

codex alimentarius commission



FOOD AND AGRICULTURE
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Agenda Item 4b)

CX/MAS 02/5

JOINT FAO/WHO FOOD STANDARDS PROGRAMME CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Twenty-fourth Session
Budapest, Hungary, 18-22 November 2002

CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS FOR CODEX PURPOSES

PROPOSED AMENDMENT TO THE PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS IN THE PROCEDURAL MANUAL

BACKGROUND

The Codex Committee on Methods of Analysis and Sampling has been discussing the application of criteria for evaluating acceptable methods of analysis for several sessions since 1992. At recent Sessions of the Committee papers have been discussed in which the arguments were given for amending the present Codex procedure whereby the specified numeric values in Codex standards are determined using prescribed methods of analysis and sampling. The methods of analysis and sampling are elaborated and agreed through defined Codex procedures. It was stated that there were a number of criticisms to be made of this Codex procedure, in particular:

1. the analyst is denied freedom of choice and thus may be required to use an inappropriate method in some situations;
2. the procedure inhibits the use of automation; and
3. it is administratively difficult to change a method found to be unsatisfactory or inferior to another currently available.

At its 23rd Session (2001), the Committee has accepted in principle an alternative approach whereby a defined set of criteria with which methods should comply without specifically endorsing specific methods should be adopted. It proposed certain procedures for the implementation of the approach into the Codex system (see ALINORM 01/23, paras 39-41 and Appendix II- Part 1), as follows.

- ◆ *Principles for the Establishment of Codex Methods of Analysis*: inclusion of a new section to establish *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*: inclusion of additional text on the criteria approach

The 16th Session of the Codex Committee on General Principles endorsed these amendments (see also CX/GP 01/2, Annex 1, Part 1). They were subsequently adopted by the 24th Session of the Commission and included in the 12th Edition of the Procedural Manual.

In addition CCMAS had proposed the inclusion of new *Guidelines and Working Instructions* to facilitate the criteria approach, in *the Principles for the Establishment of Codex Methods of Analysis*, to be included in the Codex Procedural Manual (Part 2 of Appendix II, ALINORM 01/23).

These proposals were submitted to the Committee on General Principles for endorsement (see CX/GP 01/2, Annex 1, Part 2). The Delegation of Sweden referred to its written comments proposing several amendments in order to simplify the text, to improve the format of the document, and to ensure that it would be appropriate for inclusion in the Procedural Manual. The Committee did not consider the text in detail due to time constraints and recommended that the CCMAS should reconsider the text at its next session (ALINORM 01/33A, para. 11).

The Delegation of Sweden proposed a revised text prepared in cooperation with Japan and the United Kingdom to the 24th Session of the Commission (ALINORM 01/10, Add.1- Government comments). Some delegations supported the text while other delegations indicated that in view of the highly technical nature of the document, more time was required to consider the amendments proposed and the specialized Committee should review them further. The Commission agreed that the revised text should be referred back to Committee on Methods of Analysis and Sampling for further consideration. (ALINORM 01/41, para. 90).

The revised *Working Instructions for the Implementation of Criteria Approach In Codex* are hereby circulated for comments and consideration by the Committee. Governments and international organizations wishing to submit comments on the text for inclusion in the Procedural Manual should do so in writing, preferably by Email, to the Secretary, Codex Alimentarius Commission Joint FAO/WHO Food Standards Programme, FAO, Viale delle Terme di Caracalla, 00100 Rome, Italy, with a copy to Dr. Mária Váradi, Central Food Research Institute (KÉKI), Herman Ottó út 15, H-1022 Budapest, Fax : +361 212 9853 or 361 355 8928, Email : m.varadi@mail.cfri.hu **before 18 October 2002**.

Proposed Text

WORKING INSTRUCTIONS FOR THE IMPLEMENTATION OF CRITERIA APPROACH IN CODEX

Any Codex Commodity Committee may continue to propose an appropriate method of analysis for determining the chemical entity, or develop a set of criteria to which a method used for the determination must comply. In some cases a Codex Commodity Committee may find it easier to recommend a specific method and request the Codex Committee on Methods of Analysis and Sampling (CCMAS) to “convert” that method into appropriate criteria. The Criteria will then be considered by the CCMAS for endorsement and will, after the endorsement, form part of the commodity standard replacing the recommended method of analysis. If a Codex Commodity Committee wishes to develop the criteria by itself rather than allowing the CCMAS to do so, it should follow instructions given for the development of specific criteria as outlined below. These criteria must be approved 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 the CCMAS may supply an appropriate method and “convert” that method into appropriate criteria.

The minimum “approved” Codex analytical characteristics will include the following numeric criteria as well as the general criteria for methods laid down in the Analytical Terminology for Codex Use (see page 66):

- 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.

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

When a Codex Commodity Committee submits a Type III method to CCMAS for endorsement, it should also submit information on the criteria listed below to enable the CCMAS to convert it into suitable generalized analytical characteristics:

- 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 the Analytical Terminology for Codex Use (see page 66), as are other terms of importance.

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.

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

The calculated repeatability and reproducibility values can be compared with existing methods and a comparison made. If these are satisfactory then the method can be used as a validated method. If there is no method with which to compare the precision parameters then theoretical repeatability and reproducibility values can be calculated from the Horwitz equation. (M. Thompson, *Analyst*, 2000, **125**, 385-386).

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.

Additions to ANALYTICAL TERMINOLOGY FOR CODEX USE

Terms to Be Used in the Criteria Approach

Detection Limit

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

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

Determination limit

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

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

Recovery

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

Selectivity

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

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

Linearity

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