codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 2 **CX/GP 01/2**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON GENERAL PRINCIPLES
Sixteenth Session
Paris, France, 23 - 27 April 2001

MATTERS REFERRED BY THE CODEX ALIMENTARIUS COMMISSION AND OTHER CODEX COMMITTEES

COMMITTEE ON FOOD HYGIENE (33rd Session, October 2000)

The Committee decided to propose an amendment to its terms of reference to reflect its work in the area of risk assessment and risk management, and to forward it to the CCGP for consideration, as follows:

Section III of the Procedural Manual. Subsidiary Bodies under Rule IX.1 (B) (i)

Add the following provisions to the Terms of Reference of CCFH:

- To suggest and prioritize areas where there is a need for risk assessment at the international level and to develop questions to be addressed by the risk assessors
- To consider risk management matters in relation to food hygiene and in relation to the risk assessment of FAO and WHO[1]

COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING (23rd Session, March 2001)

Background

The 11th Session of the CCGP recommended that relevant Committees consider the texts included in the G*uidelines for the Inclusion of Specific Provisions in Codex Standards and Related texts* to review and rephrase them in a way which would give them general applicability (ALINORM 95/33, para. 50). The provisions in the *Guidelines* concerning methods of analysis and sampling had not been revised as the question of criteria for the selection of methods was still under consideration in the CCMAS and no conclusion had been reached.

The 14th Session of the Committee noted that the CCMAS had undertaken work to revise the *Principles for the Establishment or Selection of Codex Methods of Analysis* and the Section of the Procedural Manual dealing with the relations between Codex Commodity Committees and General Subject Committees on the matter of methods of analysis and sampling. It was noted that this work was subsequent to recommendations made by the 11th Session of CCGP to relevant Codex Committees. The Committee agreed that the objective of such revisions was to ensure that the Procedural Manual contained only matters relating to the Commission's procedures; guidelines or other recommendations that were better directed to Member governments should be transferred from the Procedural Manual, revised as appropriate, and after adoption by the Commission, included in the Codex Alimentarius.

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1 (ALINORM 01/13A, paras. 28-30, Appendix III)

CCMAS (23rd Session, March 2001)

The CCMAS had been discussing the application of the criteria approach for several sessions. The 23rd Session of the CCMAS agreed that the criteria approach would be applied to Type III methods of analysis and that Type II methods would be retained to be used in dispute situations. The Committee proposed the following amendments to the Procedural Manual to reflect this new approach:

- General Criteria for the Selection of Methods of Analysis Using the Criteria Approach
- Relations between Commodity Committees and General committees – Methods of Analysis and Sampling

The Committee also considered Guidelines intended to clarify the application of the criteria approach. As it was recognized that these Guidelines were currently intended for use in the framework of Codex, the Committee agreed to propose their inclusion in the Procedural Manual at the end of the current Section on "Principles for the Establishment of Codex Methods of Analysis" with the following title:

- Guidelines and Working Instructions to Aid the Implementation of the Criteria Approach to the Selection of Methods of Analysis for Codex Purposes

Following the recommendations of the CCGP and the Executive Committee concerning the development of texts for application by governments and inclusion in Codex Alimentarius Volume 13, the CCMAS also agreed to redraft the current Guidelines to make them generally applicable by governments, for circulation at Step 3 and consideration by the next session of the Committee (ALINORM 01/23, paras. 33-34 and 41, Appendix II).

The CCGP is invited to consider the proposed changes included in **Annex 1**.

COMMITTEE ON FOOD IMPORT AND EXPORT INSPECTION AND CERTIFICATION SYSTEMS (9th Session, December 2000)

The Committee discussed the question of traceability and agreed that the Committee on General Principles should be informed of its discussion, as follows (ALINROM 01/30A, paras. 110-114).

TRACEABILITY

110. The Delegation of Japan introduced a brief paper on the matter of traceability[2] in which it noted that this issue had been referred to, or was currently being discussed by various Codex Committees including CCFICS, Committee on Fish and Fishery Products, Task Force on Animal Feeding, and the Task Force on Foods Derived from Biotechnology. It stated that the concept of traceability cut across a wide range of food issues. It further noted that, as yet, there had not been a forum under the Codex Alimentarius Commission in which a comprehensive discussion had taken place on the issue and that Codex had not yet defined the purpose and framework of this concept. The Delegation was of the opinion that due to the importance of this concept in relation to food import and export inspection and certification systems it would be an appropriate matter for the Committee to discuss. The Committee expressed its appreciation to the Delegation of Japan for raising the issue and agreed that the points raised needed to be addressed within the Codex framework.

111. At the request of the Chairperson, the Secretariat noted that different Codex Committees and Task Forces had undertaken either prior or current work related to traceability including the Committees on Food Hygiene, Food Labelling, and Food Additives and Contaminants in addition to the subsidiary bodies mentioned by Japan. The Secretariat noted that the modalities required for systems of traceability seemed to fall within the terms of reference of CCFICS whereas consideration of a Codex-wide definition of the concept would logically fall within the work of the Committee on General Principles.

112. The Representative of the European Commission stated that traceability was an instrument of risk management and as such should be considered by the Committee on General Principles. Moreover, in the opinion

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of the Representative, the issue was not exclusively related to food safety. For example in the area of organic foods or food claimed to be "GMO-free" it was a matter of ensuring the integrity of the product in relation to consumer confidence. Because it was such a general concept, the Representative recommended that the Committee on General Principles should establish a definition and establish general orientations.

113. The Delegation of Canada, supported by several other delegations, stated that there was a need for a general discussion paper on the status and use of the concept in which the problems, challenges and opportunities to Codex would be highlighted. The Delegation of the Republic of Korea stated that this was an important issue for food safety systems involved in international trade. The Representative of the International Association of Consumer Food Organizations proposed that consideration could be given to a "bottom up" approach, allowing a more general definition to be derived from the practical application of the concept by individual committees within their terms of reference. The Delegation of the United States was of the opinion that emphasis should be placed on the purpose and application of the concept rather than a definition. The Delegation of New Zealand was of the opinion that contemporary experience in the use of the concept at the national level should be identified and examples included in any discussion paper.

114. The Committee agreed that within its Terms of Reference it had a responsibility to consider work in this area and that there was need for a substantive discussion of the issue at its next meeting. In view of the system-wide interest and involvement in the issue, the Committee recommended that a short paper be prepared by the Secretariat for consideration by the Codex Alimentarius Commission at its next Session in order to obtain the Commission's guidance in this matter. In the meantime, the other relevant Committees and Task Forces, including the Committee on General Principles, would be informed of this recommendation.

ANNEX 1

COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

PROPOSED AMENDMENTS TO THE PROCEDURAL MANUAL

PART 1 – AMENDMENTS TO CURRENT SECTIONS

1. PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS[3]

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

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

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

2. Relations between Commodity Committees and General Committees - Methods of Analysis and Sampling[4]

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

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

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

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

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

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

INTRODUCTION AND BACKGROUND

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

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

4 Procedural Manual, 11th Edition, page 96

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

PRESENT SYSTEM

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

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

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

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

and, in addition,

- the method selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use,
- all proposed methods of analysis must have direct pertinence to the Codex Standard to which they are directed.
- methods of analysis which are applicable uniformly to various groups of commodities should be given preference over methods which apply only to individual commodities.
- official methods of analysis elaborated by international organisations occupying themselves with a food or group of foods should be preferred.

THE NEW APPROACH

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

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

Type I Methods

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

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

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

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

Type II and III Methods

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

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

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

However, the primary responsibility for supplying methods of analysis and criteria resides with the Commodity Committee. If the Commodity Committee fails to provide a method of analysis or criteria despite numerous requests, then CCMAS may supply an appropriate method and "convert" that method into appropriate criteria.

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

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

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

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

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

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

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

Type IV Methods

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

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

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

Conversion of Specific Methods of Analysis to Method Criteria by the CCMAS

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

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

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

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

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

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

Acceptability of the Values Used

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

RETROSPECTIVE ACTION

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

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

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

Terminology

The following terms are defined in the Procedural Manual:

- Accuracy
- Applicability
- Precision
- Selectivity
- Sensitivity

Other Terms to be used in the Criteria Approach

Detection Limit

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

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

Determination limit

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

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

Recovery

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

Selectivity

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

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

Linearity

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

Assessment of the Acceptability of the Precision Characteristics of a Method of Analysis

The calculated repeatability and reproducibility values can be compared with existing methods and a comparison made. If these are satisfactory then the method can used as a validated method. If there is no method with which to compare the precision parameters then theoretical repeatability and reproducibility values can be calculated from the Horwitz equation.
Horwitz trumpet and equation is $RSD_R = 2C^{-0.1505}$
The values derived from this equation are:

Concentration ratio	**RSD_R**		**Concentration ratio**	**RSD_R**
1 (100%)	2		10^{-5}	11
10^{-1}	2.8		10^{-6} (ppm)	16
10^{-2} (1%)	4		10^{-7}	23
10^{-3}	5.6		10^{-8}	32
10^{-4}	8		10^{-9} (ppb)	45

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

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

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

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

$HORRAT_r = RSD_r$ (observed)/RSD_r (predicted)

Values Less Than 120 µg/kg

It should be noted that the equation has been recalculated in the light of recent collaborative trials. This has now been published by Thompson[1], who recommends that for values less than 120 µg/kg, the a constant value for the relative standard deviation of 22% is used. However, for many purposes, e.g. mycotoxins and pesticide residues, the original form is still applicable in many cases.

REFERENCE

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