codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 4a) **CX/MAS 01/4**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Twenty-Third Session
Budapest, Hungary, 26 February – 2 March 2001

CRITERIA FOR EVALUATING ACCEPTABLE METHODS OF ANALYSIS FOR CODEX PURPOSES

PROPOSED DRAFT GUIDELINES FOR THE APPLICATION OF THE CRITERIA APPROACH BY THE COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

(At Step 3 of the Procedure)

Background

The 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. Following consideration of this question at the 20th Session of the Committee (1995), the 43rd Session of the Executive Committee (1996) approved new work on "Criteria for evaluating acceptable methods of analysis for Codex purposes" in general terms (ALINORM 97/3, Appendix III).

The 22nd Session of the Committee on Methods of Analysis and Sampling agreed to prepare working guidelines for implementation of the criteria approach. The Committee requested the United Kingdom, together with Canada, Australia, Finland, France, Germany, Netherlands, Norway, United States and the Codex Secretariat, to prepare a draft of the guidelines for consideration at its next session. In drafting the document the content of the *Recommendations for a Checklist of Information Required to Evaluate Methods of Analysis and Sampling for Endorsement* should be taken into consideration. If the paper was to contain examples, they should be drafted in such a way that they would provide practical instructions on the implementation of the criteria approach.

As the work on criteria had been approved in general terms by the CCEXEC, the Guidelines are hereby circulated for comments at Step 3. However, confirmation from the CCEXEC may be necessary as regards the specific development of guidelines and their intended status. The Committee will need to clarify whether this text should be developed as Codex Guidelines for advice to governments and therefore for subsequent inclusion in Codex Volume 13 or as instructions for Codex Committees in the Procedural Manual.

Governments and international organizations wishing to submit comments should do so in writing (if possible by Email) to the Secretary, 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 15 January 2000**

Note: As regards dispute situations, the status of discussions and the advice provided by the Committee on Food Import and Export Inspection and Certification Systems will be presented in a separate paper (CX/MAS 01/4-Add.2).

INTRODUCTION

1. At recent Sessions of the Codex Committee on Methods of Analysis and Sampling 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 sampling and analysis. 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.

2. The Committee has accepted in principle an alternative approach whereby a defined set of criteria to which methods should comply without specifically endorsing specific methods should be adopted.

3. The Committee agreed that:

1. This "criteria" approach gives greater flexibility than the present procedure adopted by Codex, and in the case of non-defining methods, eliminates the need to consider and endorse several Type III methods. The Committee recognised that the endorsement of many Type III methods for any specific determination does, in practice, rarely occur; that this reduces the effectiveness of the present Codex system for the endorsement of methods was appreciated,
2. In some areas of food analysis there are many methods of analysis which are available, which meet Codex requirements as regards method characteristics, but which are not considered by CCMAS and the Commission because of time constraints on the Committee,
3. The adoption of a more generalised approach would ensure that such methods are brought into the Codex system and does not disadvantage developments being undertaken elsewhere in the Analytical Community, and
4. It may be necessary to continue to prescribe a single Type II reference method for the dispute situation but the criteria approach could certainly be applied to the present Type III methods.

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4. The Committee requested the United Kingdom, together with Canada, Australia, Finland, France, Germany, Netherlands, Norway, Unites States and the Codex Secretariat, to prepare a draft of the guidelines for consideration at its next Session. In drafting the document the content of the *Recommendations for a checklist of Information Required to Evaluate Methods of Analysis and Sampling for Endorsement* should be taken into consideration.

5. "Proposed Draft Guidelines And Working Instructions To Aid The Implementation Of The Criteria Approach To The Selection Of Methods Of Analysis For Codex Purposes" have been drafted by that Working Group and are attached as Appendix I of this paper. [1]

RETROSPECTIVE ACTION

6. There are a large number of methods already adopted by Codex. It is suggested that these 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.

Secretariat Note

The Secretariat wishes to draw attention to the fact that as guidelines in the Procedural Manual of the Codex Alimentarius Commission, the proposed text would not have relevance for application by member governments, nationally, bilaterally or in other fora. The Committee may wish to take the opportunity to redraft the Guidelines in a manner that would establish them as an official Codex recommendation.

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[1] The text has been edited by the Secretariat to ensure that the examples are in conformity with adopted Codex standards and related texts

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

INTRODUCTION AND BACKGROUND

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

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

PRESENT SYSTEM

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

- defining or empirical procedures, where the analytical result is method dependent (e.g. the determination of "fat" in a food), and

- the determination of a discrete chemical entity where the analytical result is not, in principle, method dependent (sometimes known as rational methods).

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

- specificity

- accuracy

- precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories

- limit of detection

- sensitivity

- practicability and applicability under normal laboratory conditions

- other criteria which may be selected as required.

and, in addition,

- the method selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use,

- all proposed methods of analysis must have direct pertinence to the Codex Standard to which they are directed.

- methods of analysis which are applicable uniformly to various groups of commodities should be given preference over methods which apply only to individual commodities.

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

THE NEW APPROACH

The new approach will only apply to the determination of specific chemical analytes (i.e. Type II and 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/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: In the present system this is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below) and recommended for use in cases of dispute and for calibration purposes.

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 normally in an *ad hoc* endorsement Working Group which has been set up under the Committee's auspices at each of its Sessions. CCMAS has defined the terms to be used for each of the characteristics to be evaluated. These are given in Annex II. After generation of the criteria it will not be necessary to distinguish between Type II and III methods: all procedures conforming to the criteria will be equally valid. However, it will be necessary for a laboratory to demonstrate that whatever method it uses, its the application conforms to the laboratory quality standards as now adopted by the Codex Alimentarius Commission.

[Note: the dispute situation is to be resolved. Some countries would prefer a Type II method to be identified within the Codex system, others prefer a complete equivalence of methods on the assumption that the laboratory is "in control".]

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.

[Note: for completeness at this time the Checklist is given at Annex III, but will be removed from the final draft]

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 II 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. [Note: procedures for the development of In-House method validation procedures are currently being developed on an international basis.

When these are developed, then they could be considered by CCMAS as an appropriate quality standard for Type IV Methods of Analysis]

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 + 3s, where s 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 6? or 10? is required rather than 3? .

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 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 $RSD_R = 2^{(1-0.5\log C)}$

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.

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.

ANNEX II: EXAMPLES

The following examples are taken from CCMAS or EU documents.

GENERALISED CRITERIA FROM CCMAS FOR TRACE ELEMENTS:

Parameter	**Value/Comment**
Applicability	All foods
Detection limit	No more than one twentieth of the value of specification
Determination limit	No more than one tenth of the value of specification
Precision	$HORRAT_r$ and $HORRAT_R$ values of less than 2 in the validation collaborative trial
Recovery	80% - 105%
Specificity	No cross interferences permitted

i.e., if any method may be used provided in the validation work it meets the above criteria.

Similar criteria may be set up for each of the trace elements listed in Tables 1 and 2.

Possible method performance characteristics which may be included in an EU Commission Directive laying down the sampling methods and the methods of analysis for the official control of the levels for certain contaminants in foodstuffs:

Lead, Cadmium, Mercury and Arsenic:

Parameter	**Value/Comment**
Applicability	All foods
Detection limit	No more than one tenth of the value of specification
Determination limit	No more than one fifth of the value of specification
Precision	$HORRAT_r$ and $HORRAT_R$ values of less than 1.5 in the validation collaborative trial
Recovery	80% - 105% (as indicated in the collaborative trial)
Specificity	No cross interferences permitted

Notes:

The precision values are calculated from the Horwitz equation (Ref. 5), i.e.:

$$RSD_R = 2^{(1-0.5\log C)}$$

where:

RSD_R is the relative standard deviation calculated from results generated under reproducibility conditions [$(s_R / \bar{x}) \times 100$].

C is the concentration ratio (i.e. 1 = 100g/100g, 0.001 = 1000 mg/kg)

This is a generalised precision equation which has found to be independent of analyte and matrix but solely dependent on concentration for most “routine” methods of analysis.

ANNEX III: RECOMMENDATIONS FOR A CHECKLIST OF INFORMATION REQUIRED TO EVALUATE METHODS OF ANALYSIS SUBMITTED TO THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING FOR ENDORSEMENT

TYPE OF INFORMATION REQUIRED FOR SUBMISSION TO THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING FOR CONSIDERATION

It has been agreed that the following information should be supplied by Codex Committee Committees which seek endorsement of their methods by the Codex Committee on Methods of Analysis and Sampling. In practice this information is not forthcoming, and this leads to some difficulties to members of CCMAS.

1. REPORT FORMAT

1.1 IDENTIFICATION INFORMATION

1.1.1 **Responsible Codex Committee**

The Codex Committee requesting the endorsement for reference and referral.

1.1.2 **Codex Standard and Status**

A reference to the specific commodity item under consideration, its endorsement status, and a citation to its appearance in the Codex documentation.

1.1.3 **Analyte or Property**

The specific component, constituent, or property (**chemical, physical, microbiological**) which is to be measured and for which there exists a requirement for a limit or specification in the applicable standard.

1.1.4 **Codex Specification or Limit**

The specific specification, limit, tolerance, or guideline which is given in the standard and which provides the boundary between acceptable and unacceptable material.

1.1.5 **Method of Analysis**

(a) **Title and Principle** - A statement of the method of analysis which incorporates a summary of the principles of isolation and/or measurement.

(b) **Limit of Detection, Limit of Determination,** or **Applicable range** (as needed).

(c) **Classification (Type)** - The method classification as defined in the Codex Alimentarius Commission Procedural Manual, pp. 102-103 **as amended by the Commission in 1991 (Ref. ALINORM 91/21 or ALINORM 91/23, para. 43):**

Defining Methods (Type I)
Reference Methods (Type II)
Alternative Approved Methods (Type III)
Tentative Methods (Type IV)

(d) Reference to Source of Method of Analysis. Bibliographic citation to the scientific or technical publication, to the Codex document, **or to the internal reference number of the national or international organisation, as applicable.** The reference given should permit tracing back to original source documents discussing the application of the method to the analyte and commodity involved.

1.2 DESIGN, CONDUCT AND REPORTING OF RESULTS OF COLLABORATIVE STUDY SUPPORTING THE ENDORSEMENT OF THE METHOD

The design and conduct of the collaborative study of the method must follow the principles outlined in the 1987-IUPAC Harmonised Protocol for the Design, Conduct and Interpretation of Collaborative Studies (Ref: IUPAC (1988) Protocol for the Design, Conduct and Interpretation of Collaborative Studies, Pure and Appl. Chem. **60,** 855-864) (now up-dated revised in Protocol for the Design, Conduct and Interpretation of Method Performance Studies, ed. W. Horwitz, *Pure Appl. Chem.*, 1995, **67**, 331-343.)

1.2.1 **Bibliographic Reference to Collaborative Study**

Include citation to the published collaborative study as a literature reference, Codex document number, or to the national or international organization internal reference number, as applicable. Give sufficient documentation so that a librarian can obtain the referenced document directly from the journal, by interlibrary loan, or by request to the organization responsible for its production.

1.2.2 **Design**

State the number of materials, laboratories, determinations, replicates and tests used. If these vary from material to material, a separate line may have to be introduced in the table for the variable information.

1.2.3 **Material identification and composition**

Identify the materials in the column heads for a table of data, including information from 1.2.4 to 1.2.8.

1.2.4 **Outliers Removed**

Report number of laboratories remaining after removal of outliers and/or percent of outliers which had to be rejected in order to obtain the precision parameters reported in 1.2.8. If no outliers were rejected, report that fact as 0. Indicate identification number(s) of laboratories removed in order to note a consistent systematic bias on the part of any of them, if present.

1.2.5 **Concentration of Analyte or Value of a Property**

Report if known or assumed. If it is same throughout, it may be incorporated into the material identification, 1.2.3.

1.2.6 **Average Found and Units**

Give the average value found for each material, indicating the units in the row heading. If the number of replicates reported by each laboratory was not the same, use the average of each laboratory for averaging to avoid weighting of results.

1.2.7 **Recovery**

Report percent recovery, if amount of analyte present is known or assumed.

1.2.8 **Precision Parameters**

Report the following precision parameters

(a) Repeatability (within laboratory) standard deviation s_r in the same units as the average.
Repeatability relative standard deviation (s_r x 100/average).
Repeatability limit (2.8 x s_r).

(b) Reproducibility (among laboratories, including within -) standard deviation, s_R, in the same units as the average.
Reproducibility relative standard deviation (s_R x 100/average).
Reproducibility limit (2.8 x S_R).

Report precision parameters (1) with all valid results and (2) with results from which outliers have been removed. Do not include data from laboratories that did not follow the method, that reported problems with instruments or columns, whose system suitability parameters did not conform to specifications, or who experienced similar aberrant behaviour.

The standard deviations must be obtained material by material. The relative standard deviations are usually the most informative precision parameters in food analysis. It is important to recognise the relative standard deviation among laboratories is not obtained by calculating the standard deviation of all the data from a material (except when only single determinations are performed); it must be obtained by a "one-way analysis of variance", as demonstrated in the Steiner portion of the AOAC Statistical Manual, or ISO 5725 Section 7.2 (b).

2. NOTES

(Additional information, exceptions, and reasons for not following the recommendations.)

2.1 References to same method endorsed for other Codex Standards.

2.2 If a Codex method is available for this analyte or property for a different commodity and this method is not recommended for the commodity standard under consideration, give the reasons for not using the previously used method and for using a different method for this commodity or concentration level.

2.3 If a general Codex method is available for this analyte or property and it is not used in this standard, give the reasons for not using the general method.

2.4 Give reasons for any modifications of the previously used or endorsed method for other commodities or of the general method.

3. REFERENCES

Horwitz, W. (1988) Protocol for the Design, Conduct and Interpretation of Collaborative Studies, Pure and Appl. Chem. **60**, 855-864.

Youden W.J. & Steiner, E.H. (1975) Statistical Manual of the AOAC. AOAC International, Washington, D.C., U.S.A.

International Organization for Standardization (ISO). International Standard 5725:1986. Precision of Test Methods - Determination of Repeatability and Reproducibility for a Standard Test Method by Interlaboratory Tests. Available from ISO, Geneva, Switzerland and National Standardization Organizations. Currently being revised.