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Understanding Codex Specifications and Associated Methods of Analysis – Typing Codex Methods

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Introduction

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- **Codex Specifications**
- **Typing Methods According to Codex**
- **Methods Endorsement**
- **Listing Codex Methods**
- **Sources of Methods of Analysis**
- **Method Precision Data**
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- **Criteria Approach**
- **Proprietary Methods**
- **Primary vs Secondary Methods**





Codex Procedures Criteria for the Establishment of Work Priorities

Criteria applicable to commodities

- (a) **Volume of production** and consumption in individual countries and volume and **pattern of trade** between countries.
- (b) Diversity of national legislation and impediments to international trade.
- (c) International or regional market potential.
- (d) **Amenability of the commodity to standardisation.**
- (e) Coverage of consumer protection and trade issues by existing or proposed general standards.
- (f) Number of commodities which would need **separate standards** for raw, semi-processed or processed.
- (g) Work already undertaken by **other international organizations** in this field and/or suggested by the relevant **international intergovernmental body(ies).**



Codex Procedures

- Where commodity committees are of the opinion that the general provisions are not applicable to one or more commodity standards, they may request the responsible general subject committees to endorse deviations from the general provisions of the Codex Alimentarius.
- Such requests should be fully justified and supported by available scientific evidence and other relevant information. Sections on food additives, contaminants, hygiene, labeling, and methods of analysis and sampling which contain specific provisions or provisions supplementing the Codex General Standards, Codes or Guidelines shall be referred to the responsible general subject committees at the most suitable and earliest time in the Procedure for the Elaboration of Codex Standards and Related Texts, though such referral should not be allowed to delay the progress of the standard to the subsequent Steps of the Procedure.





Principles for the Establishment of Codex Methods of Analysis

Purpose of Codex Methods of Analysis


- The methods are primarily intended as international methods for the verification of provisions in Codex standards. They should be used for reference, in calibration of methods in use or introduced for routine examination and control purposes.



Methods of Analysis and Sampling Codex Guidance

- Principles for the Establishment of Codex Methods of Analysis. (Adopted in 1964. Amended in 1969, 1979, 2001, 2003, 2004, 2008)
- Principles for the Establishment or Selection of Codex Sampling Procedures. (Adopted in 1993. Amended 2007)
- The use of Analytical Results: Sampling Plans, Relationship between the Analytical Results, the measurement uncertainty, recovery factors and provisions in Codex Standards. (Adopted in 2006)







Codex Requirements for Specifications


Methods of Analysis and Sampling


- All methods of analysis and sampling considered necessary and should be prepared in accordance with the guidance given in the section on Methods of Analysis and Sampling in the *Relations between Commodity Committees and General Subject Committees*.
- If two or more methods have been proven to be equivalent by the Codex Committee on Methods of Analysis and Sampling, these could be regarded as alternatives and included either specifically or by reference.
- *"The methods of analysis and sampling are to be endorsed by the Codex Committee on Methods of Analysis and Sampling."*



Format for Codex Commodity Standards

- **Introduction**
- The Format is intended for use as a guide in the uniform presentation of commodity standards. The Format indicates the statements which should be included in standards under the relevant headings of the standard. The sections of the Format require to be completed in a standard only where appropriate.
- **Name of the Standard**
- **Scope**
- **Description**
- **Essential Composition and Quality Factors**
- **Food Additives**
- **Contaminants**
- **Hygiene**
- **Weights and Measures**
- **Labeling**
- **Methods of Analysis and Sampling**







Codex Specifications (1)

Methods of Analysis and Sampling: Normal Practice*

- When there are provisions on methods of analysis or sampling in a Codex commodity standard, these should be referred to the Committee on Methods of Analysis and Sampling at Step 4, to ensure Government comments at the earliest possible stage in the development of the standard.
- A commodity committee should provide information to the Committee on Methods of Analysis and Sampling for each individual analytical method proposed,
 - ❖ specificity,
 - ❖ accuracy,
 - ❖ precision (repeatability, reproducibility)
 - ❖ limit of detection, sensitivity,
 - ❖ applicability and practicability,


*Except for methods of analysis and sampling associated with microbiological criteria

Codex Specifications (2)

Methods of Analysis and Sampling: Normal Practice*

- A commodity committee should provide information to the Committee on Methods of Analysis and Sampling for each sampling plan relating to the scope or field of application,
 - the type of sampling (e.g. bulk or unit),
 - sample sizes,
 - decision rules,
 - details of plans (e.g. "Operating characteristic" curves),
 - inferences to be made to lots or processes,
 - levels of risk to be accepted
 - pertinent supportive data





Definition of Types of Codex Methods of Analysis (1)

(a) Defining Methods (Type I)

Definition: 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 measured.

Examples: Howard Mould Count, Reichert-Meissl value, loss on drying, salt in brine by density.

(b) Reference Methods (Type II)

Definition: A Type II method is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below). It should be recommended for use in cases of dispute and for calibration purposes.

Example: Potentiometric method for halides.



Definition of Types of Codex Methods of Analysis (2)

(c) Alternative Approved Methods (Type III)

Definition: A Type III Method is one 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.


Example: Volhard Method or Mohr Method for chlorides

(d) Tentative Method (Type IV)

Definition: A Type IV Method is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Codex Committee on Methods of Analysis and Sampling have not yet been determined.



Examples: chlorine by X-ray fluorescence, estimation of synthetic colours in foods.






Codex Procedural Rules

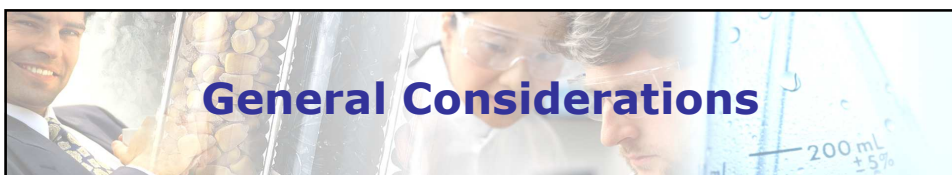
- At Step 4, commodity committees should discuss and report to the CCMAS on matters connected with:
 - Provisions in Codex standards which require analytical or statistical procedure;
 - Provisions for which elaboration of specific methods of analysis or sampling are required;
 - Provisions which are defined by the use of Defining Methods (Type I);
 - All proposals to the extent possible should be supported by appropriate documentation; especially for Tentative Methods (Type IV);
 - Any request for advice or assistance.
- CCMAS undertakes a coordinating role; the originating committee is responsible for carrying out the Procedure.
- When necessary, the CCMAS should ensure elaboration and collaborative testing of methods by **other recognized bodies with expertise in the field of analysis.**



Codex Procedural Rules (2)



- CCMAS assesses analytical performance of the method determined in its validation, taking into account the appropriate precision characteristics from collaborative trials and results from other development work carried out during method development.
- The set of criteria that are developed will form part of the report of the endorsement by 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.





General Considerations


- (a) CCMAS should maintain relations with **all interested organizations working on methods of analysis** and sampling.
- (b) CCMAS should keep under constant review all methods of analysis and sampling published in the Codex Alimentarius.
- (c) Codex methods of analysis should recognize variations in reagent concentrations and specifications from country to country.
- (d) Codex methods of analysis derived from scientific journals, theses, or publications, printed in the Codex Alimentarius *in extenso*, should follow the standard layout for methods of analysis as adopted by the CCMAS.
- (e) Methods of analysis printed as **official methods of analysis in other available publications** and which are adopted as Codex methods need only be quoted by reference in the Codex Alimentarius.



Methods Endorsement

General Criteria for the Selection of Methods of Analysis (1)

- (a) Official methods of analysis elaborated by international organizations occupying themselves with a food or group of foods should be preferred.
- (b) Preference should be given to methods of analysis the reliability of which have been established in respect of the following criteria, selected as appropriate:
 - (i) specificity
 - (ii) Accuracy
 - (iii) precision; repeatability intra-laboratory (within laboratory), reproducibility inter-laboratory (within laboratory and between laboratories)





General Criteria for the Selection of Methods of Analysis (2)

- (iv) limit of detection
 - (v) sensitivity
 - (vi) practicability and applicability under normal laboratory conditions
 - (vii) other criteria which may be selected as required.
- (c) The method selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use.
- (d) All proposed methods of analysis must have direct pertinence to the Codex Standard to which they are directed.
- (e) Methods of analysis which are applicable uniformly to various groups of commodities should be given preference over methods which apply only to individual commodities.




Criteria Approach

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

- In the case of Codex Type II and 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.







Listing Codex Methods


RECOMMENDED METHODS OF ANALYSIS AND SAMPLING
CODEX STAN 234-1999
PART A
METHODS OF ANALYSIS BY ALPHABETICAL ORDER OF COMMODITY CATEGORIES AND NAMES
PART B
METHODS OF SAMPLING BY ALPHABETICAL ORDER OF COMMODITY CATEGORIES AND NAMES

The most updated version of the method should be used, in application of ISO/IEC 17025: ~~1999~~ 2006. The present list of methods reflects the amendments adopted by the 30th Session of the Codex Alimentarius Commission in 2007.



Sources of Methods of Analysis

- Official Methods
- Reference/regulatory methods
- Fully validated methods of analysis
- Published methods and practices
- Journals, theses, application notes
- Single laboratory validated methods
- Proprietary methods (patents, “black box”, other limitations)





SDOs and IAM Members


- AACC International (AACC)
- Association of American Feed Control Officials (AAFCO)
- AOAC International (AOAC)
- American Oil Chemists' Society (AOCS)
- EURACHEM
- European Committee for Standardization (CEN)
- International Association for Cereal Sciences & Technology (ICC)
- International Commission for Uniform Methods of Sugar Analysis (ICUMSA)
- International Dairy Federation (IDF)
- International Federation of Fruit Juice Producers (IFU)
- International Federation of Glucose Industries (IFG)
- International Office of Cocoa, Chocolate and Sugar Confectionery (IOCCC)
- International Organization for Standardization (ISO)
- International Union of Microbiological Societies (IUMS)
- International Union of Pure & Applied Chemistry (IUPAC)
- International Wine and Vine Office (OIV)
- Nordic Committee on Food Analysis (NMKL)




Consensus Process for Standard Development (ISO, SDOs)



- **Consensus**
The views of all interests are taken into account: manufacturers, vendors and users, consumer groups, testing laboratories, governments, professionals and research organizations.
- **Industry-wide**
Global solutions to satisfy industries and customers worldwide.
- **Voluntary**
International standardization is market-driven and therefore based on voluntary involvement of all interest.





Collaborative Study Protocols

- The AOAC/IUPAC Harmonized Protocol
- ISO 5725 Accuracy (trueness and precision) of measurement methods and results –
 - Part 1: General principles and definitions
 - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method
 - Part 3: Intermediate measures of the precision of a standard measurement method
 - Part 4: Basic methods for the determination of the trueness of a standard measurement method
 - Part 5: Alternative methods for the determination of the precision of a standard measurement method




Requirements


New methods are collaboratively tested internationally to conform to ISO 5725 or AOAC/IUPAC Harmonized Protocol

Minimum results from:

- 8 laboratories
- 5 countries
- 5 levels of analyte

- Need 15 labs globally located/who are they?
- Need to ensure that the method is clearly understood
- Need all labs to follow the method as written
- Need all labs to return results in a timely manner







Collaborative Data Treatment

Data treatment recommendations

- Outlier testing
 - Cochran (for excess variance)
 - Grubbs tests (for extreme mean values and pairs of means)
- Outlier action
 - IUPAC: Remove at 97.5% confidence
 - ISO 5725: Inspect at 95%; Remove at 99%
- Repeated outlier tests
 - Permitted to maximum of 22.2% data set loss (IUPAC)






CT Data Processing

mean

repeatability (between duplicates/within lab)
 standard deviation of repeatability (sr)
 repeatability value (r) (2.8 sr)
 Relative standard deviation (RSDr)
 Horrat value (H0r)

Reproducibility (between lab)
 Reproducibility (between duplicates/within lab)
 standard deviation of Reproducibility (sR)
 Reproducibility value (R) (2.8 sR)
 Relative standard deviation (RSDR)
 Horrat value (HOR)
 Horwitz Ratio



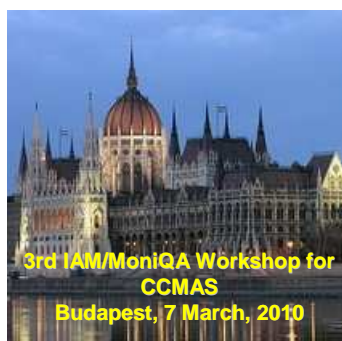


Challenges for SDOs

- Knowledge of citation in Codex Alimentarius
- Assessment of method suitability and equivalence
- Vigilance in updating Codex 234
- Participation in relevant Commodity Committees
- Active participation in the InterAgency Meeting
- Proprietary methods
- Primary vs Secondary technologies



Thank you, any questions?



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