



Agenda Item 4

CX/MAS 12/33/4

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Thirty-third Session

Budapest, Hungary, 5 - 9 March 2012

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS

This document contains the methods of analysis and/or sampling proposed by the following Committees in draft standards and related texts under elaboration or as update of current methods:

- PART I Methods of analysis
 - A. Committee on Fish and Fishery Products
 - B. Committee on Food Additives
 - C. Committee on Processed Fruits and Vegetables
 - D. FAO/WHO Coordinating Committee for the Near East
- PART II Methods of sampling
 - A. Committee on Fish and Fishery Products
 - B. Committee on Processed Fruits and Vegetables
 - C. FAO/WHO Coordinating Committee for the Near East

PART I METHODS OF ANALYSIS

A. COMMITTEE ON FISH AND FISHERY PRODUCTS (CCFFP)

Standard for Fish Sauce¹

The 31st Session of CCFFP agreed that a reference to AOAC 981.12 (general Codex method for the determination of pH) was inserted and that the explanation concerning the need to dilute fish sauce with water 1:10 prior to analysis was retained.

The Committee agreed to advance the Draft Standard for Fish Sauce to Step 8 for adoption by the 34th Session of the Codex Alimentarius Commission. The Committee noted that the provisions for methods of analysis and sampling would be forwarded to the relevant committees for endorsement.

The 34th Session of the Commission adopted the Standard as proposed at Step 8.

See **Table section A** for the complete list of the proposed methods of analysis.

¹ REP11/FFP, paras 34 – 37 and Appendix III

B. COMMITTEE ON FOOD ADDITIVES (CCFA)

Draft Revision of the Standard for Food Grade Salt²

The 43rd Session of CCFA noted that the e-WG had updated references to the analytical methods to refer to valid methods currently available from international organisations and added several EuSalt-methods, used in many laboratories, and the iodine titration method, included in the relevant WHO guidance document.

As there was concern that some laboratories might not be adequately equipped to use these newly proposed methods it was proposed that the “criteria-based approach”, recommended by the Committee on Methods of Analysis and Sampling (CCMAS), should be the preferred approach for selecting different analytical methods, in particular for heavy metals and copper. It was also suggested keeping the “methods listing approach” i.e. to list all available and acceptable analytical methods for the other elements, namely sulfate, halogens, calcium and magnesium, potassium and iodine.

The Committee agreed to ask the advice of the CCMAS concerning the possibility to convert the methods for heavy metals and copper into criteria and the suitability to use a method listing approach for the other elements.

The Committee agreed to forward the revised proposed draft Standard to the 34th Session of the Commission for adoption at Step 5 and the section on method of analysis and sampling to the CCMAS for endorsement and advice.

The 34th Session of the Commission adopted the draft revision at Step 5.

See **Table section B** for the complete list of methods of analysis.

C. COMMITTEE ON PROCESSED FRUITS AND VEGETABLES (CCPFV)

Standard for Desiccated Coconut³

The 34th Session of the Commission adopted the Standard at Step 8 by with some amendment⁴. The methods of analysis in the Standard newly proposed by CCPFV were already endorsed by the 32nd Session of CCMAS⁵.

The provisions for sampling plans as presented in REP11/PFV, Appendix III need to be endorsed. These provisions are related to Section 3.4 Lot Acceptance concerning quality requirements described in Section 3.2.

Standard for Canned Bamboo Shoots⁶

The Committee is invited to consider the ISO methods proposed for tin in view of its earlier recommendation to use the General Codex Method for processed fruits and vegetables.

See **Table section C** for the complete list of proposed methods of analysis.

D. COORDINATING COMMITTEE FOR THE NEAR EAST (CCNEA)

Regional Standard for Harissa⁷

Regional Standard for Halwa Tehenia⁸

The 34th Session of the Commission adopted the *Regional Standard for Harissa (red hot pepper paste)* and the *Regional Standard for Halwa Tehenia* at Steps 5/8 except for the section on methods of analysis and sampling, pending their endorsement.

See **Table section D** for the complete list of the proposed methods of analysis.

² REP11/FA, paras 133 – 136 and Appendix XI

³ REP11/PFV Appendix III

⁴ REP11/CAC, paras 27 – 28

⁵ REP11/MAS, paras 26 – 27 and Appendix III

⁶ REP11/PFV, Appendix V; REP11/EX para. 13

⁷ REP11/NEA, para. 75 and Appendix III

⁸ REP11/NEA, para. 79 and Appendix IV

PART II METHODS OF SAMPLING**A. COMMITTEE ON FISH AND FISHERY PRODUCTS (CCFFP)****Standard for Fish Sauce⁹**

See Section A of Part I for the background.

The proposed sampling plan is as follows:

Sampling of lots for examination of the final product shall be in accordance with the *General Guidelines on Sampling* (CAC/GL 50-2004). A sample unit is the individually packed product (bottle) or a 11 portion from bulk containers.

B. COMMITTEE ON PROCESSED FRUITS AND VEGETABLES (CCPFV)**Standard for Desiccated Coconut¹⁰**

See Section C of Part I for the background.

The proposed sampling plan and the sampling plan to be revoked are shown in **Appendix I**.

C. COORDINATING COMMITTEE FOR THE NEAR EAST (CCNEA)

See Section D of Part I for the background.

Regional Standard for Harissa¹¹

The proposed sampling plan is as follows:

Samples should be taken in accordance with an appropriate sampling plan with an AQL of 6.5. (See also *Codex General Guidelines on Sampling* CAC/GL 50-2004)

Regional Standard for Halwa Tehenia¹²

The proposed sampling plan is as follows:

Samples should be taken in accordance with an appropriate sampling plan with an AQL of 6.5. (See also *Codex General Guidelines on Sampling* CAC/GL 50-2004).

⁹ REP11/FFP, paras 34 – 37 and Appendix III

¹⁰ REP11/PFV Appendix III

¹¹ REP11/NEA, para. 75 and Appendix III

¹² REP11/NEA, para. 79 and Appendix IV

A. COMMITTEE ON FISH AND FISHERY PRODUCTS

Standard for Fish Sauce

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
Fish sauce	total nitrogen	AOAC 940.25	digestion	type I
Fish sauce	amino acid nitrogen	AOAC 2.066 AOAC 2.065	determining formaldehyde nitrogen subtracting by ammoniacal nitrogen	
Fish sauce	pH	AOAC 981.12 (Codex General Method)	electrometry	type II The pH shall be measured in a sample of fish sauce diluted with water to 1:10 using a pH meter. The dilution of fish sauce is necessary because of the high ionic strength in the undiluted sauce.
Fish sauce	sodium chloride	FAO 1981, Technical paper 219	titrimetry	
Fish sauce	sodium chloride	AOAC 937.13		<ul style="list-style-type: none"> • <i>This method is not applicable to the provision. This is a method for “mold in butter”.</i> • <i>Regarding NaCl, AOAC 937.09 (Codex general method) was already endorsed for “fish and fishery products” (type II)</i>
Fish sauce	sodium chloride	AOAC 976.18	potentiometry	type II
Fish sauce	sodium chloride	AOAC 976.19	Indicating strip method	type III
Fish sauce	histamine	AOAC 977.13 or other scientifically equivalent validated method	Fluorimetry	type II <i>AOAC 977.13 has already been endorsed for “fish and fishery products” (type II)</i>

B. COMMITTEE ON FOOD ADDITIVES

Draft Revision of the Standard for Food Grade Salt

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
food grade salt	sulphate	ISO 2480:1972	gravimetric	type II [already endorsed] (CCFA suggested keeping the “methods listing approach” for the provision)
food grade salt	sulphate	EuSalt/AS 015-2007	ICP-OES	type III See above
food grade salt	sulphate	EuSalt/AS 018-2005	Ion chromatography	type III See above
food grade salt	halogens	ISO 2481:1973	mercurimetry	type II [already endorsed] (CCFA suggested keeping the “methods listing approach” for the provision)
food grade salt	halogens	EuSalt/AS 016-2005	potentiometry	type III See above
food grade salt	halogens	EuSalt/AS 018-2005	ion chromatography	type III See above
food grade salt	calcium and magnesium	ISO 2482:1973	complexometric titrimetry	type II [already endorsed] (CCFA suggested keeping the “methods listing approach” for the provision)
food grade salt	calcium and magnesium	EuSalt/AS 009-2005	Flame atomic absorption spectrophotometry	type III See above
food grade salt	calcium and magnesium	EuSalt/AS 015-2007	ICP-OES	type III See above
food grade salt	potassium	ESPA/CN-E/104-1994 (applicable to products containing ≥ 2 mg-K/kg)	Flame atomic absorption spectrophotometry	deletion proposed
food grade salt	potassium	ESPA/CN-E/103-1994 (applicable to products containing ≥ 100 mg-K/kg)	Titrimetry	deletion proposed

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
food grade salt	potassium	EuSalt/AS 007-2005	volumetry	type II (CCFA suggested keeping the “methods listing approach” for the provision)
food grade salt	potassium	EuSalt/AS 008-2005	Flame atomic absorption spectrophotometry	type III See above
food grade salt	potassium	EuSalt/AS 015-2007	ICP-OES	type III See above
food grade salt	copper	ESPA/CN-E/101-1994	photometry	deletion proposed
food grade salt	copper	EuSalt/AS 005-2005	photometry	type II <u>Question from CCFA</u> : possibility to convert the methods into criteria ML: 2 mg/kg (expressed as Cu) ¹³
food grade salt	copper	EuSalt/AS 015-2007	ICP-OES	type III See above
food grade salt	arsenic	ESPA/CN-E/105-1996	photometry	deletion proposed
food grade salt	arsenic	EuSalt/AS 011-2005	photometry	type II
food grade salt	arsenic	EuSalt/AS 015-2007	ICP-OES	type III
food grade salt	mercury	ESPA/CN-E/106-1994	cold vapour atomic absorption spectrophotometry	deletion proposed
food grade salt	mercury	EuSalt/AS 012-2005	cold vapour atomic absorption spectrophotometry	type II <u>Question from CCFA</u> : possibility to convert the methods into criteria ML: 0.1 mg/kg (CODEX STAN 193-1995)
food grade salt	lead	ESPA/CN-E/108-1994	atomic absorption spectrophotometry	deletion proposed

¹³ REP11/FA, Appendix XI Section 3.2

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
food grade salt	lead	EuSalt/AS 013-2005	flame atomic absorption spectrometry	type II <u>Question from CCFA</u> : possibility to convert the methods into criteria ML: 2 mg/kg (CODEX STAN 193-1995)
food grade salt	lead	EuSalt/AS 015-2007	ICP-OES	type III see above
food grade salt	cadmium	ESPA/CN-E/107-1997	Atomic absorption spectrophotometry	deletion proposed
food grade salt	cadmium	EuSalt/AS 014-2005	flame atomic absorption spectrometry	type II <u>Question from CCFA</u> : possibility to convert the methods into criteria ML: 0.5 mg/kg (CODEX STAN 193-1995)
food grade salt	cadmium	EuSalt/AS 015-2007	ICP-OES	type III see above
food grade salt	iodine	ESPA/CN-E/109-1994	Titrimetry using sodium thiosulphate	deletion proposed
food grade salt	iodine	AOAC 925.56	Titrimetry using sodium thiosulphate	deletion proposed
food grade salt	iodine	EuSalt/AS 002-2005	Titrimetry using sodium thiosulphate	type II (CCFA suggested keeping the “methods listing approach” for the provision)
food grade salt	iodine	WHO/UNICEF/ICCIDD method ¹⁴	Titrimetry using sodium thiosulphate	See above
food grade salt	iodine	EuSalt/AS 019-2009	ICP-OES	type III See above

¹⁴ Assessment of iodine deficiency disorders and monitoring their elimination. A guide for programme managers. Third edition, Annex 1: Titration method for determining salt iodate and salt iodine content. World Health Organization, Geneva, 2007. The report is available from http://www.who.int/nutrition/publications/micronutrients/iodine_deficiency/WHO_NHD_01.1/en/index.html

C. COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

Standard for Canned bamboo Shoots

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
<u>Canned Bamboo Shoots</u>	Tin	ISO 7240:2004 and ISO 2447:1998	Flame atomic absorption spectrometry	type II

Note: The 24th CCPFV (2006) agreed to delete method ISO 2447:1998 in the Standard for Pickled Fruits and Vegetables following the request from CCMAS to clarify why this method was used and to consider using the General Codex Method AOAC 980.19 (Type II) (ALINORM 07/30/27, Appendix II)

D. COORDINATING COMMITTEE FOR THE NEAR EAST

Regional Standard for Harissa

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
harissa	acidity	ISO 750:1998	titrimetry	type I
harissa	dry extract – soluble solids	ISO 2173:2003	refractometry	type I
harissa	acid insoluble ash	ISO 763:2003	gravimetry	type I
harissa	colour	“Hunter” method		

Regional Standard for Halwa Tehenia

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
halwa tehenia	moisture	AOAC 925.45	gravimetry	type I
halwa tehenia	fatty substances	AOAC 963.15	gravimetry	type I
halwa tehenia	ash	AOAC 900.02	gravimetry	type I
halwa tehenia	sugars (estimated as sucrose)	AOAC 930.15		<i>This method is not applicable to the provision. This is a method for “loss on drying” and “dry matter on oven drying”</i>
halwa tehenia	acidity	AOAC 900.02		<i>This method is not applicable to the provision. This is a method for “ash of sugars and syrups”</i>

Appendix I

Proposed Sampling Plan for Desiccated Coconut**Sampling Plans**

The appropriate inspection level is selected as follows:

Inspection level I - Normal Sampling

Inspection level II -Disputes, (Codex referee purposes sample size), enforcement or need for better lot estimate

**SAMPLING PLAN 1
(Inspection Level I, AQL = 6.5)**

NET WEIGHT IS EQUAL TO OR LESS THAN 1 KG (2.2 LB)		
Lot Size (N)	Sample Size (n)	Acceptance Number (c)
4,800 or less	6	1
4,801 - 24,000	13	2
24,001 - 48,000	21	3
48,001 - 84,000	29	4
84,001 - 144,000	38	5
144,001 - 240,000	48	6
more than 240,000	60	7
NET WEIGHT IS GREATER THAN 1 KG (2.2 LB) BUT NOT MORE THAN 4.5 KG (10 LB)		
Lot Size (N)	Sample Size (n)	Acceptance Number (c)
2,400 or less	6	1
2,401 - 15,000	13	2
15,001 - 24,000	21	3
24,001 - 42,000	29	4
42,001 - 72,000	38	5
72,001 - 120,000	48	6
more than 120,000	60	7
NET WEIGHT GREATER THAN 4.5 KG (10 LB)		
Lot Size (N)	Sample Size (n)	Acceptance Number (c)
600 or less	6	1
601 - 2,000	13	2
2,001 - 7,200	21	3
7,201 - 15,000	29	4
15,001 - 24,000	38	5
24,001 - 42,000	48	6
more than 42,000	60	7

SAMPLING PLAN 2
(Inspection Level II, AQL = 6.5)

NET WEIGHT IS EQUAL TO OR LESS THAN 1 KG (2.2 LB)		
Lot Size (N)	Sample Size (n)	Acceptance Number (c)
4,800 or less	13	2
4,801 - 24,000	21	3
24,001 - 48,000	29	4
48,001 - 84,000	38	5
84,001 - 144,000	48	6
144,001 - 240,000	60	7
more than 240,000	72	8
NET WEIGHT IS GREATER THAN 1 KG (2.2 LB) BUT NOT MORE THAN 4.5 KG (10 LB)		
Lot Size (N)	Sample Size (n)	Acceptance Number (c)
2,400 or less	13	2
2,401 - 15,000	21	3
15,001 - 24,000	29	4
24,001 - 42,000	38	5
42,001 - 72,000	48	6
72,001 - 120,000	60	7
more than 120,000	72	8
NET WEIGHT GREATER THAN 4.5 KG (10 LB)		
Lot Size (N)	Sample Size (n)	Acceptance Number (c)
600 or less	13	2
601 - 2,000	21	3
2,001 - 7,200	29	4
7,201 - 15,000	38	5
15,001 - 24,000	48	6
24,001 - 42,000	60	7
more than 42,000	72	8

Sampling Plan to be Revoked

The provisions for sampling as stated in CODEX STAN 177-1991 should be revoked and replaced by sampling plans 1 and 2, AQL = 6.5.

The sections indicated in the proposal for revocation corresponds to those of CODEX STAN 177-1991.

SAMPLING PLANS AS CURRENTLY STANDS IN CODEX STAN 177-1991

- ~~1. Instructions for drawing primary samples according to ISO 2170-1980 (Cereals and Pulses) or ICC Method of Sampling No. 101-1960 (Sampling of Milled Products).~~
- ~~2. The size of the sample to be undertaken from a homogeneous lot should be in accordance with Table 3 of the Instructions on Codex Sampling Procedures (CX/MAS 1-1987, Appendix V).~~
- ~~3. For all determinations the laboratory sample should be prepared according to the variables plan for proportion defective (CX/MAS 1-1987, Appendix IV).~~
- ~~4. For all determinations, except granularity (Section 2.2), analysis should be performed on the "blended bulk sample".~~

~~For verification of granulation, i.e. size grade (Sections 2.2 and 7.1) as declared on the label, the determination in consignments of repacked product should be on individual packages.~~