

codex alimentarius commission



FOOD AND AGRICULTURE
ORGANIZATION
OF THE UNITED NATIONS

WORLD
HEALTH
ORGANIZATION



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Agenda Item 6

CX/MAS 02/7-Add.2

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Twenty-fourth Session

Budapest, Hungary, 18-22 November 2002

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS

This document contains the Methods of analysis proposed by the following Committees in Draft Standards and Proposed Draft Standards under elaboration; as general methods for groups of commodities; or as a revision of existing methods included in adopted standards.

- A. Codex Committee on Food Additives and Contaminants (35th Session, March 2002, ALINORM 03/12, Appendix XVII)
- B. Codex Committee on Processed Fruits and Vegetables (21st Session, September 2002, ALINORM 03/27, Appendix VI)

A. COMMITTEE ON FOOD ADDITIVES AND CONTAMINANTS

1) Food Additives

- Sulphites and Intense Sweeteners (At Step 6 in the General Standard for Food Additives) (CL 2002/44-FAC)
- Nitrites in meat products (Provisions in Codex Standards for processed meat products)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note - Type proposed
All foods	Sulphites	EN 1988-1 : 1998-02	Part 1: Optimized Monier-Williams method	Type III
All foods	Sulphites	EN 1988-2 : 1998-02	Part 2: Enzymatic method	Type III
Table top sweeteners	Saccharin	EN 1376 : 1996-09 (confirmed 2001)	Spectrometric method	Type III
Table top sweeteners	Acesulfame K	EN 1377 : 1996-09 (confirmed 2001)	Spectrometric method	Type II
Table top sweeteners	Aspartame	EN 1378 : 1996-09 (confirmed 2001)	High performance liquid chromatography	Type II
Liquid table top sweeteners preparations	Cyclamate and Saccharin	EN 1379 : 1996-09 (confirmed 2001)	High performance liquid chromatography	Type II
All foods	Acesulfame K, Aspartame and Saccharin	EN 12856 : 1999-04	High performance liquid chromatography	Type II
All Foods	Cyclamate	EN 12857 : 1999-04	High performance liquid chromatography	Type II
All foods	Nitrates and/or Nitrites	EN 12014-1:1997-04 Part 1- General considerations		Type III
Meat Products	Nitrates and/or Nitrites	ENV 12014-3:1998-06 Part 3	Spectrometric determination of nitrate and nitrite content of meat products after enzymatic reduction of nitrate to nitrite	Type III ¹
Meat Products	Nitrates and/or Nitrites	ENV 12014-3:1998-06 Part 4	Ion-exchange chromatographic (IC) method	Type III

¹ Current methods for nitrites are AOAC 973.31 as Type II and ISO 2918.1975 as Type IV

2) Contaminants

Draft Maximum Level for Ochratoxin A: 5µg/kg in raw wheat, barley and rye and derived products at Step 8 (ALINORM 03/12, Appendix IX)

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE
Cereal and Cereal Products	Ochratoxin A	EN ISO 15141-1:1998-10 Part 1	High performance liquid chromatographic method with silica gel clean up (ISO 15141-1:1998)	Type II
		EN ISO 15141-2:1998-10 Part 2	High performance liquid chromatographic method with bicarbonate clean up (ISO 15141-2:1998)	Type III

3) Other Methods

The following methods have also been proposed for contaminants. They do not correspond to maximum levels under consideration.

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note: Type proposed
Foodstuffs	Nitrates/Nitrites	EN 12014-1:1997-04 Part 1: General considerations		Type III
Vegetables and vegetable products	Nitrates/Nitrites	EN 12014-2:1997-04 Part 2	HPLC/IC	Type II
		EN 12014-5:1997-04 Part 5	Enzymatic determination of nitrate content of vegetable-containing food for babies and infants	Type II
		EN 12014-7:1998-06 Part 7	Continuous flow method for the determination of nitrate content of vegetables and vegetable products after cadmium reduction	Type III
Fatty food	Pesticides and PCBs	EN 1528-1: 1996-10 (confirmed 2001) Part 1: General considerations		Type III
		EN 1528-2: 1996-10 Part 2:	Extraction of fat, pesticides and PCBs and determination of fat content	
		EN 1528-3: 1996-10 Part 3	Clean-up methods	
		EN 1528-4: 1996-10 Part 4:	Determination, confirmatory tests, miscellaneous	
Cereals, shell-fruits and derived products	aflatoxin B ₁ , and sum of aflatoxins B ₁ , B ₂ , G ₁ and G ₂	EN 12955 : 1999-07	HPLC with post column derivatization and immunoaffinity column clean up	Type III
Maize	fumonisin B ₁ and B ₂	EN 13595 : 2001 - 11	HPLC with solid phase extraction clean-up	Type I

B. CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

1) General Methods of Analysis for Processed Fruits and Vegetables

STANDARD	PROVISION	METHOD	PRINCIPLE	NOTE - Type proposed
Processed fruits and vegetables	Calcium	AOAC 968.31	Complexometric titrimetry	Replaces CAC/RM 38-1970
Processed fruits and vegetables (except pickled cucumbers)	Fill of containers	CAC/RM 46-1972	Weighing	Retain - Type I Delete references to “metal containers”.and refer to ISO 90.1:1986 for determination of water capacity in metal containers
Processed fruits and vegetables	Packing medium ≥ 10°Brix Canned berry fruits (raspberry, strawberry)	AOAC 932.12 ISO 2173:1978	Refractometry	Type I
Processed fruits and vegetables	Sodium chloride	ISO 3634:1979	Potentiometry	Type II - In addition to the Codex General Method AOAC 971.27
Processed fruits and vegetables	Determination of Drained Weight - Method I	AOAC 968.30		Replaces CAC/RM 36-1970 ²
Processed fruits and vegetables	pH	AOAC 981.12 ISO 11289:1993	Potentiometry	

² The following changes are proposed to the AOAC method:

- Revise Section 2.1 Specifications for Circular Sieves to read: If total quantity of contents is less than ~~1.5 kg. (3 lbs)~~ 1 kg. (2 lbs) use a sieve.
- Revise second sentence of Section 3. Procedure to read: Without shifting the contents, ~~so~~ incline the sieve *approximately 20° from the horizontal* to facilitate drainage
- Insert new sentence at the end of the paragraph: *“This determination should be performed at 20°C ±5°C.”*

The instructions omit two important steps: (1) the weighing of the full container; and (2) the weighing of the dry empty container. Both weights are required to calculate the percentage drained weight (solid content) and/or the percent liquid.

STANDARD	PROVISION	METHOD	PRINCIPLE	NOTE - Type proposed
Processed fruits and vegetables	Sulphites	AOAC 990.28 ISO 522:1981		No current provisions
Processed fruits and vegetables	Total solids	AOAC 920.151	Gravimetry	

2) Methods of Analysis included in Draft Standards

- Draft Standard for Aqueous Coconut Products - Coconut Milk and Coconut Cream (Step 8)
- Draft Standard for Canned Stone Fruits (Step 8)
- Draft Standard for Pickled Products (Step 6)

STANDARD	PROVISION	METHOD	PRINCIPLE	NOTE
Aqueous Coconut Products	Moisture	Subtracting total solids from 100		
Aqueous Coconut Products	Non-fat solids	Subtracting total fats from total solids		
Aqueous Coconut Products	Total fats	AOAC 989.05 ³	Ether extraction	
Aqueous Coconut Products	Total solids	AOAC 990.20 ⁴	Oven extraction	
Canned Stone Fruits	Drained weight	AOAC 968.30	Gravimetry	General method for PFFV
Canned Stone Fruits	Soluble solids	AOAC 932.14C	Refractometry	General method for PFFV
Pickles	Benzoic acid < 250 mg/kg	AOAC 983.16 ⁵	Liquid Chromatography	

³ New method. This method replaces AOAC 945.48G which had not been endorsed by CCMAS due to lack of information on validation of the cited method for this application (23rd CCMAS, Budapest, Hungary, 26 February - 2 March 2001, ALINORM 01/23 App. IV-Part I/E). The new method is an IDF-ISO-AOAC method.

⁴ New method. This method replaces AOAC 925.23A (repealed) which had not been endorsed by CCMAS due to lack of information on validation of the cited method for this application (23rd CCMAS, Budapest, Hungary, 26 February - 2 March 2001, ALINORM 01/23 App. IV-Part I/E).

⁵ ISO 5518:1978 repealed in view of the recommendation of CCMAS to review more modern methods such as the AOAC 983.16 which had been endorsed as a Type II Codex General Method (22nd CCMAS, Budapest, Hungary, 23-27 November 1998, ALINORM 99/23, App. III-Part 1/B).

STANDARD	PROVISION	METHOD	PRINCIPLE	NOTE
Pickles	Lead ≤ 1.0 mg/kg	ISO 6633:1984	Flameless atomic absorption spectrophotometry	TE as Type IV (1998)
Pickles	Sorbate < 1000 mg/kg	AOAC 983.16 ⁶	Liquid Chromatography	
Pickles	Sulphur Dioxide <30 mg/kg	AOAC 990.28 ⁷	Titrimetry	
Pickles	Tin ≤ 250.0 mg/kg	ISO 2447:1974		TE as Type IV (1998)

In addition, in the **Draft Standard for Pickles**, the Committee proposed methods for the determination of

- acidity
- salt
- drained weight

that are not specified in the standard. The CCMAS may consider recommending to the CCPFFV to delete these methods as they do not correspond to a specific provision.

⁶ ISO 5519:1978 repealed in view of the recommendation of CCMAS to review more modern methods such as the AOAC 983.16 which had been endorsed as a Type II Codex General Method (22nd CCMAS, Budapest, Hungary, 23-27 November 1998, ALINORM 99/23, App. III-Part 1/B).

⁷ ISO 5522:1981 & ISO 5523:1981 repealed in view of the recommendation of CCMAS to review the Optimized Monier-Williams method (AOAC 990.28), which had been endorsed as a Type II Codex General Method (22nd CCMAS, Budapest, Hungary, 23-27 November 1998, ALINORM 99/23, App. III-Part I/B).