

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
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World Health
Organization

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Agenda Item 5

MAS/40 CRD/03

**ORIGINAL LANGUAGE ONLY JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
40th Session**

Budapest, Hungary, 27 - 31 May 2019

REVISION OF THE RECOMMENDED METHODS OF CXS 234 –PREAMBLE AND STRUCTURE

**Report of the Physical Working Group (pWG) on
Proposed Preamble and Document Structure for General Standard on Methods of Analysis and
Sampling (CXS 234-1999)**

The pWG, chaired by Brazil and Uruguay, met on Sunday, 26 May 2019, to further discuss the Proposed Preamble and Document Structure for General Standard on Methods of Analysis and Sampling (CXS 234-1999) considering recommendations and comments received on eWG.

The Chair introduced the topic and highlighted key aspects of standard that pWG should focus on referring to the comments presented in CX/MAS 19/40/5-Add. 1, CRD 9 and CRD 13. The proposed changes to text and structure and recommendations made by the pWG are presented below.

The revised version with the amendments agreed by the pWG is presented in Appendix I.

INTRODUCTION

Replace the term 'CXStdards' by 'Codex Standards'

The pWG agreed to replace 'CXStdards' by 'Codex Standards' throughout the text.

Paragraphs 1, 2 and 5

Observing that paragraphs 1, 2 and 5 of Introduction present somewhat different versions of purpose of Codex methods of analysis and sampling, the pWG agreed with a new paragraph to replace paragraphs 1, 2 and 5.

Footnote 1

The pWG agreed with a new version of footnote 1 as suggested.

Define what is the best place for the statement "All Codex Methods including Type IV can be used for control, inspection and regulation and when parties agree for resolution of disputes"

The pWG agreed that the statement should be retained under Part II of the document, as it is the specific section for methods of analysis.

Scope

There was a suggestion to include a paragraph regarding the Committees' methods of analysis not included on current scope of this Standard. The Codex Secretariat noted that the suggestion is in line with the Codex Procedural Manual. Thus, the pWG agreed to include a new paragraph.

Definition of Terms

2.1. Codex Methods of Analysis

The pWG agreed not to include the definitions of the types of methods of analysis under section 2.1 since it is already contained in the Codex Procedural Manual. It was agreed that it would not be advisable to repeat the definitions in CXS 234 in order to avoid inconsistencies between documents. For electronic version, additional clarification would be provided by hyperlink.

2.3. Provision:

The pWG agreed to replace “analytical parameter” by “attribute”, since “attribute” is a broad term. It was also pointed out the redundancy between the terms “analytical parameter” and “analysis” within the text.

2.4. Method criteria:

The pWG agreed to replace “method criteria” by “method performance criteria” since the term “method criteria” is not referred throughout the text. There was also an agreement on deleting “criterion or characteristic” from the text considering that the term “provision” is already defined in the document.

PART II. METHODS OF ANALYSIS

The pWG agreed on deleting the text strikethrough of the following paragraph as it is already in Codex Procedural Manual:

~~All Codex methods, including Type IV methods, can be used for control, inspection and regulation and when parties so agree, for resolution of disputes. A Type I method 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 measurand. A Type II method is the one designated Reference Method where Type I methods do not apply. A Type III Method is one which meets the criteria required by the Committee on Methods of Analysis and Sampling and a Type IV is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Committee on Methods of Analysis and Sampling have not yet been determined.~~

Regarding other proposals received for the text of Part II, the Chair pointed out that further definitions it would dependent of ongoing work under Agenda item 4 (Guidance on endorsement). Thus, it would be advisable to await the work's conclusions.

Structure

It was discussed a condensed version for the structure of the document. The Chair observed that the structure proposed was similar to that one presented during the 39th CCMAS. There was support from the pWG for returning to this proposal with some amendments. All methods would be placed in a single table. The column “Type” should be renamed for “Criteria/Type”. The methods would be presented on this table as followed: a) for methods with full description on Codex Standard 234, it would be assigned the letter “M” on “Method Column” and “Criteria/Type” column should be kept in blank. b) for methods with performance criterion would be assigned the letter “C” on “Criteria/Type” column, and “Method” column would be kept in blank. An example of new structure for Table is shown on revised version of document.

It was also agreed to include detection principle for methods involving separation techniques on method description column. In order to simplify description of methods principle on methods table it should be included on Codex Standard 234 a list of acronyms as an appendix. For electronic database the acronyms definition would be directed access by hyperlink and for printed pdf version, the list would be automatically generated as demanded.

There was also a proposal to delete the list of methods (currently contained in Part II section IV of the proposed draft revision) and migrate it to an Excel file that would be available for consulting on Codex Website. The Codex Secretariat mentioned that they are working with the Hungarian Codex Secretariat aiming to update the list in order to make it available as a searchable tool.

It was also highlighted that a database does not preclude the need of a printed version.

Appendix I

PROPOSED DRAFT PREAMBLE AND DOCUMENT STRUCTURE FOR THE GENERAL STANDARD ON METHODS OF ANALYSIS AND SAMPLING (CXs 234-1999)

INTRODUCTION

1. This Standard contains definitions, lists of methods of analysis, method performance criteria, descriptions of some methods and a list of methods of sampling. The methods of analysis and sampling contained in this Standard are the recommended ones to be used to assess compliance for a specific provision described in Codex standards and can be used for reference, in calibration of methods in use or introduced for routine examination and control purposes.
2. It is recommended that this Standard should be read in conjunction with the related Codex Standards, guidelines and other documents¹.
3. In case of disputes of analytical results, guidance is given in the *Guidelines for Settling Disputes over Analytical (Test) Results* (CXG 70-2009), including guidance on the use of methods of analysis.

¹ Harmonized IUPAC Guidelines for the Use of Recovery Information in Analytical Measurement (CXG 37-2001), Harmonized IUPAC Guidelines for Single-Laboratory Validation of Methods of Analysis (CXG 49-2003), Guidelines on Sampling (CXG 50-2004), Guidelines on Measurement Uncertainty (CXG 54-2004), Harmonized Guidelines for Internal Quality Control in Analytical Chemistry Laboratories (CXG 65-1997), protocols for method performance determination through collaborative study. (CXG 64-1995 and ISO 5725), Food Control Laboratory Management: Recommendations (CXG 28-1995).

PART I. PREAMBLE

1. Scope

This Standard is intended to provide a single reference to methods of analysis and sampling for food as adopted by the Codex Alimentarius Commission.

This Standard is not applied to methods of analysis and sampling for residues of pesticides or veterinary drugs in food, the assessment of micro biological quality and safety in food, and the assessment of specifications for food additives.

2. Definition of Terms

2.1 Codex Methods of Analysis: methods for the verification of provisions in Codex Standards. The methods are classified as Defining Methods (Type I), Reference Methods (Type II), Alternative Approved Methods (Type III), & Tentative Methods (Type IV) (see Codex Procedural Manual, Section II: Elaboration of Codex texts, Definition of types of methods of analysis).

2.2 Methods of Analysis Principle: The science-based analytical principle of the method of analysis, described concisely, focusing on the technique.

2.3 Provision: Attribute of a commodity that needs to be confirmed by analysis to ensure that it conforms to that standard.

2.4 Method performance criteria: Set of performance characteristics to which a method used must comply when determining a provision.

PART II. METHODS OF ANALYSIS

The part II contains 3 sections.

Section I

This section presents all the methods by commodities and provisions.

Method listed in this section could be used for any purpose in line with the principle of this standard and Codex Procedural Manual. They could be used for national regulation, control and inspection. Method Type III and IV can be used in case of disputes, if it was agreed between the respective competent authorities, according to the Guidelines for Settling Disputes on Analytical (Test) Results (CAC/GL 70-2009). Methods Type III and IV can also be used in cases of disputes, if it was agreed between parties.

The most updated version of the method should be used in application of ISO/IEC 17025 unless it is not appropriate or possible to do so.

[Each line of the methods list corresponds to one method of analysis or more than one when multiple methods are needed to reach a result. In case of multiple methods use, they are called complementary being presented on table with an "and" between them. When a provision is determined by calculation, a brief description of the calculation shall be given in the principle column.

When the methods are in the same row separated by a vertical bar "|", they are identical and published in a single document by different standards development organizations. When methods are separated by a forward slash "/", technical procedures are identical but published in separate documents which may have different editorial formats. Separate lines, under commodity column are used when two or more methods are required to cover the full range of values.]

When there is the letter "M" on "Method" Column, a full description of method is provided in Section III. When there is the letter "C" on column "Criteria/Type", a method performance criterion is provided in Section II.

Section II

This section presents method performance criteria.

Section III

This section presents complete descriptions of methods of analysis

SECTION I – METHODS OF ANALYSIS AND METHOD PERFORMANCE CRITERIA BY COMMODITY

This section contains:

- a) Commodity;
- b) Provision;
- c) Method;
- d) Principle;
- e) Codex Stan;
- f) Criteria/Type.

SECTION II – PROVISIONS FOR WHICH THERE ARE METHOD PERFORMANCE CRITERIA

This section contains:

- a) Commodity;
- b) Provision;
- c) Maximum level (ML)
- d) Minimum applicable range;
- e) Limit of detection (LOD);
- f) Limit of quantification (LOQ);
- g) RSD_R (Relative Standard Deviation of Reproducibility);
- h) % Recovery;
- i) Examples of Methods that meet the criteria and their principles also can be mentioned. However, any method that complies with the established performance criteria can be used;
- j) Principle.

SECTION III – COMPLETE DESCRIPTION OF THE METHODS OF ANALYSIS

This section contains:

- a) Description and scope of the method that includes the commodity and provision.

PART III. METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

This part contains:

- a) The name of the commodity/product;
- b) Identification of method of sampling;
- c) Notes.

ANNEX 1. NOTES TO THE STANDARD FOR METHODS OF ANALYSIS AND SAMPLING**ANNEX 2. ACRONYMS LIST FOR METHOD PRINCIPLE**

SECTION I - METHODS OF ANALYSIS BY COMMODITY CATEGORIES

<i>Commodity</i>	<i>Provision</i>	<i>Method</i>	<i>Principle</i>	<i>Codex Standard</i>	<i>Criteria/Type</i>
Food grade salt	Cadmium	EuSalt/AS 014	AAS	150-85	IV
Food grade salt	Calcium and magnesium	ISO 2482	Titrimetry	150-85	II
Food grade salt	Calcium and magnesium	EuSalt/AS 009	FAAS	150-85	III
Food grade salt	Calcium and magnesium	EuSalt/AS 015	ICP OES	150-85	III
Food grade salt	Copper	EuSalt/AS 015	ICP OES	150-85	III
Fish and fishery products	Histamine			36-1981	C
Canned Applesauce	Fill of containers	M	Volumetry	17-1981	

PART II. METHODS OF ANALYSIS**SECTION II - PROVISIONS FOR WHICH THERE ARE METHOD PERFORMANCE CRITERIA**

The following are minimum method performance criteria which an analytical method validation need to have achieved to be considered suitable.

Commodity	Provision	ML	Minimum applicable range	LOD	LOQ	RSDR (%)	Recovery (%)	Examples of methods that meet the criteria	Principle
Fish and fishery products	Histamine	10 mg/100 g (average)	8 – 12 mg/100 g	1 mg/100 g	2 mg/100 g	16.0	90 – 107	AOAC 977.13 NMKL 99, NMKL 196,	HPLC-FL
Fish and fishery products	Histamine	20 mg/100 g (each unit)	16 – 24 mg/100 g	2 mg/100 g	4 mg/100 g	14.4	90 – 107	AOAC 977.13 NMKL 99, NMKL 196,	HPLC-FL

PART II. METHODS OF ANALYSIS

SECTION III - COMPLETE DESCRIPTION OF THE METHODS OF ANALYSIS

DETERMINATION OF WATER CAPACITY OF CONTAINERS (CAC/RM 46)

1. SCOPE

This method applies to glass containers.

2. DEFINITION

The water capacity of a container is the volume of distilled water at 20°C which the sealed container will hold when completely filled.

3. PROCEDURE

3.1 Select a container which is undamaged in all respects.

3.2 Wash, dry and weigh the empty container.

3.3 Fill the container with distilled water at 20°C to the level of the top thereof, and weigh the container thus filled.

4. CALCULATION AND EXPRESSION OF RESULTS

Subtract the weight found in 3.2 from the weight found in 3.3. The difference shall be considered to be the weight of water required to fill the container. Results are expressed as mL of water.

PART III. METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

Commodity Categories	Method of Sampling	Notes
Cereals, Pulses and Legumes and Derived Products		
Wheat protein products including wheat gluten	ISO 13690	
Fats and Oils		
Olive Oils and Olive-Pomace Oils	ISO 661 and ISO 5555.	
Fish oils	ISO 5555	
Milk and Milk Products		
Milk products	ISO 707 IDF 50	General instructions for obtaining a sample from a bulk
Milk products	ISO 5538 IDF 113	Inspection by attributes
Milk products	ISO 3951-1	Inspection by variables
Processed Fruits and Vegetables		
Desiccated coconut	Described in the Standard	
Certain canned vegetables, jams and jellies	Described in the Standard	
Chili sauce	Described in the Standard	
Table Olives	Described in the Standard	

ANNEX1. NOTES TO THE STANDARD FOR METHODS OF ANALYSIS AND SAMPLING**ANNEX 2. ACRONYMS LIST FOR METHOD PRINCIPLE****[to be further developed]**

Acronyms	Method Principle Description
HPLC	High Performance Liquid Chromatography
ICP OES	Inductively Coupled Plasma with Optical Emission Spectrometry
FAAS	Flame Atomic Emission Spectrometry