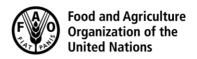
## CODEX ALIMENTARIUS COMMISSION





Viale delle Terme di Caracalla, 00153 Rome, Italy - Tel: (+39) 06 57051 - E-mail: codex@fao.org - www.codexalimentarius.org

Agenda Item 4

MAS/39 CRD/18 ORIGINAL LANGUAGE ONLY

## JOINT FAO/WHO FOOD STANDARDS PROGRAMME

#### CODEX COMMITTEE ON METHODS OF ANALYSIS SAMPLING

Thirty-ninth Session
Budapest, Hungary, 7 – 11 May 2018

Report of the in-session WG on

Proposed Preamble and Document Structure for General Standard on Methods of Analysis and Sampling (CXS 234-1999)

The in-session WG met on Tuesday, 8 May 2018. The In-session WG considered comments submitted on the preamble and structure of the CXS 234.

The in-session WG had the following discussion and made recommendations.

#### INTRODUCTION

This Standard contains definitions, lists of methods of analysis, methods performance criteria, descriptions of some methods and a list of methods of sampling to verify the provisions in Codex standards to be applied to commodities moving in international trade.

The methods are primarily intended to allow competent national and/or regional authorities to select appropriate methods of analysis and sampling for the verification of provisions' commodities found in Codex standards.

It is recommended that this Standard should be read in conjunction with the related Codex standards, quidelines and other documents<sup>1</sup>.

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.

When confirming compliance to a Codex standard the methods of analysis and sampling contained in this General Standard that relates to the provision identified in the commodity standard should be used.

<sup>&</sup>lt;sup>1</sup> 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 (CAC/GL 54-2004), Protocol for the Design, Conduct and Interpretation of Method Performance Studies (CXG 64-1995), Harmonized Guidelines for Internal Quality Control in Analytical Chemistry Laboratories (CXG 65-1997)

This Standard consists of three main parts and three annexes:

PART I. PREAMBLE

PART II. METHODS OF ANALYSIS

SECTION I - PROVISIONS FOR WHICH THERE ARE METHODS PERFORMANCE CRITERIA

SECTION II - METHODS OF ANALYSIS BY COMMODITY CATEGORIES

SECTION III. COMPLETE DESCRIPTION OF THE METHOD OF ANALYSIS

SECTION IV.

PART III. METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

Annex 1: LIST OF COMMODITIES CATEGORIES AND NAMES

Annex 2: LIST OF PROVISIONS

Annex 3: LIST OF THE PRINCIPLES OF THE METHODS

#### **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.

#### 2. Definition of Terms

- **2.1 Codex Method 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 Method of Analysis Principle:** The science-based analytical principle of the method of analysis, described concisely, focusing on the technique.
- **2.3 Provision:** A criterion of a commodity that needs to be confirmed by analysis to ensure that it conforms to that standard.
- **2.4 Methods criteria:** Set of performance characteristics to which a method used for the determination of a criterion or characteristic must comply.

#### **PART II - METHODS OF ANALYSIS**

This part contains 4 sections, the first one list all the commodities and provisions including a link to the other sections, depending on how the methodologies are proposed, endorsed and approved by CAC:

#### SECTION I. METHODS OF ANALYSIS BY COMMODITY CATEGORIES

This section contains:

- a) The name of the commodity/product;
- b) The provision to which the methods apply;
- c) Codex Standard to which the method is directed;
- d) Link to the performance criteria or method

#### **SECTION II. METHODS PERFORMANCE CRITERIA**

This section contains:

- a) The name of the commodity/product;
- b) The provision to which the methods apply;
- c) Codex Standard to which the method is directed;
- d) Minimum applicable range;
- e) Limit of detection (LOD);
- f) Limit of quantification (LOQ);

- g) RSDR (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.

#### SECTION III. COMPLETE DESCRIPTION OF THE METHOD OF ANALYSIS

This section contains:

- a) The name of the commodity (to choose from Annex 1);
- b) The provision to which the methods apply;
- c) Description.

#### SECTION IV. STANDARDIZED METHODS OF ANALYSIS BY COMMODITY CATEGORIES

This section contains:

- a) The name of the commodity/product;
- b) The provision to which the methods apply;
- c) Identification of the method;
- d) Method of Analysis Principle (to choose from Annex 3);
- e) Type of analytical method;

#### **PART II - METHODS OF ANALYSIS**

The selected methods for analysis shall full fill the performance criteria required by the Codex Alimentarius Commissions to ensure fit for purpose for provision quality and/or identity assessment.

The section I presents all the methods by commodities and provisions.

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 if they are necessary to reach a result, in this case they are called complementary 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 "/", the technical procedures are identical and published in separate documents that may have different formats.

All Codex methods, including Type IV methods, could be used for control, inspection and regulation and when parties so agreed, 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 item measured. 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.

Observation: The examples below will be deleted in the agreed document.

#### SECTION I - METHODS OF ANALYSIS BY COMMODITY CATEGORIES

Commodity	Provision	Codex Stan	Method
Processed fruits and vegetables	Benzoic acid	CXS 13	See Section IV (link to section IV)
Processed fruits and vegetables	Fill of containers (metals containers)	CXS 13	CAC/RM 46 ( link to complete description on Section III)
Natural Mineral Waters	Mercury	CXS 108	See Section II (link to section II)

### **SECTION II - METHODS PERFORMANCE CRITERIA**

Commodity	Provision	Applicable Codex Stan	Minimum applicable range	LOD	LOQ	RSD <sub>R</sub> (%)	Recovery (%)	Examples of methods that meet the criteria	Principle
Natural Mineral Waters	Mercury	108-1981	0.00056 mg/L	0.0002 mg/L	0.0004 mg/L	44	80-110	EN 1483 ISO 17852 ISO 5666 ISO 16590 EPA 200.8	AAS Enrichment by amalgamation (III) AFS AAS after tin(II) chloride reduction Enrichment by amalgamation (III) ICP-MS

#### SECTION III. COMPLETE DESCRIPTION OF THE METHOD OF ANALYSIS

Commodity	Provision
Processed fruits and	Fill of (metals) container
vegetables	

# DESCRIPTION OF THE METHOD: 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.

#### SECTION IV - STANDARDIZED METHODS OF ANALYSIS BY COMMODITY CATEGORIES

Commodity	Provision	Method	Principle	Туре
Processed fruits	Benzoic acid	NMKL 124	Liquid	II
and vegetables			Chromatography	

#### PART III- RECOMMENDED METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

Commodity Categories	Method of Sampling	Notes
Cereals, Pulses and Legur	mes 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 Vege	etables	
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	