUMSA Methods Book, and that ICUMSA recommended that users adopt Method GS9/1/2/3-8 where possible, while accepting that Method GS2/3-9 still has utility and will give similar results over its scope of application.

82) As both methods are empirical and only one method can be retained, the Committee endorsed Method GS9/1/2/3-8 as Type I for plantation and mill white sugars.

83) The Committee also noted that ICUMSA was reviewing its method numbering system and that in the meantime an explanation of the ICUMSA system would be presented in the ICUMSA Method Book.

84) The Committee expressed its appreciation to Dr Wood and to the working group for their excellent work and agreed that the working group would be reconvened prior to the next session. The status of endorsement of methods of analysis and sampling is presented in Appendix IV.

GUIDELINES FOR ESTABLISHING METHODS CRITERIA FOR THE IDENTIFICATION OF RELEVANT ANALYTICAL METHODS (Agenda Item 6)[11]

85) The Committee recalled that its 29th session had agreed to establish an electronic working group led by Sweden with assistance of NMKL to redraft Section II of the *Working Instructions for the Implementation of the Criteria Approach in Codex* that provide guidelines (examples) for establishing method criteria for inclusion in the Procedural Manual for consideration at the present session.

86) The Delegation of Sweden, speaking as Chair of the electronic working group, recalled that since its 27th Session the efforts had made to simplify the text, however it was too difficult to further simplify the document without losing information. The Committee considered the document in Annex II of Document CX/MAS 09/30/7, which prescribed acceptance requirements for establishment of numerical values; criteria for applicability associated with concentration values; levels and minimum applicable ranges; criteria for precision, including examples of assessing methods for compliance.

87) The Committee discussed the definition of recovery as it was proposed to expand the definition to allow covering the whole analytical methods including determination, but it should not be defined only as the yield of extraction step. After some discussion, the Committee agreed to delete the second sentence in Section 1.4 as it was noted that the definition of recovery was discussed under Agenda Item 4. It was also agreed that expression of precision ($PRSD_R$) should be consistent throughout the document. Some other comments were also discussed regarding criteria on use of bias in section 1.5; consistent use of definitions for LOD/LOQ; harmonization for use of symbols. In Section 1.1.1, $RSDR_R$ was replaced with $PRSD_R$ and the term "theoretical" was replaced with "predicted". Consequently, the expressions of precision were amended in Table 1 of the *Working Instructions.*

88) The Observer of NMKL, referring to CRD 18, proposed to add a table and other text in Annex II, therefore the Committee requested Sweden and NMKL to present a revised text for discussion at the present session, bearing in mind changes made and comments raised above. The Committee considered the text in CRD 19 with the revision in either bold or strike-out. Discussion held and decisions made are summarized as follows:

Section 1

89) It was agreed to a proposed footnote below the chapeau paragraph stating that "these criteria are applicable to fully validated methods except for methods such as PCR and ELISA which requires other set of criteria". Consequently, it was also agreed to add the same Note above Table 1 of the *Working Instruction* in the Procedural Manual.

Section 1.1.1

90) A proposal for the deletion of this section was not accepted, while noting that it was not clear how to apply the criteria, in particular for the use of Type II and III methods.

General discussion

91) A question was raised as to whether the draft guidelines should be included in the Procedural Manual or become a stand alone Codex Document, noting that the Committee on General Principles (CCGP) had recommended that the documents intended for use by governments should be published as a part of Codex Alimentarius, the Committee reaffirmed the original intention of this work to develop the document for use by Codex committees, and in particular CCMAS. Several delegations supported the inclusion of the document in the Procedural Manual as they provided guidance to Codex Committees.

Status of the Guidelines for Establishing Methods Criteria for the Identification of Relevant Analytical methods

92) The Committee agreed to forward the Guidelines to the Committee on General Principles for endorsement and to the 32nd Session of the Commission for adoption (see Appendix V).

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[11] CX/MAS 09/30/7, CRD 5 (comments of Kenya), CRD 6 (comments of Brazil), CRD 7 (comments of ISO), CRD 17 (comments of United States of America), CRD 18 (comments of NMKL), CRD 19 (Revised text prepared by Sweden and NMKL)

PROPOSED DRAFT GUIDELINES ON CRITERIA FOR METHODS FOR THE DETECTION AND IDENTIFICATION OF FOODS DERIVED FROM BIOTECHNOLOGY (Agenda Item 7)[12]

93) The Committee recalled that the 31st Session of the Commission approved new work to develop Guidelines on Criteria and had recommended that the Committee consider the concern and recommendations regarding the scope expressed during the session.

Consideration of the expansion of scope

94) The Committee first considered the possible expansion of the scope. Some delegations were of the opinion that the guidelines were valuable and should be applicable not only for genetically modified materials but also to address a wide range of food safety issues such as allergens, contaminants and pathogens and proposed to broaden the scope of the guidelines. Some other delegations opposed the expansion of the scope as this might cause potential delay of work. These delegations noted that there was an urgent need for technical guidance on methodology applied to genetically modified foods and a need to facilitate harmonization at the international level.

95) In order to reach consensus on this matter in the plenary, an in-session physical working group was established, co-chaired by Argentina and United Kingdom, to consider a possible expansion of the scope, bearing in mind that the Committee should not delay progress of the work to develop the guidelines.

96) The Committee discussed a proposed statement and new title of the Guidelines that were prepared by the physical working group as presented in CRD 20.

97) The Delegation of Canada expressed its concern on the absence of Codex standards that required methods of analysis and did not support the inclusion of allergens and microbial pathogens in the scope.

98) The Delegation of Australia was of the view that the inclusion of the examples in the new scope shifted the emphasis of the document more towards health protection issues than the original document.

99) Some amendments were made to delete references to varietal identification, allergen and microbial pathogens. The Committee agreed to the following scope:

> *These guidelines provide information for the validation of methods for the detection, identification, and quantification of specific DNA sequences and specific proteins in foods derived from modern biotechnology. These Guidelines may also provide information on the validation of methods for other specific DNA sequences and proteins of interest in other foods. Information relating to general considerations for the validation of methods for the analysis of specific DNA sequences and specific protein in foods is given in the first part of these Guidelines. Specific annexes are provided that contain information on definitions, validation of quantitative PCR methods, validation of qualitative PCR methods, validation of protein-based methods, and proficiency testing.*

100) The Committee considered two options of the proposed title and agreed to replace the current title with "Proposed draft guidelines on criteria for methods for detection, identification and quantification of specific DNA sequences and specific proteins, in particular in foods derived from modern biotechnology".

101) The Delegation of Australia reserved its position to the decision on the amendments to the title as it did not support any extension of the scope. The Delegation of the United States expressed its reservation regarding the inclusion of the term "modern biotechnology" in the title. The United States preferred the second title option, which included biotechnology and several other applications in a footnote to the title in order to clarify the broadened scope of the document and the applications of these methods criteria.

102) The Committee agreed to inform the Commission of the amendment to the scope and title.

General comments on the guidelines

103) The Committee considered the proposed draft guidelines to share general views on how to improve the text. Comments made are summarized as follows:

104) The Committee agreed to change the structure and outline, taking into account a proposal of Japan in its written comment in CX/09/30/8-Add.1 aimed at making the guidelines easier to understand.

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[12] CX/MAS 09/30/8, CX/MAS 09/30/08-Add.1(comments of Australia, Brazil, Colombia, Japan, Kenya, United States, EUROPABIO, ICGMA, ILSI), CX/MAS 09/30/08-Add.2(comments of Argentina), CRD 11 (comments from CropLife International), CRD 16 (comments of Republic of Korea), CRD 20 (Report of the in-session physical working group)

105) The Committee noted that the guidelines were prepared for use by governments, therefore should not include texts describing Codex procedural matters (e.g. endorsement of CCMAS) and agreed to delete the entire section on "INFORMATION TO BE PROVIDED TO CODEX WHEN A METHOD FOR FOODS DERIVED FROM BIOTECHNOLOGY IS TO BE CONSIDERED FOR ENDORSEMENT BY CCMAS" and replaced the current title of Annex I with "Required information when methods are to be considered for use" and deleted the first paragraph.

106) The Committee noted that the subsection on 'Applicability of the Method' needed significant changes to include protein-based test methods. It was further noted that there was a need for update of detection methods and technical/scientific references, the estimation of measurement uncertainty, elimination of repetition, harmonization of terms and other necessary editorial amendments throughout the documents.

107) The Observer from AOCS, speaking on behalf of the Interagency Meeting, and the Observer from ISO noted that projects were ongoing and had already produced guidelines and validated methods for molecular biomarker analysis.

Status of the Proposed Draft Guidelines on Criteria for Methods for the Detection and Identification of Foods Derived from Biotechnology

108) The Committee agreed to return the text to Step 2 and to establish an electronic working group, co-chaired by Argentina, Germany and the United Kingdom, working in English, open to all members and observer organizations, to revise the proposed draft guidelines, taking into account comments submitted and raised at the present session. The revised text would be circulated for comments at Step 3 and consideration at its next session.

PROPOSED DRAFT REVISED GUIDELINES ON MEASUREMENT UNCERTAINTY (Agenda Item 8)[13]

109) The Committee recalled that its last session had put forward a proposal for new work on the revision of the Guidelines on Measurement Uncertainty (CAC/GL 50-2004) that had been approved as new work by the Commission and that an electronic working group led by the United Kingdom had prepared the Proposed Draft Revised Guidelines.

110) The Delegation of the United Kingdom introduced the paper and indicated that the document was not intended for metrological experts but routine providers of analytical data, customers of laboratories reporting analytical data and Codex delegates. The document attempted to clarify the significance of measurement uncertainty and answer several specific questions in sections 1 to 9. The Delegation recalled that measurement uncertainty has to be estimated under the requirements of ISO 17025:2005, adopted in Codex by reference, and that it was anticipated that this request would be made for the purposes of international trade; that the document did not address sampling uncertainty; and that no specific procedures were recommended for estimating uncertainty, only that the procedure should be scientifically credible. The document highlighted the implications of the recommendations in the Procedural Manual on the *Use of Analytical Results* and in particular that Codex Commodity Committees should recognise the difference between the numeric value in the specification and the numeric value at which the specification would be enforced. The various situations that might occur at the enforcement stage were analysed in Section 9.1 and summarised in the diagram, and dispute situations were addressed under Section 10.

111) The Committee expressed its thanks to the Delegation of the United Kingdom and the working group for the development of this important document addressing complex issues.

112) Some delegations recalled that the Committee on Pesticide Residues (CCPR) was also working on guidelines for measurement uncertainty in the area of pesticides and that duplication should be avoided through adequate cooperation between CCMAS and CCPR. It was also suggested that the CCPR should wait until CCMAS had competed its work in order to ensure consistency of approach.

113) The Secretariat recalled that the documents developed by the Committee from a general perspective were regularly forwarded to the Committee on Pesticide Residues for information, and CCMAS was regularly informed of relevant work in CCPR or other committees, as required in order to ensure consistency of approach, while it was the responsibility of CCPR to develop guidance for specific pesticide

[13] CX/MAS 09/30/9, CX/MAS 09/30/9-Add.1 (comments of Australia, Japan, New Zealand), CRD 13 (comments of Chile), CRD 12 (comments of European Community), CRD 14 (comments of New Zealand)

residues. The revision of the Guidelines on the Estimation of Uncertainty of Results (CAC/GL 54-2006) applicable to pesticides was approved by the Commission and the document was still at an early stage of development in CCPR, and the Committee would be kept informed of its progress under matters referred from other committees.

114) The Delegation of New Zealand expressed the view that the measurement uncertainty should be statistically sound, should take account of the practicalities of compliance assessment and trade in food, should not impose unreasonable costs or unnecessary food rejection, and should recommend sampling plans that provide consumers and producers with known levels of protection. The Delegation also pointed out that section 1 was open to misinterpretation that the range $a \pm 2u$ is a 95% confidence interval for the true value whereas this is a tolerance interval, and indicated that CRD 14 examined the coverage rates achieved by the recommended procedures. This paper presented the background to support the view that the coefficients for cut offs and uncertainty intervals are underestimated and considered a recommended procedure to estimate measurement uncertainty.

115) Some delegations expressed the view that the documents should not be too prescriptive and reflect that decisions should be based on risk; that the consequences for import and exporting countries should be considered carefully, especially to the assessment of compliance.

116) The Committee noted a proposal to redraft the sections referring to the need for accreditation to avoid confusion as the Codex guidelines do not require accreditation status of laboratories but compliance with the international standard on accreditation. It also noted a suggestion that this work should not address dispute situations as it was considered in another document under Agenda Item 3b.

117) The Committee noted the view that measurement uncertainty is not always taken into account in order to determine compliance as in case of serious health hazard, there is always a need to take action even with a low probability of non compliance, for example if there is a risk of exceeding the acute reference dose for pesticides.

118) Some specific drafting suggestions were also made in the discussion, such as the inclusion of a scope section; the use of a different format more appropriate for Codex Guidelines; inserting references for the diagram or similar figures; and reviewing the document to avoid direct or indirect references to sampling uncertainty.

119) The Delegation of the United Kingdom provided some further clarification in response to the comments, highlighting the objective of the document to provide a science based document that could be easily used by regulators and Codex Committees, and also noted that for the purposes of export, laboratories had to accredited, as mentioned in CAC/GL 27-1997 *Guidelines for the Competence of Testing Laboratories Involved in the Import and Export Control of Food* and this requirement should be reflected in the guidelines under consideration as they were intended for use in international trade. The Delegation indicated that the contributions and comments put forward would be taken into account in order to revise the document, but that the original intent should not be lost in the process.

120) The Committee noted that in view of the above discussion, the document could not be discussed in detail at this stage and should be redrafted by an electronic working group led by the Delegation of the United Kingdom, open to all members and observers and working in English.

Status of the Proposed Draft Revised Guidelines on Measurement Uncertainty

121) The Committee agreed to return the Proposed Draft Guidelines to Step 2 for redrafting as mentioned above, circulation for comments at Step 3 and consideration by the next session.

GUIDANCE ON UNCERTAINTY OF SAMPLING (Agenda Item 9)[14]

122) The Committee recalled that uncertainty of sampling had been introduced at previous sessions and that the importance of the issue was recognised but it was agreed to consider it in more detail before undertaking specific new work.

123) The Delegation of the United Kingdom introduced the discussion paper prepared at the request of the last session and stressed the importance of addressing this issue in Codex, taking into account the guidance produced at the international level in the EURACHEM/ EUROLAB/ CITAC/ Nordtest Guide on

[14] CX/MAS 09/30/10, CRD 8 (comments of Australia), CRRD 9 (comments of Brazil), CRD 12 (comments of the European Community), CRD 21 (comments of Argentina)

the Estimation of Measurement Uncertainty Arising from Sampling and the Nordtest Handbook for sampling planners. It was especially critical for the Committee to decide whether sampling uncertainty should be taken into account when assessing compliance, or to follow the non-scientific approach of defining sampling uncertainty as zero. The Delegation suggested to take a similar approach as in the case of measurement uncertainty with the development of guidelines, with the final objective of developing a guide integrating both measurement and sampling uncertainty, and invited the Committee to consider an initial draft of guidelines on measurement uncertainty including sampling uncertainty.

124) Several delegations supported the development of an overarching document on measurement uncertainty combining analytical and sampling uncertainty in order to address uncertainty as a whole on a scientific basis.

125) Other delegations expressed the view that it was premature to undertake new work at this stage on uncertainty of sampling as priority should be given to progress on analytical measurement uncertainty, as discussed under Agenda Item 8, which had been initiated recently and required considerable work to address difficult issues.

126) The Delegation of Argentina noted the difficulties related to measurement and sampling uncertainty estimation when determining compliance with specifications, for example in the area of pesticide and veterinary drug residues, and expressed some concerns as to the implications for MRL setting and enforcement, including export and import control, and pointed out that the measure should be proportional to the risk in the area of food safety.

127) The Delegation of New Zealand expressed the view that measurement and sampling uncertainty could be combined to provide general guidance on uncertainty, but expressed its concerns with several aspects of the proposed outline: the duplicate method based on the EURACHEM and Nordtest Guides; likelihood of underestimation; and the implications as regards enforcement, especially at the import stage. In addition, the Delegation pointed out that the General Guidelines on Sampling were scientifically sound and did not support their amendment at this stage.

128) The Delegation of the Netherlands expressed the view that to address such concerns, sampling and measurement uncertainty could be combined and the risk for the exporter and the importer should be made clear.

129) The Chairperson noted that, although several concerns were expressed as to how to address sampling uncertainty, there was general agreement to consider this issue further in the Committee, taking into account the work already developed in specific areas such as mycotoxins, and the specific issues related to pesticide and veterinary drugs residues control.

130) The Delegation of the United Kingdom noted that the EURACHEM and Nordtest Guides and the provisions in the document based on these references were presented as a basis for discussion, that many issues raised in the comments required further consideration, and therefore proposed to revise the document for further consideration and possible decision on new work at the next session.

131) The Delegation of Brazil suggested that the document on uncertainty on sampling should clarify the relationship between sampling uncertainty and the already approved sampling methods that are mentioned in the General Guidelines on Sampling (CAC/GL-50-2004).

132) The Committee agreed that an electronic working group led by the Delegation of the United Kingdom would revise the current document in the light of the comments received, to develop basic principles applicable to sampling uncertainty in order to allow the next session to discuss this issue further and decide how to proceed.

DISCUSSION PAPER ON SAMPLING FOR MILK AND MILK PRODUCTS (Agenda Item 10)[15]

133) The Committee recalled that its last session had considered the question from the Committee on Milk and Milk Products (CCMMP) concerning conformity assessment in the presence of significant measurement error and had agreed to consider this matter in conjunction with the general approach to uncertainty of sampling and welcomed the offer of the Delegation New Zealand to prepare a discussion paper outlining the problems and indicating how it could be addressed in a horizontal manner.

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15 CX/MAS 09/30/11, CRD 12 (comments of European Community)

134) The Delegation of New Zealand presented the discussion paper in CX/MAS 09/30/11, which incorporated a wider review of the work regarding the sampling plans initiated by the CCMMP that addressed specific issues for milk and milk products only and an approach using statistic criteria to quantify the degree of acceptance or rejection level to ensure fair and valid sampling procedures. The Delegation highlighted that the Codex General Guidelines on Sampling could not be applied to many standards for foods due to the presence of significant measurement error and recommended that the Committee should establish a working group to review and investigate the sampling plans for foods in the presence of significant measurement uncertainty; prepare recommended plans based on valid statistical principles; provide tools for evaluation of risk for consumer and producers, and consider whether it is necessary to specify a maximum producer's risk a maxim chance of rejection and make recommendations about the maximum chance of rejection allowable for conforming products.

135) The Committee noted a general view that there was a need for the development of specific provisions not only for a certain category of foods but for all types of foods. The Committee agreed that the electronic working group established during the discussion on uncertainty of sampling under Item 9 should also cover this matter. The working group would consider the recommendations above, however the Committee noted that this work was not intended to amend the General Guidelines on Sampling at this stage.

DISCUSSION PAPER ON METHODS OF ANALYSIS FOR DIOXINS AND DIOXIN-LIKE PCBs (Agenda Item 11)[16]

136) The Committee recalled that at its last session it had been agreed to prepare a discussion paper aimed at answering the question from the Committee on Contaminants in Foods (CCCF) on the applicability of the methods for the indicated ranges and commodities concerned, review the validation data for the methods, and set criteria for dioxin analysis, for consideration at the present session.

137) The Delegation of Germany, speaking as Chair of the electronic working group, referring to the discussion paper in CX/MAS 09/30/12, highlighted that this document was not to intended to gather a long list of analytical methods for detection of dioxins and dioxin-like PCBs but to provide a criteria approach to the selection of detection methods for monitoring and recommended that the Committee should consider further the procedure for establishing these criteria in Codex and whether this paper should be forwarded to the CCCF.

138) The Committee noted that the list in Annex II did not intend to identify any status and purposes (confirmative or screening purposes), but simply provided information of methods available submitted by government and organizations and agreed to add a footnote in Annex II to state this point. It was also noted that generally high-resolution gas chromatography/high resolution mass spectrometry methods (GC-HRMS) was used as a confirmatory method and GC-MS methods were used for screening.

139) The reference "MS" was added to the title *GCxGC* for clarity and the number of Codex members in the EC was updated. In Annex 4, information from Japan on methods for confirmation for fish, tea, meat, dairy products, milk and egg submitted was deleted as they were repetition and a scientific reference regarding TEQ – value for determination of animal feed was added for the screening method provided by Belgium.

140) The Committee agreed to forward this discussion paper as amended above with some editorial changes, for consideration by the Committee on Contaminants in Foods.

REPORT OF AN INTER-AGENCY MEETING ON METHODS OF ANALYSIS AND SAMPLING (Agenda Item 12)[17]

141) The Secretary of the Inter-Agency Meeting, Dr Richard Cantrill (AOCS), introduced the report of the 20th meeting of international organisations working in the field of methods of analysis and sampling (IAM) held prior to the session. In addition to the matters under consideration by the CCMAS, the IAM considered the activities of the organisations concerned, some of which are highlighted below.

142) A meeting of experts was convened in the framework of the ISO International Workshop Agreement on Sampling to attempt to rationalise existing international standards for sampling grain and

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16 CX/MAS 09/30/12, CRD 12 (comments of European Community)

17 CRD 2 (Report of the 21st Meeting of the International Organisations working in the field of methods of analysis and sampling (Inter Agency Meeting))

oilseeds. As a result a modification of ISO/DIS 24333 with tables representing current dockside and trade practices had been prepared and was in the process of further elaboration.

143) The IAM considered the criteria approach to methods of analysis selection and agreed that it should not replace the need for official methods of analysis, and that methods should be selected according to the criteria specified in Codex provisions to ensure that they are "fit-for purpose". It recognised that the criteria approach focused on methods of analysis for small molecules and might not be applicable to PCR and ELISA.

144) The Committee noted that the IAM/MoniQA, following the success of the workshop held prior to the 29th Session (2008) on measurement uncertainty, had organised another workshop prior to the present session on development of methods of analysis and measurement uncertainty, which was attended by many CCMAS delegates The Secretary of the IAM also invited CCMAS participants to make proposals for a future workshop which might be held in 2010.

145) The Committee was informed of the recent formation of ISO/TC 34/SC 16 "Horizontal Methods for Molecular Biomarker Analysis" with the recently amended scope "Standardisation of biomolecular testing methods applied to: foods; feeds; seeds and other propagules of food and feed crops"

146) The IAM took note of the work of CEN TC 275 WG0 to develop overall guidance to TC 275 working groups in the foods sector on questions such as measurement uncertainty and various aspects of method validation.

147) The IUPAC/MoniQA project was undertaking some modelling exercises concerning the validation of qualitative methods, some results of which were presented to the IAM/MoniQA workshop.

148) The IAM also considered the question of the availability of reagents and the concerns related to the proprietary nature of monoclonal antibodies and whether this restricted the development of rapid methods of detection in some areas

149) The Committee was informed that the work programmes of several IAM Members were available through links to the IAM Secretariat on the IAM website, hosted by AOCS.

150) The Committee expressed its appreciation to the international organisations participating in the meeting of the IAM for their contribution to the work of the Committee and the organisation of the IAM/MoniQA workshop, and to the Hungarian Food Safety Office for hosting the IAM. The next meeting of the IAM would be held prior to the 31st Session of the Committee.

OTHER BUSINESS AND FUTURE WORK (Agenda Item 13)

Updating references for analytical methods

151) The Committee considered a request to regularly update the references in Codex methods of analysis as the methods developed by international organisations were updated and this should be reflected in Codex methods. The Delegation of AOCS, on behalf of IAM, offered that the members of IAM would provide their updated references to the relevant Codex Committees. It was noted that active Codex commodity committees regularly considered the update of the methods in the commodities under their responsibility, but that references might become outdated, when committees were adjourned or standards were not reviewed for a long time.

152) It was proposed to delete the reference to the year in the method in order to solve this problem. The Committee however recalled that the matter had been discussed in the past and that this had not been agreed in view of the need to retain the reference year for regulatory purposes. The Committee recalled that under ISO/IEC 17025: 2005, referred under CAC/GL 27, analysts were required to use the most updated version of methods of analysis and that it had been agreed to insert a note to CODEX STAN 234 to this effect.[18]

153) The Committee concluded that the IAM member organisations could provide information for update as regards methods of analysis and that for the update of other texts such as Guidelines, the need for update and revision needed to be raised by members.

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[18] ALINORM 05/28/23, para. 88

Source of Documents

154) The Committee noted that the sources of tables, pictures and graphs inside some Codex document were missing and agreed that the sources and relevant references should be provided when they were proposed for inclusion in Codex working documents.

Methods for the Analysis of Melamine in Food and Feeds

155) The Delegation of the Nigeria, referring to CRD 15, sought the advice of the Committee and the IAM on methods for the determination of melamine in food and feed, as the recent incident of melamine contamination in food was a challenge to food control authorities and the availability of suitable method was essential to prevent marketing of contaminated food. The Delegation indicated that at the moment screening methods using ELISA were used in Nigeria and that the HPLC methods was to be implemented soon by the control agency.

156) The Committee was informed that an AOAC method was in the process of validation, and that test kits were also to be collaboratively studied.

157) The Observer from IDF informed the Committee that IDF and ISO are working together on a specific project on intentional adulteration of milk and milk products including the detection and determination of melamine with a confirmatory method LC-MS-MS for melamine and cyanuric acid. It was also noted that another, more long term project, concerned the integrity of the raw milk supply chain by screening milk for adulteration.

158) The Delegation of Australia drew the attention of the Committee to the impact of the presence of preformed crystals on the results of melamine analysis. The Delegation of the United Kingdom indicated that the EU JRC laboratory had conducted extensive proficiency testing in this area and the results would be soon available.

Proprietary Methods Issues

159) The Chair of the IAM informed the Committee that several issues regarding proprietary methods had been discussed in the IAM and in some previous sessions of the Committee, such as the availability of reagents, restricted licensing of antibodies and the question of how to describe the method more generically for the purpose of use as a Codex method.

160) The Committee welcomed the offer from IAM to consider this issue among the standard setting organisations and invited delegates to provide their contribution, with a view to preparing a discussion paper on proprietary methods for consideration by the next session.

DATE AND PLACE OF THE NEXT SESSION (Agenda Item 14)

161) The Committee was informed that the 31st Session of the Committee would be held in Hungary in March 2010 and that the exact date and venue would be determined by the host country and the Codex Secretariat.

SUMMARY STATUS OF WORK

Subject Matter	**Step**	**Action by**	**Document Reference in ALINORM 09/32/23**
Draft Guidelines for Settling Disputes on Analytical (Test) Results	8	Governments 32nd CAC	para. 25 Appendix II
Draft Guidelines on Analytical Terminology	8	Governments 32nd CAC	para. 43 Appendix III
Consequential Amendment to the *General Criteria for the Selection of Methods of Analysis* (terminology)	(*)	CCGP Governments 32nd CAC	para, 44 Appendix VI
Endorsement of methods of analysis in Codex Standards		Governments 32nd CAC	paras. 46-82 Appendix IV
Draft Guidelines for Evaluating Acceptable Methods of Analysis	7(**)	32nd CAC	para. 18
Proposed Amendment to the *Working Instructions for the Implementation of the Criteria Approach in Codex*	(*)	CCGP Governments 32nd CAC	para. 92 Appendix V
Proposed Draft Guidelines on Criteria for Methods for the Detection and Identification of Foods Derived from Biotechnology	2/3	Governments 31st CCMAS	para. 108
Proposed Draft Revision of the *Guidelines on Measurement Uncertainty*	2/3	Governments 31st CCMAS	para. 121
Guidance on Uncertainty from Sampling		United Kingdom/ Governments 31st CCMAS	para. 132
Consideration of Methods of Analysis for Dioxins and Dioxin-like PCBs		CCCF	para. 140
Methods of Analysis for Natural Mineral Waters		Governments 31st CCMAS	para. 8

(*) Procedural Manual
(**) Discontinuation of work

ALINORM 09/32/23
APPENDIX I

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DRAFT GUIDELINES FOR SETTLING DISPUTES OVER ANALYTICAL (TEST) RESULTS

(At Step 8 of the Procedure)

1. SCOPE

These guidelines provide guidance to governments on the procedures to resolve disputes which arise between food control authorities about the status of a food consignment[1], when the assessment based on test results made in the importing country disagrees with the assessment made by the exporting country on the same lot[2].

These guidelines only address disputes related to methods of analysis or laboratory performance and do not address questions of sampling. The procedure examines only the validity of the importing country's results on which non-compliance is alleged. It is recognised that disputes may arise from other cause(s), which should also be investigated[3].

These guidelines do not cover microbiological test results.

2. PREREQUISITES/ASSUMPTIONS

The procedure described in these guidelines is operable and effective only when the conditions listed below are met. Competent authorities should therefore ensure that these are satisfied wherever possible. These conditions are:

- both countries agree on using this guideline;
- laboratories[4] comply with quality assurance provisions and with the Codex *Guidelines for the Assessment of the Competence of Testing Laboratories Involved in the Import and the Export of Food (CAC-GL 27)*, and the laboratories have been designated by their respective Competent Authorities in both the importing and exporting countries;
- at least one representative sample[5] from the same food lot has been taken by the Competent Authority at import in accordance with established sampling plans and/or good sampling practices, where applicable; this sample has been split into three essentially identical parts for the purposes of primary analysis and for confirmatory analysis (reserve samples)[6]; the split reserve samples should be kept in a satisfactory condition for the appropriate length of time;

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[1] Status of the food consignment depends on the "interpretation" of the test result(s), in the light of measurement uncertainty, sampling error and the closeness of those test results to the limit. It could still be that the results do not differ by an amount which is significant, but nevertheless one result indicates conformity, but the other result does not.

[2] As defined in the General Guidelines for Sampling (CAC/GL 50 -2004).

[3] Possible reasons for disagreement may include one or several causes such as: the existence, appropriateness and statistical validity of the sampling plan used to assess the product; the allowances made for normal measurement error and within-lot product variation; differences in physical sampling procedures; differences in composition of the samples tested due to product in homogeneity or changes occurring during storage and/or transport of the product.

[4] For the purpose of these guidelines, the word "laboratory" applies to both official and officially recognised laboratories. An official laboratory would be a laboratory administered by a government agency having jurisdiction empowered to perform a regulatory or enforcement function or both. An officially recognised laboratory would be a laboratory that has been formally approved, designated or recognised by a government agency having jurisdiction.

[5] This may be a set of samples. When the wording "sample" is used it might refer to a set of samples

[6] However, if the applicable sample has been split into two essentially identical parts, then the procedure as outlined might be followed, with the omission of the step described in section 4.

- laboratories report quantitative analytical results in the form of "a ± 2u" or "a ± U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "u" is the standard uncertainty and "U" (equal to 2u) is the expanded uncertainty. The range "a ± 2u" represents a 95% level of confidence where the true value would be found. The value of "U" or "2u" is the value which is normally used and reported by analysts and is referred to as the "measurement uncertainty"; it may be estimated in a number of different ways (*see Codex Guidelines on Measurement Uncertainty, CAC/GL 54-2004*);
- laboratories/competent authorities report the sampling plan (including acceptance criteria) that was used and the analytical results that were used to determine the acceptance status, including any information necessary to interpret the results such as:
    a) whether analytical results are expressed on a recovery-corrected basis (and if so the method by which recovery was taken into account and the recovery rate),
    b) the units in which results are expressed, and
    c) the number of significant figures.
- laboratories use specific methods of analysis, which have been endorsed by the Codex Alimentarius Commission (CAC) or use methods of analysis which comply with performance parameters which have been endorsed by the CAC when they are available. Otherwise, methods must have been validated according to the requirements of the CAC.

3. THE RESULTS AND PROCEDURES OF THE LABORATORY OF THE EXPORTING COUNTRY AND ITS COUNTERPART IN THE IMPORTING COUNTRY ARE COMPARED[7]

The competent authorities have the option to agree on comparison of the background information of the analysis of the sample. In accordance with relevant Codex Guidelines[8], the following information should be shared between competent authorities of the importing and exporting country to allow comparison of the results and procedures of the laboratory of the exporting country and its counterpart in the importing country. The relevant information covers:

- validation status of the methods of analysis used (including method-specific sample handling and preparation procedures within the laboratory);
- raw data (including spectral data, calculations, chemical standards used);
- results of repeat analysis;
- internal quality assurance/control (control charts, sequence of analysis, blank data, recovery data, uncertainty data, use of appropriate reference standards and materials);
- performance in relevant proficiency testing or collaborative studies;
- official accreditation status of the laboratories.

Each competent authority reviews its initial assessment on the basis of the additional information received from the other. This may lead to agreement on conformity or agreement on non-conformity, e.g. by recognising the validity of the results of only one of the two laboratories. In this way, the dispute is resolved without further analysis.

If the dispute still exists the competent authorities continue with the step in section 4.

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[7] In cases where a dispute needs to be resolved quickly, for instance where perishable food is in question or where demurrage costs are high, it is recommended that the competent authorities should consider performing the steps outlined in sections 3 and 4 in parallel.

[8] See ANNEX to GUIDELINES FOR THE EXCHANGE OF INFORMATION BETWEEN COUNTRIES ON REJECTIONS OF IMPORTED FOOD (CAC/GL 25-1997): "Where imported food has been rejected on the basis of sampling and/or analysis in the importing country, details should be made available on request as to sampling and analytical methods and test results and the identity of the testing laboratory."

4. ANALYSING RESERVE SAMPLE

A reserve sample is analysed, subject to it being established that sample integrity and the chain of custody have not been compromised and subject to agreement between the respective competent authorities on the following procedures for analysis of the sample(s):

1. the timeline, and the time of availability of the sample[9];
2. the analysis of the reserve sample by either

   the importing country's laboratory in the presence of an expert from the exporting country

   or

   a laboratory chosen by the exporting country;
3. the methods of analysis to be used by the laboratory.

If the original test result of the importing country and the result of the reserve sample differ by less than the critical difference Δ that would be expected from measurement uncertainty of the results (see Annex), the importing country's original assessment of the lot shall stand, and the dispute is thus resolved.

If the dispute still exists, the measures outlined in section 5 of this procedure, using arbitration by a third laboratory, should be applied.

5. ANALYSIS OF REMAINING RESERVE SAMPLE

The remaining reserve sample should be analysed by a suitably qualified laboratory agreed on by the two countries, and a final assessment of conformity is based on the results from this laboratory. Failing agreement on the choice of laboratory the competent authority of the importing country can select a laboratory. The original result and the result from the reserve sample tested in the step outlined in section 4 are discarded. If possible this laboratory should be independent of the laboratory or laboratories whose results were compared in the step in section 4.

[9] The dispute shall be resolved within the shortest possible time, which should not adversely affect the quality of the commodity during storage, where appropriate.

ANNEX

The critical difference Δ between the two results to be compared is

$$\Delta = \sqrt{U_1^2 + U_2^2}$$

Where U_1 and U_2 are the expanded measurement uncertainties of the two results.

In case a set of samples is involved, a different formulation for the critical difference should be used.

DRAFT GUIDELINES ON ANALYTICAL TERMINOLOGY

(At Step 8 of the Procedure)

INTRODUCTION

The Codex Committee on Methods of Analysis and Sampling has agreed on Analytical Terminology for Codex Alimentarius and government use. A number of these terms were previously included in the Codex Procedural Manual. In most cases terms used in the Procedural Manual were adopted over time with an underlying hierarchy and can be traced verbatim to specific editions of ISO 3534, the GUM, the VIM, the IUPAC Orange Book or other international standards already adopted by Codex. Definitions of terms that have changed with newer editions of the international standards from which they were originally adopted have been updated preserving the original hierarchy found in the Procedural Manual. In cases where terms have been added in addition to those originally found in the procedural manual an effort has been made to preserve the conceptual continuity and relationship of the newer terms with extant ones. These terms, together with the terms which are included in specific International Protocols/Guidelines already adopted by Codex by reference are given below.

ANALYTICAL TERMS

The following analytical terms are defined below:

Accuracy

Analyte

Applicability

Bias

Calibration

Certified reference material

Conventional quantity value

Critical value

Defining (Empirical) method of analysis

Error

Expanded measurement uncertainty

Fitness for purpose

HorRat

Inter-laboratory study

Laboratory performance (Proficiency) study

Limit of detection

Limit of quantification

Linearity

Material certification study

Measurand

Measurement method

Measurement procedure

Measurement uncertainty

Method-performance study

Metrological Traceability

Outlier

Precision

Quality assurance

Rational method of analysis

Recovery/recovery factors

Reference material

Reference value

Repeatability (Reproducibility)

Repeatability conditions

Repeatability (Reproducibility) limit

Repeatability (Reproducibility) standard deviation

Repeatability (Reproducibility relative standard deviation

Reproducibility conditions

Result

Robustness (ruggedness)

Selectivity

Sensitivity

Surrogate

Systematic error

Trueness

True value

Validated range

Validated Test Method

Validation

Verification

DEFINITIONS OF ANALYTICAL TERMS

Accuracy: The closeness of agreement between a test result or measurement result and a reference value.

Notes:

The term "accuracy," when applied to a set of test results or measurement results, involves a combination of random components and a common systematic error or bias component.

When applied to a test method, the term accuracy refers to a combination of trueness and precision.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Analyte: The chemical substance sought or determined in a sample.

Note:

This definition does not apply to molecular biological analytical methods.

Reference:

Codex Guidelines on Good Laboratory Practice in Residue Analysis (CAC/GL 40-1993)

Applicability: the analytes, matrices, and concentrations for which a method of analysis may be used satisfactorily.

Note:

In addition to a statement of the range of capability of satisfactory performance for each factor, the statement of applicability (scope) may also include warnings as to known interference by other analytes, or inapplicability to certain matrices and situations.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Bias: The difference between the expectation of the test result or measurement result and the true value. In practice conventional quantity value (VIM, 2007) can be substituted for true value.

Notes:

Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value.

The bias of a measuring instrument is normally estimated by averaging the error of indication over the appropriate number of repeated measurements. The error of indication is the: "indication of a measuring instrument minus a true value of the corresponding input quantity".

Expectation is the expected value of a random variable, e.g. assigned value or long term average {ISO 5725-1}

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Calibration: Operation that, under specified conditions, in a first step, establishes a relation between the values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and in a second step uses this information to establish a relation for obtaining a measurement result from an indication.

Notes:

A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Calibration should not be confused with adjustment of a measuring system often mistakenly called "self calibration," or with verification of calibration.

Often the first step alone in the above definition is perceived as being calibration.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Certified reference material (CRM): Reference material accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceability, using valid procedures

Notes:

Documentation is given in the form of a "certificate" (see ISO guide 30:1992).

Procedures for the production and certification of certified reference materials are given, e.g. in ISO Guide 34 and ISO Guide 35.

In this definition, "uncertainty" covers both measurement uncertainty and uncertainty associated with the value of the nominal property, such as for identity and sequence. Traceability covers both metrological traceability of a value and traceability of a nominal property value.

Specified values of certified reference materials require metrological traceability with associated measurement uncertainty {Accred. Qual. Assur., 2006}

ISO/REMCO has an analogous definition {Accred. Qual. Assur., 2006} but uses the modifiers metrological and metrologically to refer to both quantity and nominal properties.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

New definitions on reference materials, Accreditation and Quality Assurance, 10:576-578, 2006

Conventional quantity value: quantity value attributed by agreement to a quantity for a given purpose.

Notes:

The term "conventional true quantity value" is sometimes used for this concept, but its use is discouraged.

Sometimes a conventional quantity value is an estimate of a true quantity value.

A conventional quantity value is generally accepted as being associated with a suitably small measurement uncertainty, which might be zero.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Critical value (L_C): The value of the net concentration or amount the exceeding of which leads, for a given error probability α, to the decision that the concentration or amount of the analyte in the analyzed material is larger than that in the blank material. It is defined as:

$\Pr(\hat{L} > L_C \mid L=0) \leq \alpha$

Where $\hat{L}$ is the estimated value, L is the expectation or true value and L_C is the critical value.

Notes:

The definition of critical value is important for defining the Limit of Detection (LOD).

The critical value L_c is estimated by

$L_C = t_{1-\alpha\nu} s_{o,}$

Where $t_{1-\alpha\nu}$ is Student's-t, based on ν degrees of freedom for a one-sided confidence interval of $1-\alpha$ and s_o is the sample standard deviation.

If L is normally distributed with known variance, i.e. $\nu = \infty$ with the default α of 0.05, $L_C = 1.645 s_o$.

A result falling below the L_C triggering the decision "not detected" should not be construed as demonstrating analyte absence. Reporting such a result as "zero" or as < LOD is not recommended. The estimated value and its uncertainty should always be reported.

References:

ISO Standard 11843: Capability of Detection-1, ISO, Geneva, 1997

Nomenclature in evaluation of analytical methods, IUPAC, 1995

Defining (empirical/conventional) method of analysis: A method in which the quantity measured is defined by the result found on following the stated procedure.

Notes:

Empirical methods are used for purposes that cannot be covered by rational methods.

Bias in empirical methods is conventionally zero.

Reference:

Harmonised guidelines for single-laboratory validation of methods of analysis, 2002

Error: Measured quantity value minus a reference quantity value.

Note:

The concept of measurement 'error' can be used both: when there is a single reference value to refer to, which occurs if a calibration is made by means of a measurement standard with a measured value having a negligible measurement uncertainty or if a conventional value is given, in which case the measurement error is not known and if a measurand is supposed to be represented by a unique true value or a set of true values of negligible range, in which case the measurement error is not known.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Expanded measurement uncertainty: product of a combined standard measurement uncertainty and a factor larger than the number one

Notes:

The factor depends upon the type of probability distribution of the output quantity in a measurement model and on the selected coverage probability.

The term factor in this definition refers to a coverage factor.

Expanded measurement uncertainty is also termed expanded uncertainty.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Fitness for purpose: Degree to which data produced by a measurement process enables a user to make technically and administratively correct decisions for a stated purpose.

Reference:

Eurachem Guide: The fitness for purpose of analytical methods: A laboratory guide to method validation and related topics, 1998

HorRat: The ratio of the reproducibility relative standard deviation to that calculated from the Horwitz equation,

Predicted relative standard deviation $(PRSD)_R = 2C^{-0.15}$:

$HorRat(R) = RSD_R/PRSD_R$,

$HorRat(r) = RSD_r/PRSD_R$

Where C is concentration expressed as a mass fraction (both numerator and denominator expressed in the same units).

Notes:

The HorRat is indicative of method performance for a large majority of methods in chemistry.

Normal values lie between 0.5 and 2. (To check proper calculation of $PRSD_R$, a C of 10^{-6} should give a $PRSD_R$ of 16 %.)

If applied to within-laboratory studies, the normal range of HorRat(r) is 0.3-1.3.

For concentrations less than 0.12 mg/kg the predicted relative standard deviation developed by Thompson (The Analyst, 2000), 22% should be used.

References:

A simple method for evaluating data from an inter-laboratory study, J AOAC, 81(6):1257-1265, 1998

Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing, The Analyst, 125:385-386, 2000

Inter-laboratory study: A study in which several laboratories measure a quantity in one or more "identical" portions of homogeneous, stable materials under documented conditions, the results of which are compiled into a single document.

Notes:

The larger the number of participating laboratories, the greater the confidence that can be placed in the resulting estimates of the statistical parameters. The IUPAC-1987 protocol (Pure & Appl. Chem., 66, 1903-1911(1994)) requires a minimum of eight laboratories for method-performance studies.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Laboratory-performance (proficiency) study: An inter-laboratory study that consists of one or more measurements by a group of laboratories on one or more homogeneous, stable, test samples by the method selected or used by each laboratory. The reported results are compared with those from other laboratories or with the known or assigned reference value, usually with the objective of improving laboratory performance.

Notes:

Laboratory-performance studies can be used to support laboratory accreditation of laboratories or to audit performance. If a study is conducted by an organization with some type of management control over the participating laboratories: organizational, accreditation, regulatory or contractual, the method may be specified or the selection may be limited to a list of approved or equivalent methods. In such situations, a single test sample is insufficient to judge performance.

A laboratory-performance study may be used to select a method of analysis that will be used in a method-performance study. If all laboratories, or a sufficiently large subgroup, of laboratories, use the same method, the study may also be interpreted as a method-performance study, provided that the test samples cover the range of concentration of the analyte.

Laboratories of a single organization with independent facilities, instruments, and calibration materials, are treated as different laboratories.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Limit of Detection (LOD): The true net concentration or amount of the analyte in the material to be analyzed which will lead, with probability (1-β), to the conclusion that the concentration or amount of the analyte in the analyzed material is larger than that in the blank material. It is defined as:

$Pr(\hat{L} \leq L_C \mid L=LOD) = \beta$

Where $\hat{L}$ is the estimated value, L is the expectation or true value and L_C is the critical value.

Notes:

The limit of detection LOD is estimated by,

$LOD \approx 2t_{1-\alpha\nu}\sigma_o$ [where $\alpha = \beta$],

Where $t_{1-\alpha\nu}$ is Student's-t, based on ν degrees of freedom for a one-sided confidence interval of 1-α and σ_o is the standard deviation of the true value (expectation).

$LOD = 3.29\ \sigma_o$, when the uncertainty in the mean (expected) value of the blank is negligible, $\alpha = \beta = 0.05$ and L is normally distributed with known constant variance. However, LOD is not defined simply as a fixed coefficient (e.g. 3, 6, etc.) times the standard deviation of a pure solution background. To do so can be extremely misleading. The correct estimation of LOD must take into account degrees of freedom, α and β, and the distribution of L as influenced by factors such as analyte concentration, matrix effects and interference.

This definition provides a basis for taking into account exceptions to simple case that is described, i.e. involving non-normal distributions and heteroscedasticity (e.g. "counting" (Poisson) processes as those used for real time PCR).

It is essential to specify the measurement process under consideration, since distributions, σ's and blanks can be dramatically different for different measurement processes.

At the limit of detection, a positive identification can be achieved with reasonable and/or previously determined confidence in a defined matrix using a specific analytical method.

References:

ISO Standard 11843: Capability of Detection-1, ISO, Geneva, 1997

Nomenclature in evaluation of analytical methods, IUPAC, 1995

Guidance document on pesticide residue analytical methods, Organization for Economic Cooperation and Development, 2007

Limit of Quantification (LOQ): A method performance characteristic generally expressed in terms of the signal or measurement (true) value that will produce estimates having a specified relative standard deviation (RSD), commonly 10% (or 6%). LOQ is estimated by:

$LOQ = k_Q\ \sigma_Q,\ k_Q = 1/RSD_Q$

Where LOQ is the limit of quantification, σ_Q is the standard deviation at that point and k_Q is the multiplier whose reciprocal equals the selected RSD. (The approximate RSD of an estimated σ, based on ν-degrees of freedom is $1/\sqrt{2\nu}$.)

Notes:

If σ is known and constant, then $\sigma_Q = \sigma_o$, since the standard deviation of the estimated quantity is independent of concentration. Substituting 10% in for k_Q gives:

$LOQ = (10 * \sigma_Q) = 10\ \sigma_o$

In this case, the LOQ is just 3.04 times the limit of detection, given normality and $\alpha = \beta = 0.05$

At the LOQ, a positive identification can be achieved with reasonable and/or previously determined confidence in a defined matrix using a specific analytical method.

This definition provides a basis for taking into account exceptions to the simple case that is described, i.e. involving non-normal distributions and heteroscedasticity (e.g. "counting" (Poisson) processes as those used for real time PCR).

References:

Nomenclature in evaluation of analytical methods, IUPAC, 1995

Guidance document on pesticide residue analytical methods, Organization for Economic Co-operation and Development, 2007

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 an acceptable uncertainty.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Material-Certification Study: An inter-laboratory study that assigns a reference value ("true value") to a quantity (concentration or property) in the test material, usually with a stated uncertainty.

Note:

A material-certification study often utilizes selected reference laboratories to analyse a candidate reference material by a method(s) judged most likely to provide the least-biased estimates of concentration (or of a characteristic property) and the smallest associated uncertainty.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Measurand: Quantity intended to be measured.

Notes:

The specification of a measurand requires knowledge of the kind of quantity, description of the state of the substance carrying the quantity, including any relevant component and the chemical entities involved.

In chemistry, 'analyte' or the name of a substance or compound are terms sometime used for measurand. This usage is erroneous because these terms do not refer to quantities.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Measurement method: Generic description of a logical organization of operations used in a measurement.

Note:

Measurement methods may be qualified in various ways such as: substitution measurement method, differential measurement method, and null measurement method; or direct measurement method, and indirect measurement method.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Measurement procedure: Detailed description of a measurement according to one or more measurement principles and to a given measurement method, based on a measurement model and including any calculation to obtain a result.

Notes:

A measurement procedure is usually documented in sufficient detail to enable an operator to perform a measurement.

A measurement procedure can include a statement concerning a target measurement uncertainty.

A measurement procedure is sometimes called a standard operating procedure (SOP).

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Measurement uncertainty: Non-negative parameter characterizing the dispersion of the values being

attributed to a measurand, based on the information used.

Notes:

Measurement uncertainty includes components arising from systematic effects, such as components associated with corrections and the assigned values of measurement standards, as well as the definitional uncertainty. Sometimes estimated systematic effects are not corrected for but, instead associated measurement uncertainty components are incorporated.

The parameter may be, for example, a standard deviation called standard measurement uncertainty (or a given multiple of it), or the half-width of interval having a stated coverage probability.

Measurement uncertainty comprises, in general many components. Some of these components may be evaluated by Type A evaluation of measurement uncertainty from the statistical distribution of the values from a series of measurements and can be characterized by experimental standard deviations. The other components which may be evaluated by Type B evaluation of measurement uncertainty can also be characterized by standard deviations, evaluated from assumed probability distributions based on experience or other information.

In general, for a given set of information, it is understood that the measurement uncertainty is associated with a stated quality value attributed to the measurand. A modification of this value results in a modification of the associated uncertainty.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Method-Performance Study: An inter-laboratory study in which all laboratories follow the same written protocol and use the same test method to measure a quantity in sets of identical test samples. The reported results are used to estimate the performance characteristics of the method. Usually these characteristics are within-laboratory and among-laboratories precision, and when necessary and possible, other pertinent characteristics such as systematic error, recovery, internal quality control parameters, sensitivity, limit of quantification, and applicability.

Notes:

The materials used in such a study of analytical quantities are usually representative of materials to be analyzed in actual practice with respect to matrices, amount of test component (concentration), and interfering components and effects. Usually the analyst is not aware of the actual composition of the test samples but is aware of the matrix.

The number of laboratories, number of test samples, number of determinations, and other details of the study are specified in the study protocol. Part of the study protocol is the procedure which provides the written directions for performing the analysis.

The main distinguishing feature of this type of study is the necessity to follow the same written protocol and test method exactly.

Several methods may be compared using the same test materials. If all laboratories use the same set of directions for each method and if the statistical analysis is conducted separately for each method, the study is a set of method-performance studies. Such a study may also be designated as a method-comparison study.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Metrological Traceability: Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the stated measurement uncertainty.

Notes:

A reference can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard.

Metrological traceability requires an established calibration hierarchy.

Specification of the reference must include the time at which this reference was used in establishing the calibration hierarchy, along with any other relevant metrological information about the reference, such as when the first calibration in the calibration hierarchy was performed.

For measurements with more than one input quantity each of the input values should itself be traceable and the calibration hierarchy involved may form a branched structure or network. The effort involved in establishing the metrological traceability for each input value should be commensurate with its relative contribution to the measurement result.

Metrological traceability of a measurement result does not ensure that the measurement uncertainty is adequate for a given purpose or that there is an absence of mistakes.

A comparison between two measurement standards may be viewed as a calibration if the comparison is used to check and if necessary correct the value and measurement uncertainty of the measurement standards.

The ILAC considers the elements for confirming metrological to be an unbroken metrological traceability chain to an international measurement standard or a national measurement standard, a documented procedure, accredited technical competence, metrological to the SI and calibration intervals (see ILAC P-10:2002)

The abbreviated term ‘traceability’ is sometimes used to mean ‘metrological traceability’ as well as other concepts, such as sample traceability or document traceability or instrument traceability or material traceability, where history (trace) is meant. Therefore the full term of metrological traceability is preferred if there is any risk of confusion.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Harmonized guidelines for internal quality control in analytical chemistry laboratories, 1995

ILAC P-10, 2002

Outlier: A member of a set of values which is inconsistent with other members of that set

Note:

The following practice is recommended for dealing with outliers.

a) Tests such as Cochran’s or Grubb’s tests are applied to identify stragglers or outliers:
   - if the test statistic is less than or equal to its 5 % critical value, the item tested is accepted as correct;
   - if the test statistic is greater than its 5 % critical value and less than or equal to its 1 % critical value, the item tested is called a straggler and is indicated by a single asterisk;
   - if the test statistic is greater than its 1 % critical value, the item is called a statistical outlier and is indicated by a double asterisk.

b) It is next investigated whether the stragglers and/or statistical outliers can be explained by some technical error, for example:
   - a slip in performing the measurement,
   - an error in computation,
   - a simple clerical error in transcribing a test result,
   - analysis of the wrong sample.

   Where the error was one of the computation or transcription type, the suspect result should be replaced by the correct value; where the error was from analyzing a wrong sample, the result should be placed in its correct cell. After such correction has been made, the examination for stragglers or outliers should be repeated. If the explanation of the technical error is such that it proves impossible to replace the suspect test result, then it should be discarded as a “genuine” outlier that does not belong to the experiment proper.

c) When any stragglers and/or statistical outliers remain that have not been explained or rejected as belonging to an outlying laboratory, the stragglers are retained as correct items and the statistical outliers are discarded unless the statistician for good reason decides to retain them.

References:
ISO Standard 5725-1: Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions, ISO, Geneva, 1994

ISO Standard 5725-2: Accuracy (trueness and precision) of measurement methods and results Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method, ISO, Geneva, 1994

Precision: The closeness of agreement between independent test/measurement results obtained under stipulated conditions.

Notes:

Precision depends only on the distribution of random errors and does not relate to the true value or to the specified value.

The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is reflected by a larger standard deviation.

Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

Intermediate conditions between these two extreme conditions are also conceivable, when one or more factors within a laboratory (intra-laboratory e.g. the operator, the equipment used, the calibration of the equipment used, the environment, the batch of reagent and the elapsed time between measurements) are allowed to vary and are useful in specified circumstances.

Precision is normally expressed in terms of standard deviation.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

ISO Standard 5725-3: Accuracy (trueness and precision) of measurement methods and results Part 3: Intermediate measures of the precision of a standard measurement method, ISO, Geneva, 1994

Quality assurance: All those planned and systematic actions necessary to provide adequate confidence that analytical results will satisfy given requirements for quality.

Reference:

Harmonized guidelines for internal quality control in analytical chemistry laboratories, 1995

***Rational method of analysis*:** A method that determines an identifiable chemical(s) or analytes(s) for which there may be several equivalent methods of analysis available.

Reference:

Harmonized guidelines for the use of recovery information in analytical measurement, 1998

ISO/IEC Guide 17025:2005: General requirements for the competence of calibration and testing laboratories, ISO, Geneva, 2005

Recovery/recovery factors: Proportion of the amount of analyte, present in, added to or present in and added to the analytical portion of the test material, which is presented for measurement.

Notes:

Recovery is assessed by the ratio $\boldsymbol{R = C_{obs} / C_{ref}}$ of the observed concentration or amount $\boldsymbol{C_{obs}}$ obtained by the application of an analytical procedure to a material containing analyte at a reference level $\boldsymbol{C_{ref}}$.

C_{ref} will be: (a) a reference material certified value, (b) measured by an alternative definitive method, (c) defined by a spike addition or (d) marginal recovery.

Recovery is primarily intended for use in methods that rely on transferring the analyte from a complex matrix into a simpler solution, during which loss of analyte can be anticipated.

Reference:

Harmonized guidelines for the use of recovery information in analytical measurement, 1998

Use of the terms "recovery" and "apparent recovery" in analytical procedures, 2002

Reference material: Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process or in examination of nominal properties.

Notes:

Examination of a nominal property provides a nominal property value and associated uncertainty. This uncertainty is not a measurement uncertainty.

Reference materials with or without assigned values can be used for measurement precision control whereas only reference materials with assigned values can be used for calibration and measurement trueness control.

Some reference materials have assigned values that are metrologically traceable to a measurement unit outside a system of units. In a given measurement, a given reference material can only be used for either calibration or quality assurance.

The specification of a reference material should include its material traceability, indicating its origin and processing. {Accred. Qual. Assur., 2006}

ISO/REMCO has an analogous definition that uses the term measurement process to mean examination which covers both measurement of a quantity and examination of a nominal property.

References:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

New definitions on reference materials, Accred. Qual. Assur., 10:576-578, 2006

Reference value: Quantity value used as a basis of comparison with values of quantity of the same kind.

Notes:

A reference quantity value can be a true quantity value of a measurand, in which case it is unknown, or a conventional quantity value in which case it is known.

A reference quantity value with an associated measurement uncertainty is usually provided with reference to

a) a material, e.g. a certified reference material
b) a reference measurement procedure
c) a comparison of measurement standards.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Repeatability (Reproducibility): Precision under repeatability (reproducibility) conditions.

Reference:

ISO 3534-1 Statistics, vocabulary and symbols-Part 1: Probability and general statistical terms, ISO, 1993

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis, 1999)

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

AOAC International methods committee guidelines for validation of qualitative and quantitative food microbiological official methods of analysis, 2002.

Repeatability conditions: Observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in the same test or measuring facility by the same operator using the same equipment within short intervals of time.

Note:

Repeatability conditions include: the same measurement procedure or test procedure; the same operator; the same measuring or test equipment used under the same conditions; the same location and repetition over a short period of time.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Repeatability (Reproducibility) limit: The value less than or equal to which the absolute difference between final values, each of them representing a series of test results or measurement results obtained under repeatability (reproducibility) conditions may be expected to be with a probability of 95%.

Notes:

The symbol used is r [R]. {ISO 3534-2}

When examining two single test results obtained under repeatability (reproducibility) conditions, the comparison should be made with the repeatability (reproducibility) limit, r [R] = 2.8σr[R]. {ISO 5725-6, 4.1.4}

When groups of measurements are used as the basis for the calculation of the repeatability (reproducibility) limits (now called the critical difference), more complicated formulae are required that are given in ISO 5725-6: 1994, 4.2.1 and 4.2.2.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

ISO 5725-6 "Accuracy (trueness and precision) of a measurement methods and results—Part 6: Use in practice of accuracy value", ISO, 1994

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

***Repeatability (reproducibility) standard deviation*:** Standard deviation of test results or measurement results obtained under repeatability (reproducibility) conditions.

Notes:

It is a measure of the dispersion of the distribution of the test or measurement results under repeatability (reproducibility) conditions.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Repeatability (reproducibility) relative standard deviation (coefficient of variation): Repeatability (reproducibility) standard deviation divided by the mean.

$RSD_{r[R]}$ is computed by dividing the repeatability (reproducibility) standard deviation by the mean.

Notes:

Relative standard deviation (RSD) is a useful measure of precision in quantitative studies.

This is done so that one can compare variability of sets with different means. RSD values are independent of the amount of analyte over a reasonable range and facilitate comparison of variabilities at different concentrations.

The result of a collaborative test may be summarized by giving the RSD for repeatability (RSDr) and RSD for reproducibility (RSD_R).

The RSD is also known as coefficient variation.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 1: General statistical terms used in probability, ISO, Geneva, 2006

AOAC International methods committee guidelines for validation of qualitative and quantitative food microbiological official methods of analysis, 2002.

***Reproducibility conditions*:** Observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in different test or measurement facilities with different operators using different equipment.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Result: Set of values being attributed to a measurand together with any other available relevant information

Notes:

A result of measurement generally contains 'relevant information' about the set of values, such that some may be more representative of the measurand than others. This may be expressed in the form of a probability density function.

A result of measurement is generally expressed as a single measured value and a measurement uncertainty. If the measurement uncertainty is considered to be negligible for some purpose, the measurement result may be expressed as a single measured value. In many fields, this is the common way of expressing a measurement result.

In the traditional literature and in the previous edition of the VIM, result was defined as a value attributed to a measurand and explained to mean an indication or an uncorrected result or a corrected result according to the context.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

***Robustness (ruggedness*):** A measure of the capacity of an analytical procedure to remain unaffected by small but deliberate variations in method parameters and provides an indication of its reliability during normal usage

Reference:

ICH Topic Q2 Validation of Analytical Methods, the European Agency for the Evaluation of Medicinal Products: ICH Topic Q 2 A - Definitions and Terminology (CPMP/ICH/381/95), 1995

Harmonized guidelines for single laboratory validation of methods of analysis, Pure and Appl. Chem., 2002

Selectivity: Selectivity is the extent to which a method can determine particular analyte(s) in a mixture(s) or matrice(s) without interferences from other components of similar behaviour.

Note:

Selectivity is the recommended term in analytical chemistry to express the extent to which a particular method can determine analyte(s) in the presence 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.

Reference:

Selectivity in analytical chemistry, IUPAC, Pure Appl Chem, 2001

Codex Alimentarius Commission, Alinorm 04/27/23, 2004

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Sensitivity: Quotient of the change in the indication of a measuring system and the corresponding change in the value of the quantity being measured.

Notes:

The sensitivity can depend on the value of the quantity being measured

The change considered in the value of the quantity being measured must be large compared with the resolution of the measurement system.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Surrogate: Pure compound or element added to the test material, the chemical and physical behaviour of which is taken to be representative of the native analyte.

Reference:

Harmonized guidelines for the use of recovery information in analytical measurement, 1998

Systematic error: Component of measurement error that in replicate measurements remains constant or varies in a predictable manner.

Notes:

A reference value for a systematic error is a true quantity value, or a measured value of a measurement standard of negligible measurement uncertainty, or a conventional value.

Sytematic error and its causes can be known or unknown. A correction can be applied to compensate for a known systematic error.

Systematic error equals measurement error minus random measurement error.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Trueness: The closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value.

Note 1: Measurement trueness is not a quantity and thus cannot be expressed numerically, but measures for closeness of agreement are given in ISO 5725.

Note 2: Measurement trueness is inversely related to systematic measurement error, but is not related to random measurement error.

Note 3: Measurement accuracy should not be used for 'measurement trueness' and vice versa.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

True value: Quantity value consistent with the definition of a quantity.

Notes:

In the error approach to describing measurement, a true quantity value is considered unique and in practice unknowable. The uncertainty approach is to recognize that, owing to the inherently incomplete amount of detail in the definition of quantity, there is not a single true quantity value, but rather a set of quantity values consistent with the definition of a quantity. However, this set of values is, in principle and in practice unknowable. Other approaches dispense altogether with the concept of true quantity value and rely on the concept of metrological compatibility of measurement results for assessing their validity.

When the definitional uncertainty associated with the measurand is considered to be negligible compared to the other components of the measurement uncertainty the measurand may be considered to have an essentially “unique” true value.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Validation: Verification, where the specified requirements are adequate for an intended use.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Validated Test Method: An accepted test method for which validation studies have been completed to determine the accuracy and reliability of this method for a specific purpose.

Reference:

ICCVAM Guidelines for the nomination and submission of new, revised and alternative test methods, 2003

Validated range: That part of the concentration range of an analytical method which has been subjected to validation.

Reference:

Harmonized guidelines for single-laboratory validation of methods of analysis, 2002

Verification: Provision of objective evidence that a given item fulfils specified requirements.

Notes:

When applicable method uncertainty should be taken into consideration.

The item may be e.g. a process, measuring procedure, material, compound or measuring system.

The specified requirement may be that a manufacturer's specifications are met.

Verification in legal metrology, as defined in VIM and in conformity assessment in general pertains to the examination and marketing and/or issuing of a verification certificate for a measuring system.

Verification should not be confused with calibration. Not every verification is a validation.

In chemistry, verification of the identity of the entity involved or of the activity, requires a description of the structure and properties of that entity or activity.

References:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

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APPENDIX IV

STATUS OF ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING

A. Codex Committee on Nutrition and Foods for Special Dietary Uses

B. Codex Committee on Processed Fruits and Vegetables

C. FAO/WHO Coordinating Committee for Asia

D. Codex Committee on Sugars

A. COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants, CODEX STAN 72-1981[1]

Provision	Method	Principle	Type
Calories (by calculation)	Method described in CAC/Vol IX-Ed.1, Part III[2]	Calculation	**Type I**
Total fat	AOAC 989.05 ISO 8381|IDF 123:2008	Gravimetry (Röse-Gottlieb)	**Type I**
Total fat for milk-based infant formula (Products not completely soluble in ammonia)	ISO 8262-1 |IDF 124-1: 2005	Gravimetry (Weibull-Berntrop)	**Type I**
Fatty acids (including trans fatty acid)	AOAC 996.06	Gas chromatography	**Type II**
Fatty acids (including trans fatty acid)	AOCS Ce 1h-05	Gas chromatography	**Type III**
Total phospholipids	AOCS Ja7b-91	Gas chromatography with suitable extraction and preparation procedures	**Type III**

[1] ALINORM 09/32/26, Appendix VI

[2] Section 9. Calories by calculation – Section 9.2 Conversion Factors
(a) protein 4 kcal per g
(b) carbohydrate 4 kcal per g
(c) fat 9 kcal per g
(d) monosaccharides 3.75 kcal per g
(e) specific food ingredients See "Energy and Protein Requirements"(FAO Nutrition Meeting Report Series No. 52 or WHO Technical Report Series No. 522)
(f) other specific calorie conversion factors maybe used where the formulation of the food and the nutrient content are known and where such specific conversion factors are physiologically more meaningful than the factors listed above

Total carbohydrates Moisture/Total Solids Ash	AOAC 986.25 AOAC 990.19 or AOAC 990.20 IDF 21B:1987 or ISO 6731:1989 AOAC 942.05	Determination by difference Gravimetry Gravimetry	**Type I**
Vitamin A	AOAC 992.04 (retinol isomers)	HPLC	**Type II**
Vitamin A above 500 IU/l milk after reconstitution	AOAC 992.06 (retinol)	HPLC	**Type III**
Vitamin A	EN 12823-1:2000 (all-trans-retinol and 13-cis-retinol) Vitamin A (both natural + supplemental ester forms) aggregated and quantified as individual retinol isomers (13 - cis and all-trans)	HPLC	**Type III**
Vitamin D	AOAC 992.26 D_3 measured	HPLC	**Type III**
Vitamin D	EN 12821:2000 (D2 and/or D3 measured as single components. Hydroxylated forms not measured.) NMKL 167: 2000	HPLC	**Type II**
Vitamin D	AOAC 995.05 D2 and D3 measured	HPLC	**Type III**
Vitamin E	AOAC 992.03 Measures all rac-vitamin E (both natural + supplemental ester forms) aggregated and quantified as α-congeners	HPLC	**Type III**
Vitamin E	EN 12822: 2000 (Measures Vitamin E (both natural +	HPLC	**Type II**

	supplemental ester forms) aggregated and quantified as individual tocopherol congeners (α,.β,.γ, δ).		
Vitamin K	AOAC 999.15 EN 14148:2003 (vitamin K_1) (Measures either aggregated cis + trans K_1 or can measure individual cis and trans forms depending on LC column.)	HPLC with C30 column to separate the cis- and the trans- K vitamins	**Type II**
~~Thiamine~~	~~AOAC 942.23~~	~~Fluorimetry~~	**Delete from the list of methods for Special Foods**
Thiamine	AOAC 986.27[3]	Fluorimetry	**Type III**
Thiamine	EN 14122:2003 (Measures all vitamin B_1 forms (natural and added free, bound and phosphorylated) following extraction and conversion to thiamine)	HPLC with pre-or post column derivatization to thiochrom	**Type II**
Riboflavin	AOAC 985.31[4]	Fluorimetry	**Type III**
Riboflavin	EN 14152:2003 (Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin.)	HPLC	**Type II**
Niacin	AOAC 985.34 (niacin (preformed) and nicotinamide)	Microbioassay and turbidimetry	**Type III**
Niacin	prEN 15652:2009 (Free and bound and phosphorylated forms measured either as aggregate of nicotinic acid + nicotinamide, or as individual forms)	HPLC	**Type II** when published as EN method
Vitamin B_6	AOAC 985.32	Microbioassay	**Type III**

[3] Care should be taken in the application of the method due to spectral interference
[4] Care should be taken in the application of the method due to spectral interference

Vitamin B_6	EN 14166:2008 (Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine)	Microbioassay	**Type III**
Vitamin B_6	AOAC 2004.07 EN 14164:2008 (Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine)	HPLC	**Type II**
Vitamin B_6	EN 14663:2005 (includes glycosylated forms) (Free and bound phosphorylated and glycosylated forms measured as the individual forms pyridoxal, pyridoxine and pyridoxamine)	HPLC	**Type III**
Vitamin B_{12}	AOAC 986.23 (Measures total vitamin B_{12} as cyanocobalamin)	Turbidimetric Method	**Type II**
Pantothenic acid	AOAC 992.07 (Measures total pantothenate (free pantothenic acid + CoA- + ACP-bound) and measured as D-pantothenic acid (or calcium D-pantothenate)	Microbioassay	**Type II**
Folic acid	AOAC 992.05 (Measures free folic acid + free, unbound natural folates, aggregated and measured as folic acid) EN 14131:2003 (Total folate (free + bound), aggregated and measured as folic acid)	Microbioassay	**Type II**
Folic acid	J AOAC Int. 2000:83; 1141-1148	Optical Biosensor	**Type IV**

	(Measures free folic acid + proportion of free, natural folate)	Immunoassay	
Folic acid	J Chromatogr. A., 928, 77-90, 2001 (Measures total folates after conversion to, and measurement as 5-Me-H4PteGlu)	HPLC, incorporating immunoaffinity clean-up and conversion to 5-methyltetrahydrofolate	**Type IV**
Vitamin C	EN 14130:2003 (Measures ascorbic acid + dehydroascorbic acid)	HPLC	**Type II**
Biotin	EN 15607:2008 (d-biotin) (Measures total D-biotin (free + D-biocytin)	HPLC	**Type II**
Iron[5]	AOAC 985.35	Flame atomic absorption spectrophotometry	**Type III**
Iron	AOAC 984.27	ICP emission spectroscopy	**Type III**
Iron	AOAC 999.11 | NMKL139:1991		**Type II**
Calcium	ISO 8070 | IDF 119: 2007	Flame atomic absorption spectrophotometry	**Type II**
Calcium	AOAC 985.35	Flame atomic absorption spectroscopy	**Type III**
Calcium	AOAC 984.27	ICP emission spectroscopy	**Type III**
Phosphorus	AOAC 986.24	Spectrophotometry (molybdovanadate)	**Type II**
Phosphorus	AOAC 984.27	ICP emission spectroscopy	**Type III**
Magnesium	ISO 8070 | IDF 119: 2007	Flame atomic absorption spectrophotometry	**Type II**
Magnesium	AOAC 985.35	Flame atomic absorption spectroscopy	**Type III**
Magnesium	AOAC 984.27	ICP emission spectroscopy	**Type III**

[5] General Codex methods are also available

Chloride	AOAC 986.26	Potentiometry	**Type III**
Manganese	AOAC 985.35	Flame atomic absorption spectrophotometry	**Type II**
Manganese	AOAC 984.27	ICP emission spectroscopy	**Type III**
Iodine (for milk-based formula)	AOAC 992.24	Ion-selective potentiometry	**Type II**
Selenium	AOAC 996.16 or AOAC 996. 17	Continuous hydride generation Flame atomic absorption spectrometry (HGAAS)	**Type III**
Selenium	EN 14627:2005	Hydride generation atomic absorption spectrometry (HGAAS)	**Type II**
Selenium	AOAC 2006.03	ICP emission spectroscopy	**Type III**
Copper	AOAC 985.35	Flame atomic absorption spectroscopy	**Type II**
Copper	AOAC 984.27	ICP emission spectroscopy	**Type III**
Zinc	AOAC 985.35	Flame atomic absorption spectroscopy	**Type II**
Zinc	AOAC 984.27	ICP emission spectroscopy	**Type III**
Choline	AOAC 999.14	Enzymatic Colorimetric Method with limitations on applicability due to choline and ascorbate concentration.	**Type II**
Chromium (Section B of STAN 72 only)	EN 14082:2003	Graphite furnace atomic absorption after dry ashing	**Type II**
Chromium (Section B of STAN 72 only)	EN 14083:2003	Graphite furnace AAS after pressure digestion	**Type III**
Chromium (Section B of STAN 72 only)	AOAC 2006.03	ICP emission spectroscopy	**Type III**

Molybdenum (Section B of STAN 72 only)	EN 14083:2003	Graphite furnace AAS after pressure digestion	**Type II**
Molybdenum (Section B of STAN 72 only)	AOAC 2006.03	ICP emission spectroscopy	**Type III**

B. CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES[6]

1. Methods of Analysis

Draft Standard for Certain Canned Vegetables (At Step 8)

Draft Standard for Jams and Jellies (at Step 8)

Standard for Aqueous Coconut Products: Coconut Cream and Coconut Milk (CODEX STAN 240-2003)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Type
Jams and jellies	Fill of Containers	CAC/RM 46-1972	Weighing	**Type I**
Jams and Jellies	Soluble solids	ISO 2173:2003 AOAC 932.12	Refractometry	**Type I**
Jams and Jellies	Calcium	AOAC 968.31	Complexometry/ Titrimetry	**Revocation**
Jams and Jellies	Mineral impurities (sand)	AOAC 971.33	Gravimetry	**Revocation**
Certain Canned Vegetables	Mineral impurities (sand)	AOAC 971.33	Gravimetry	**Type I** Current method for canned palmito: ISO 762:1982 (confirmed 1992)
Canned Green beans	Tough strings	CAC/RM 39-1970	Stretching	**Type I**
Canned Green peas	Types of peas, distinguishing	CAC/RM 48-1972	Visual inspection	**Type I**
Canned Green Peas	Proper fill (in lieu of drained weight)	CAC/RM 45-1972	Pouring and measuring	**Revocation**

[6] ALINORM 09/32/27, Appendices II, III and VI

Canned Green Peas	Solids, alcohol insoluble	AOAC 938.10	Gravimetry including sieving	**Revocation**
Canned Green Peas	Calcium	AOAC 968.31	Complexometry/ Titrimetry	**Revocation**
Canned mature processed peas	Solids, total	AOAC 964.22	Gravimetry (vacuum oven)	**Revocation**
Aqueous Coconut Products	Total Fats	ISO 1211:1999 IDF 1D: 1996	Gravimetry (Röse-Gottlieb)	**Type I**
Aqueous Coconut Products	Total solids	ISO 6731:1989 IDF 21B: 1987	Gravimetry	**Type I**
Aqueous Coconut Products	Non-fat solids	ISO 1211:1999 IDF 1D: 1996 and ISO 6731:1989 IDF 21B: 1987	Calculation: Gravimetry (Röse-Gottlieb) Gravimetry	**Type I**
Aqueous Coconut Products	Moisture	ISO 6731:1989 IDF 21B: 1987	Calculation: Gravimetry	**Type I**

2. Sampling Plans

Commodity	**Sampling Plan**	**Status**
Certain Canned Vegetables	Described in the Draft Standard	Not endorsed (see Agenda Item 5)
Jams and Jellies	Described in the Draft Standard	Not endorsed (see Agenda Item 5)

C. FAO/WHO COORDINATING COMMITTEE FOR ASIA[7]

1. Draft Standard for Gochujang (At Step 8)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Type
Gochujang	Capsaicin	AOAC 995.03	HPLC	Type II (endorsed by 28th CCMAS)
		According to the method described in the Annex to the Standard (see below)	Gas chromatography	Type IV
	Crude protein	AOAC 984.13 (Nitrogen conversion factor: 6.25)	Kjeldahl	Type I
	Moisture	AOAC 934.01 (≤ 70°C, ≤ 50 mm Hg)	Gravimetry	Type I

Determination of capsaicin in *Gochujang* using Gas Chromatography (GC) detection (as proposed by 16th CCASIA with no amendments).

2. Proposed Draft Standard for Fermented Soybean Paste (At Step 5/8)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Type
Fermented Soybean Paste	Total Nitrogen	AOAC 984.13	Kjeldahl	Type I
Fermented Soybean Paste	Amino Nitrogen	AOAC 920.154 on the conditions specified in the standard (see below)	Volumetry	Type I
Fermented Soybean Paste	Moisture	AOAC 934.01 (≤70°C, ≤ 50 mm Hg)	Gravimetry	Type I

Section 9.2 Determination of Amino Nitrogen

Preparation of test samples: Weigh 2 g of sample into a 250 ml beaker and mix the sample with 100 ml of cold (15°C) NH_3-free H_2O and then stir the mixture for 60 min. Next, decant the mixture through a quantitative filter and collect the filtrate in a 100 ml volumetric flask.

Endpoint - A pH meter shall be used to determine the endpoint instead of optical verification of colours

[7] ALINORM 09/32/15, Appendix II, IV and V

3. Proposed Draft Standard for Edible Sago Flour (At Step 5)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Type
Sago Flour	Moisture Content	ISO 712:1998	Gravimetry	Type I
Sago Flour	Ash (inorganic extraneous matter)	ISO 2171: 2007	Gravimetry	Type I
Sago Flour	Acidity	AOAC 939.05	Titrimetry	Type I
Sago Flour	Crude Fibre	ISO 6541:1981	Gravimetry	Type I
Sago Flour	Starch	AOAC 920.44.	Gravimetry	Type I

D. CODEX COMMITTEE ON SUGARS

Standard for Sugars

COMMODITY	PROVISION	METHOD	PRINCIPLE	Type
Plantation and Mill White Sugar	Colour	ICUMSA GS9/1/2/3-8	Photometry	I

PROPOSED AMENDMENTS TO THE PROCEDURAL MANUAL

WORKING INSTRUCTIONS FOR THE IMPLEMENTATION OF THE CRITERIA APPROACH IN CODEX

Add the following note above Table 1

Note: These criteria are applicable to fully validated methods except for methods such as PCR and ELISA, which requires other set of criteria.

In Table 1
Precision should be described as
$RSD_R \leq 2.\ PRSD_R$

Add the following text at the end of the section:

GUIDELINES FOR ESTABLISHING NUMERIC VALUES FOR METHOD CRITERIA AND/OR ASSESSING METHODS FOR COMPLIANCE THEREOF.

1. RECOMMENDATIONS FOR ESTABLISHING NUMERIC VALUES FOR METHOD CRITERIA

Only the provision for the commodity along with its ML (maximum level, minimum level, normative level or concentration range) is needed when establishing numeric values for method criteria.

Note: These criteria are applicable to fully validated methods except for methods such as PCR and ELISA, which requires other set of criteria.

1.1 The applicability

The method has to be applicable to the particular analyte(s)/provision(s) in the specified matrix/ commodity or food category. For horizontal methods the relevant food categories should have been tested. Furthermore, it should have been shown that the method is applicable for concentrations levels around the specified ML, i.e. the ML should be within the validated range.

- For ML $\geq 10^{-7}$, the minimum applicable range should be: ML $\pm 3s_R$
- For ML $< 10^{-7}$, the minimum applicable range should be: ML $\pm 2s_R$

The minimum applicable concentration range should correspond to an interval containing a large fraction of the expected variation (due to measurement uncertainty) in the results around the specified limit (ML). For collaboratively validated methods the expected variation would be the reproducibility standard deviation (s_R) multiplied with a coverage factor. A coverage factor of 2 corresponds to a confidence level of approx. 95%, and a coverage factor of 3 corresponds to a confidence level about 99%. As 99% is often used as an action level in control charts, a coverage factor of 3 is recommended for concentration ratios at or above 10^{-7}, (≥ 0.1 mg/kg). For concentrations lower than 0.1 mg/kg, a coverage factor of 2 is recommended, as a coverage factor of 3 would make it hard to find applicable methods for certain analytes/provisions due to the low level.

Calculation of the minimum applicable range for specified MLs:

The minimum applicable range can be estimated based on the Horwitz/Thompson equation for reproducibility standard deviation, s_R.

1.1.1: For concentration ratios $\geq 10^{-7}$ (≥ 0.1 mg/kg) the Horwitz' equation is applied:

$$PRSD_R\,(\%) = 100 \cdot s_R/c = 2C^{-0.1505}$$

where

$PRSD_R$ is the "predicted" relative standard deviation,
s_R is the predicted standard deviation
c is the concentration of interest, which here is the ML and
C is the concentration ratio, i.e. the concentration ratio of ML ($\mathbf{C}_{ML}$)

By rearranging the equation with respect of s_R, the following equation is obtained:

$$s_R = \frac{c \cdot 2C^{-0.1505}}{100} = \frac{ML \cdot 2 \cdot C_{ML}^{-0.1505}}{100}$$

Example 1: ML =0.1 mg/kg, $C_{ML} = 10^{-7}$:

$$0.1 \pm 3 \cdot s_R = 0.1 \pm 3 \cdot \frac{0.1 \cdot 2 \cdot (0.0000001)^{-0.1505}}{100} = 0.1 \pm 0.07 \text{ mg/kg}$$

The minimum applicable range for a ML of 0.1 mg/kg is then 0.03 to 0.17 mg/kg

Example 2: For a ML of 1 mg/kg (i.e. 10^{-6}):

$$1.0 \pm 3 \cdot s_R = 1.0 \pm 3 \cdot \frac{1.0 \cdot 2 \cdot (0.000001)^{-0.1505}}{100} = 1.0 \pm 0.48 \text{ mg/kg}$$

The minimum applicable range for ML of 1 mg/kg is then 0.5 to 1.5 mg/kg

1.1.2: For concentration ratios < 10^{-7}, the Thompson theory is applied, i.e. $PRSD_R$ = 22% and hence $s_R = 0.22 \cdot ML$

Example 3: ML = 0.01 mg/kg (i.e. 10^{-8}):

$$0.01 \pm 2 \cdot s_R = 0.01 \pm 2 \cdot (0.22 \cdot ML) = 0.01 \pm 0.44 \cdot 0.01 = 0.01 \pm 0.0044 \text{ mg/kg}$$

The minimum applicable range for a ML of 0.01 mg/kg is then 0.006 to 0.014 mg/kg.

In table 1, a number of minimum applicable concentration ranges for specified MLs are given.

Table 1: Recommended criteria for minimum application range for specified MLs

ML (mg/kg)	**0.01**	**0.02**	**0.05**	**0.1**	**1**	**10**	**100**
Lower level:	0.006	0.011	0.028	0.03	0.52	6.6	76
Upper level: *	0.014	0.029	0.072	0.17	1.48	13.3	124

* Upper level will seldom be the limiting factor like the lower level.

1.2 Limit of Detection (LOD) and limit of Quantification (LOQ)

As an alternative to establishing minimum applicable range, the criteria could be numeric values for LOD and LOQ.

The numeric value for the limit of detection (LOD), should be:

- no more than 1/10 of the specified ML for levels at or above 0.1 mg/kg, and
- no more than 1/5 of the specified ML for levels below 0.1 mg/kg.

The numeric value for the limit of quantification (LOQ) should be:

- no more than 1/5 of the specified ML for levels at or above 0.1 mg/kg, and
- no more than 2/5 of the specified ML for levels below 0.1 mg/kg.

1.3 The method precision, derived from collaborative method performance studies

The precision should be expressed as the obtained relative reproducibility standard deviation (RSD_R) obtained from collaborative method performance studies, which is compared to the predicted relative reproducibility standard deviation ($PRSD_R$)
According to Horwitz, the ratio between the found and the predicted value should be ≤ 2 (known as the HorRat value), this is also applicable for Thompson equation of $PRSD_R$ = 22%:

$$\frac{RSD_R}{PRSD_R} \leq 2 \quad \Leftrightarrow \quad RSD_R \leq 2 \cdot PRSD_R$$

The numeric values for the precision given in table 2 are also based on the Horwitz/Thompson equation. For some analyses, using advanced techniques, a better precision can be obtained.

Table 2. Precision requirement at different concentrations based on the Horwitz/Thompson equation.

	Thompson	*Horwitz equation* $(2C^{-0.1505})$							
Concentration ratio (C)	$< 10^{-7}$	10^{-7}	10^{-6}	10^{-5}	10^{-4}	10^{-3}	10^{-2}	10^{-1}	1
Concentration unit	< 0.1 mg/kg	0.1 mg/kg	1 mg/kg	10 mg/kg	0.1 g/kg	1 g/kg	10 g/kg	100 g/kg	1000 g/kg
$PRSD_R$ (%)	22	22	16	11	8	6	4	3	2
RSD_R ≤ 2 · $PRSD_R$ (%)	≤ 44	≤ 44	≤ 32	≤ 22	≤ 16	≤ 12	≤ 8	≤ 6	≤ 4

$PRSD_R$ = predicted value for relative standard deviation of reproducibility.
RSD_R = found value for the relative standard deviation of reproducibility in a collaborative study.

1.4 Recovery

Evaluation and estimation of recovery is included in the method validation. Whether or not recovery is of relevance depends on the method procedure.

1.5 Trueness

For the evaluation of trueness preferably appropriate certified reference materials (CRMs) should be analysed and demonstrated to give the certified value (allowing for measurement uncertainty) is achieved.

1.6 Examples on how to establish criteria for a provision

In order to illustrate how to set criteria for a provision the following example is used:

According to Codex Standard 1993-1995, Rev 2-2006, General Standard for contaminants and toxins in food, the ML for lead in fruit juices is 0.05 mg/kg. According to the recommendations for obtaining numeric values for the characteristics based on the ML, the criteria would be those in table 3:

Table 3. Recommendation for numeric criteria values for lead in fruit juice

Applicability: Analyte:	Lead
Matrix/provision:	Juice
ML:	0.05 mg/kg
Lower level of min. application range:	≤ 0.03 mg/kg (= ML - $2s_R$ = 0.05 mg/kg - 0.44 · 0.05 mg/kg). See Table 1
LOD:	≤ 0.01 mg/kg (= ML · 1/5 = 0.05 mg/kg · 1/5)
LOQ:	≤ 0.02 mg/kg (= ML · 2/5 = 0.05 mg/kg · 2/5)
Precision:	For concentration at 0.05 mg/kg, the RSD_R ≤ 44%, See Table 2
Recovery:	The method procedure does not include an extraction step and hence recovery is of no relevance.
Trueness:	Use of CRM.

2. METHOD CRITERIA AT DIFFERENT MLS (MAXIMUM LEVEL, MINIMUM LEVEL, NORMATIVE LEVEL OR CONCENTRATION RANGE)

In table 4 examples on method criteria are given for certain MLs.

Table 4: Method criteria for MLs at increasing orders of magnitude.

ML unit	0.001 mg/kg	0.01 mg/kg	0.1 mg/kg	1 mg/kg	10 mg/kg	100 mg/kg	1 g/kg	10 g/kg
Concentration ratio of ML (C_{ML})	10^{-9}	10^{-8}	10^{-7}	10^{-6}	10^{-5}	10^{-4}	10^{-3}	10^{-2}
Minimum applicable Range	From 0.0006 to 0.0014 (mg/kg)	From 0.006 to 0.014 (mg/kg)	From 0.03 to 0.17 (mg/kg)	From 0.52 to 1.48 (mg/kg)	From 6.6 to 13.3 (mg/kg)	From 76 to 124 (mg/kg)	From 0.83 to 1.2 (g/kg)	From 8.8 to 11 (g/kg)
LOD (≤ mg/kg)	0.0002	0.002	0.01	0.1	1	10	100	1000
LOQ (≤ mg/kg)	0.0004	0.004	0.02	0.2	2	20	200	2000
RSD_R (≤ %)	44	44	44	32	22	16	12	8
Recovery (%) *	40 - 120	60 - 115	80 - 110	80-110	80 - 110	90 - 107	95 - 105	97 - 103

* Other guidelines are available for expected recovery ranges in specific areas of analysis. In cases where recoveries have been shown to be a function of the matrix other specified requirements may be applied.

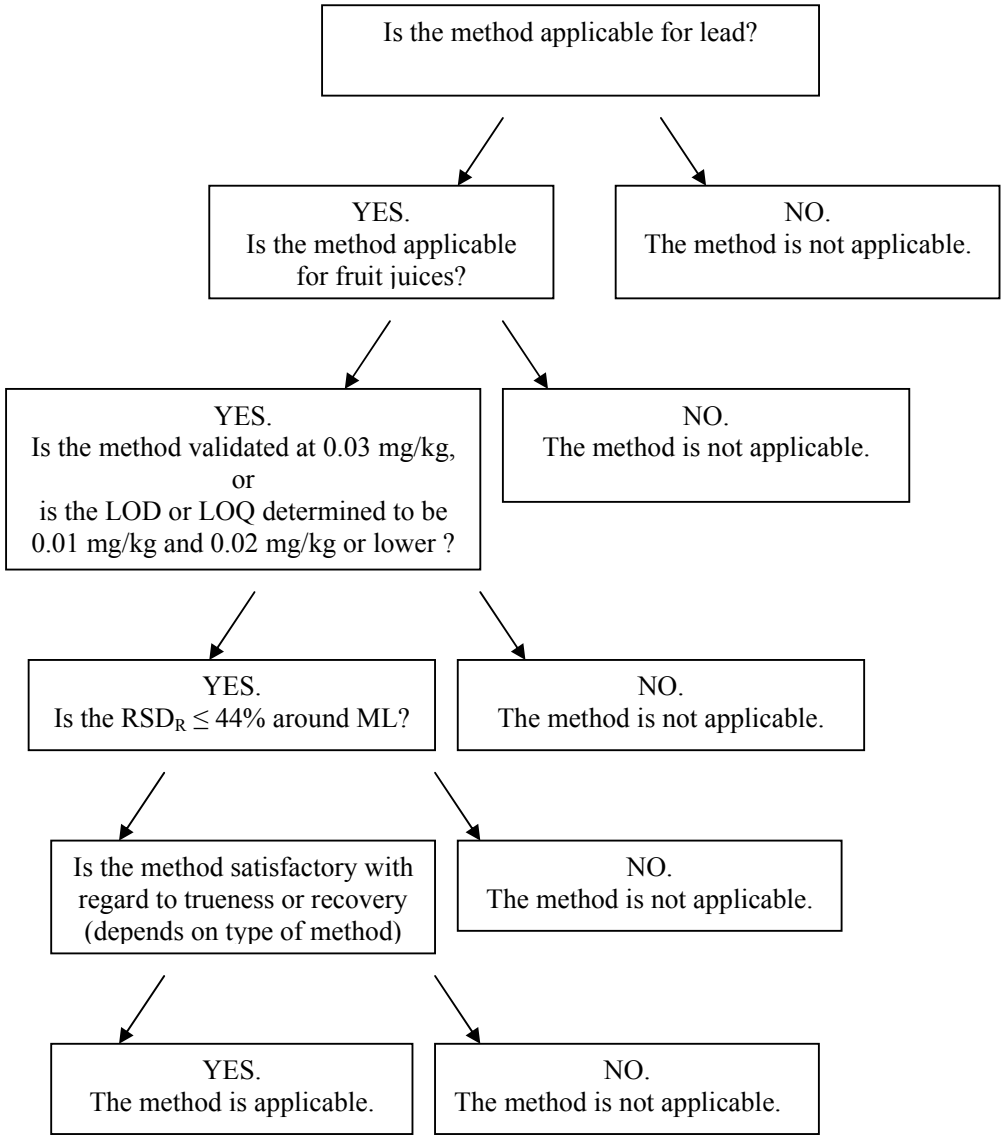
2.1 How to elucidate a method's compliance with the criteria.

To review a method for possible compliance with the established criteria, the method performance characteristics have to be assessed. The result of a method performance study is available in the method and/or published in an international journal.

2.1.1 Example on assessing methods for compliance

Continuing the example above on lead in fruit juice, having ML of 0.05 mg/kg, the methods considered should be able to quantify lead in fruit juice as low as 0.03 mg/kg, with a precision, $PRSD_R$ of 22%, the RSD_R obtained from the method performance study should then not be higher than 44% (corresponding to a 95% confidence interval).

When assessing a method for compliance, the following steps should be considered:



In order to find appropriate methods for this purpose, information are collected on methods for determination of lead. (As this is an example in the Procedural Manual, the methods' identification is omitted):

Table 5: Collaboratively validated methods for analysis of lead

Method No	Applicability	Principle	Assessed level (mg/kg)	LOD (mg/kg)	RSD_R (%)	Applicable Yes/No and why
1	All foods	Flame AAS	2.2 – 29		4.9-36	NO Flame AAS will not be able to detect at 0.05 mg/kg
2	All Foods (Chicken, apple)	Anodic stripping voltammetry	0.03-2.8	0.03	17-106	NO The RSD_R is 106% (not <44%) at 0.03 mg/kg
3	Sugars	GF-AAS	0.03-0.50		12-30	YES Even if the applicability does not say Juice (or all foods) it should be considered applicable as fruit juice contains a lot of sugar. The precision is satisfactory.
4	Fats and Oils	GF-AAS	0.018-0.090		5.9-30	NO The method describes sample prep. for fats and oils only.
5	Natural mineral water	AAS	0.0197-0.977	< 0.01	2.8-4.2	NO The method describes sample prep. for water only.
6	All foods	GF-AAS after dry ashing	0.045-0.25	< 0.01	26-40	NO The lowest validated level is not low enough, however as the technique is GF-AAS, it should be applicable for 0.03 mg/kg.
7	All foods except oils, fats and extremely fatty products.	AAS after microwave oven digestion under pressure.	0.005-1.62	0.014	26-44	YES Validation level and RSD_R are ok
8	All foods	ICP-MS after pressure digestion	0.013-2.45	< 0.01	8-47	YES Validation level and RSD_R are ok for levels of 0.03 mg/kg and above.

AAS = Atomic Absorption Spectrometry
GF-AAS = Graphite Furnace Atomic Absorption Spectrometry
ICP-MS = Inductive Coupled Plasma - Mass Spectrometry

Conclusion: Methods No. 3, 7 and 8 are found applicable for the determination of lead in fruit juices for the given ML of 0.05 mg/kg. Assessing methods for compliance requires knowledge about the methods; sample preparation, procedures and instrumentation. Thus the methods cannot be "judged" by numeric values for the criteria alone.

PROPOSED AMENDMENT TO THE PROCEDURAL MANUAL

Guidelines for the Inclusion of Specific Provisions in Codex Standards and Related Texts

General Criteria for the Selection of Methods of Analysis

Section (b) is amended as follows:

(b) Preference should be given to methods of analysis the reliability of which has been established in respect of the following criteria, selected as appropriate:

(i) ~~specificity~~ **selectivity**

The remainder of the section is unchanged.