CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Item 4 **CX/MAS 11/32/4**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Thirty-second Session
Budapest, Hungary, 7 - 11 March 2011

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:

- A. Codex Committee on Contaminants in Foods
- B. Codex Committee on Processed Fruits and Vegetables
- C. Codex Committee on Nutrition and Foods for Special Dietary Uses
- D. FAO/WHO Coordinating Committee for Asia
- E. Natural Mineral Waters
- F. Milk and Milk Products
- G. Sugars and honey

PART I. METHODS OF ANALYSIS

A. COMMITTEE ON CONTAMINANTS IN FOODS

Proposed Draft Maximum Levels for Melamine in Food and Feed (at Steps 5/8) (ALINORM 10/33/41, paras 66 and 68 and Appendix IV)

In relation to methods of analysis for verification of compliance with the MLs, the 4th Session of CCCF was agreed to request CCMAS to identify appropriate methods for the measurement of melamine in powdered infant formula and foods (other than infant formula) and feeds.

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

B. COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

Proposed Draft Standard for Desiccated Coconut (At Steps 5/8) (REP 11/PFV, Appendix III)

The 25th Session of CCPFV agreed to propose to CCMAS to endorse the ISO 660:1996 for total acidity of the extracted oil and to revoke the corresponding Codex Recommended Method for total acidity as previously described in the Standard, and as an alternative to the ISO method, to endorse the AOCS Cd 3d-63. Due to the revision of the Standard, the method for granularity was deleted as unnecessary and to inform CCMAS accordingly.

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

Codex Standard for Certain Canned Vegetables (palmito) (REP11/PFV paras 12, 13)

The 25th Session of CCPFV noted that the 30th Session of CCMAS agreed to seek clarification as to whether ISO 762:1982 for the determination of mineral impurities in canned palmito should be retained in view of the endorsement of AOAC 971.33 for the determination of mineral impurities in canned vegetables as Type I.

CCPFV acknowledged that both methods were equivalent and should be retained in the Standard for Certain Canned Vegetables. The Committee agreed to keep AOAC 971.33 as the general Codex method for the determination of mineral impurities (sand) in processed fruits and vegetables (Type I) and to retain ISO 762:1982 as an alternative method.

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

C. COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

Method of analysis of dietary fibre (REP 11/NFSDU, paras 14 – 16 and Appendix VI)

The Committee recalled that the 31st Session of CCMAS had indicated that most of the methods of analysis for dietary fibre were empirical and some of them might be overlapping, and therefore had agreed that they could be endorsed as Type IV in order to make them available as Codex methods and asked the CCNFSDU to define their scope more precisely.

The Committee agreed to change the provisions for six general methods of analysis to describe them more precisely and proposed them as Type I methods. Regarding eight methods that measure individual specific components, the Committee agreed to propose them as Type I methods. Regarding the three "other methods", the Committee agreed to propose that they should be maintained as Type IV methods. Some delegations indicated that they were unable to comment at this stage and would make their comments to the CCMAS.

In reply to the proposal of CCMAS to delete the AOAC 2001.03 method, the Committee agreed to keep it because it was applicable when resistant starches are not present and AOAC 2009.01 was applicable to food that may, or may not, contain resistant starches.

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

D. FAO/WHO COORDINATING COMMITTEE FOR ASIA

Proposed Draft Regional Standard for Chili Sauce (At Steps 5/8) (REP 11/ASIA, Appendix III)

The 17th Session of CCASIA agreed to forward the sections on food additives, labelling and methods of analysis and sampling respectively to CCFA, CCFL and CCMAS for endorsement and to forward the Proposed Draft Regional Standard to the Commission for adoption at Steps 5/8, with the recommendation to omit Steps 6 and 7.

See **Table section D** for the complete list of methods of analysis and **Annex III** for the proposed sampling plans.

E. NATURAL MINERAL WATERS

Methods of Analysis in Codex Standards at Different Steps, including Methods of Analysis for Natural Mineral Waters (ALINORM 10/33/REP, para. 38)

During the 33rd Codex Alimentarius Comission (CAC), the Delegation of Malaysia, referring to its comments in CRD 12, proposed to insert several additional methods of analysis for the determination of health-related substances in mineral waters. The Commission adopted the methods as proposed by the Committee on Methods of Analysis and Sampling and noted that additional methods for natural mineral waters could be proposed for consideration by the next session of the Committee of Method Analysis and Sampling (CCMAS).

Hence, Malaysia would like to make the following proposals:-

i) In addition to the proposed methods from ISO methods as listed, Malaysia would like to suggest the inclusion of American Public Health Association (APHA) and the US Environmental Protection Agency (EPA) for method of analysis for water testing. These American based official methods have commonly being used in some countries including Malaysia for analysis for water testing including natural mineral waters. In addition the performance characteristics of these suggested methods are also within those stated in the proposed list (See Appendix II, matters D). The detailed performance characteristics are as listed in the table.

Therefore, Malaysia would like the Committee to consider our proposals to include the methods based on APHA and EPA for all the provisions in the CODEX STAN 108-1981.

See **Table Section E** for the complete list of methods of analysis.

F. MILK AND MILK PRODUCTS (ALINORM 10/33/23, para. 70)

It was noted that the Committee on Milk and Milk Products had completed its work and had proposed to adjourn *sine die,* while work on methods of analysis and sampling for milk and milk products was ongoing in IDF and ISO. The Committee agreed that it would continue reviewing the methods applicable to milk and milk products following the adjournment of the Committee on Milk and Milk Products.

See **Table Section F** for the proposed update for methods of analysis.

G. SUGARS AND HONEY (ALINORM 01/23, Appendix IV)

As the Committee on Sugars was adjourned *sine die* in 2001, the questions from CCMAS to the committee remained pending. CCMAS is therefore invited to review these methods to decide on their status. See **Table Section G** for the methods of analysis for honey.

PART II. SAMPLING

A. COMMITTEE ON CONTAMINANTS IN FOODS

Proposed Draft Maximum Levels for Total Aflatoxins in Brazil Nuts (at Steps 5/8) (ALINORM 10/33/41, para. 75 and Appendix V)

The 4th Session of CCCF agreed that the sampling plans for total aflatoxins in Brazil nuts should be integrated into the sampling plans for aflatoxin contamination in ready-to-eat treenuts and treenuts destined for further processing and amended the document accordingly. The Committee further noted that only those sections relating to Brazil nuts were for adoption by the Commission.

See **Annex I** for the proposed sampling plans.

B. COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

Codex Standards for Processed Fruits and Vegetables (REP 11/PFV paras 9, 10)

The 25th Session of CCPFV noted that the 30th Session of CCMAS (2009) could not identify the purpose of the sampling plans in the annexes of the Standard for Jams, Jellies and Marmalades (CODEX STAN 296-2009) and the Standard for Certain Canned Vegetables (CODEX STAN 297-2009) therefore requested the Committee to clarify which provisions in these standards the sampling plans applied to.

CCPFV clarified that provisions for lot acceptance (sampling plans with an AQL of 6.5) in the Standard for Jams, Jellies and Marmalades and in the Standard for Certain Canned Vegetables applied to provisions falling under the quality criteria (section 3.3 for jams, jellies and marmalades and section 3.2 for canned vegetables) and the minimum fill (section 7.1 for jams, jellies and marmalades and sections 7.1.1 – 7.1.2 for canned vegetables).

See **Annex II** for the proposed sampling plans.

A. COMMITTEE ON CONTAMINANTS IN FOODS

Proposed Draft Standard for Desiccated Coconut

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
Milk, milk products and infant formulae	melamine	ISO/TS 15495 | IDF/RM 230:2010	LC-MS/MS	Guidelines for the quantitative determination of melamine and cyanuric acid by LC-MS/MS

B. COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

Proposed Draft Standard for Desiccated Coconut

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
Desiccated Coconut	Ash	AOAC 950.49	Gravimetry	Type I
Desiccated Coconut	Extraneous vegetable material	See below	Counting extraneous material with the naked eye	Type IV
Desiccated Coconut	Moisture	AOAC 925.40	Gravimetry (loss on drying)	Type I
Desiccated Coconut	Oil content	AOAC 948.22	Gravimetry	Type I
Desiccated Coconut	Total acidity of the extracted oil	ISO 660:1996 amended 2003; or AOCS Cd 3d-63	Titrimetry	Type I

Determination of extraneous vegetable matter

The determination is carried out by spreading 100 g of the sample in a thin layer against a white background and counting the extraneous material with the naked eye.

Standard for Certain Canned Vegetables (palmito)

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
Certain canned vegetables	mineral impurities (sand)	ISO 762:1982	Gravimetry	to retain as alternative method (AOAC 971.33 was endorsed as Type I at the 30th CCMAS)

C. COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

Method of Analysis of Dietary Fibre

Standard	Provisions	Method	Principle	Type
General methods that do not measure the lower molecular weight fraction (i.e. monomeric units<=9)[(2)]				
All foods (1)	~~Dietary fibre based on precipitation in 4 parts alcohol and 1 part water. Resistant insoluble and soluble polysaccharides, lignin, and plant cell wall. (4)~~ ~~(Total dietary fibre)~~ Method applicable for determining dietary fibres that do not include the lower molecular weight fraction. (4)	AOAC 985.29 AACC Intl 32-05.01 (1991,1999)	Enzymatic gravimetric	I~~V~~
All foods (1)	~~Dietary fibre based on precipitation in 80% ethanol. Resistant insoluble and soluble polysaccharides, lignin, and plant cell wall (4).~~ ~~(Can determine total, but also determines soluble a insoluble dietary fibre~~) Method applicable for determining dietary fibres that do not include the lower molecular weight fraction and also includes determination for soluble and insoluble dietary fibres (4)	AOAC 991.43 AACC Intl 32-07.01 (1999, 1991) NMKL 129, 2003	Enzymatic gravimetric	I~~V~~
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction, in foods and food products containing more than 10% dietary fibres and less than 2% starch (e.g. fruits) ~~(Foods with >10% TDF and < 2% starch (fruits))~~ (4)	AOAC 993.21	Non-enzymatic gravimetric	I~~V~~
All foods (1)	~~Dietary fibre based on precipitation in 4 parts alcohol and 1 part water, quantitated as component neutral sugars, uronic acids, plus Klason lignin. (4)~~ ~~(Determine sugars, useful for commodity where fibre a sugar are both necessary)~~ Method applicable for determining dietary fibres that do not include the lower molecular weight fraction. Provides sugar residue composition of dietary fibre polysaccharides, as well as content of Klason lignin (4).	AOAC 994.13 AACC Intl 32- 25.01 (1999, 1994) NMKL 162, 1998	Enzymatic chemical	I~~V~~

General methods that measure both the higher (monomeric units > 9) and the lower molecular weight fraction (monomeric units <=9) (2)				
All foods (1)	~~Dietary fibre based on precipitation in 4 parts alcohol and 1 part water. Resistant insoluble and soluble polysaccharides, resistant maltodextrins, lignin, and plant cell wall. (3)~~ Method applicable for determining the content of dietary fibres of higher and lower molecular weight, in food where resistant starches are not present	AOAC 2001.03 AACC Intl 32-41.01 (2002)	Enzymatic gravimetric and Liquid chromatography	~~IV~~
All foods (1)	~~Dietary fibre (Soluble + insoluble polysaccharides + lignin + resistant starch + oligosaccharides)~~ Method applicable for determining the content of dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches.	AOAC 2009.01 AACC Intl 32-45.01 (2009)	Enzymatic-Gravimetric-High Pressure Liquid Chromatography Method	~~IV~~
Methods that measure individual specific components (monomeric units: the whole range for each type of components is covered) (2)				
All foods (1)	Insoluble dietary fibres in food and food products	AACC Intl 32-20.01 (1999, 1982) AOAC 991.42 (Specific for insoluble fibre)	Enzymatic gravimetric	~~IV~~
All foods (1)	Soluble dietary fibres in food and food products	AOAC 993.19 (Specific for soluble fibre)	Enzymatic gravimetric	~~IV~~
All foods (1)	(1→3)(1→4) *Beta*-D-Glucans	AOAC 995.16 AACC Intl 32-23.01 (1999, 1995)	Enzymatic	~~IV~~
All foods (1)	Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, fructooligosaccharides) (applicable to added fructans)	AOAC 997.08 AACC Intl 32-31.01 (2001)	Enzymatic & HPAEC-PAD	~~IV~~
All foods (1)	Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, fructooligosaccharides) (not applicable highly depolymerised fructans)	AOAC 999.03 AACC Intl 32-32.01 (2001)	Enzymatic & colorimetric	~~IV~~
All foods (1)	Polydextrose	AOAC 2000.11 AACC Intl 32-28.01 (2001)	HPAEC-PAD	~~IV~~
All foods (1)	Trans-galacto-oligo saccharides	AOAC 2001.02 AACC Intl 32-33.01 (2001)	HPAEC-PAD	~~IV~~
All foods (1)	Resistant starch (Recommended for RS3)	AOAC 2002.02 AACC Intl 32-40.01 (2002)	Enzymatic	~~IV~~

Other methods[(2)] that have not been subjected to interlaboratory evaluation under AOAC international guidelines				
All foods	Insoluble glucans and mannans of yeast cell wall (for yeast cell wall only)	Eurasyp (European association for specialty yeast product) – LM Bonanno. Biospringer- 2004 – online version : http://www.eurasyp.org/public.technique.home.screen.	Chemical & HPAEC-PAD	IV
All foods	Fructo-oligosaccharides (monomeric units<5)	Ouarné et al. 1999 in *Complex Carbohydrates in Foods*. Edited by S. Sungsoo, L. Prosky & M. Dreher. Marcel Dekker Inc, New York	HPAEC-PAD	IV
All foods	Non-starch polysaccharides (NSP) (3)	Englyst H.N, Quigley M.E., Hudson G. (1994) Determination of dietary fibre as non-starch polysaccharides with gas-liquid chromatographic high performance liquid chromatographic or spectrophotometric measurement of constituent sugars – Analyst 119, 1497-1509	Gas-Liquid Chromatography	IV

[(1)] Users should consult the description of each method for the food matrices that were the subject of interlaboratory study in the Official methods of Analysis of AOAC International.
[(2)] Two issues are left for national authorities: to include monomeric units 3-9 and which isolated or synthetic compounds have physiological benefit. (Refer to the Guidelines for Nutrition Labelling (CAC/GL 2-1985), as revised in 2009.
[(3)] Quantitation lost for resistant starch.Refer to specific methods.
[(4)] Quantitation lost for inulin, resistant starch, polydextrose and resistant maltodextrins. Refer to specific methods.

D. FAO/WHO COORDINATING COMMITTEE FOR ASIA

Proposed Draft Regional Standard for Chili Sauce

COMMODITY	PROVISION	METHOD	PRINCIPLE	Notes and Type proposed
Chili sauce	pH	AOAC 981.12	Potentiometry	Type III – Codex General Method for processed fruits and vegetables
Chili sauce	Fill of containers	CAC/RM 46-1972	Weighing	Type I – Codex General Method for processed fruits and vegetables

E. NATURAL MINERAL WATERS

Methods of Analysis for Natural Mineral Waters (the inclusion of the suggested methods are in italic bold):

Provision	**ML (mg/L)**	**Min Applicable (mg/L)**	**LOD (mg/L)**	**LOQ (mg/L)**	**Precision RSDR (%) Not more than**	**Recovery (%)**	**Suggested method meeting the criteria**	**Principle**
Antimony	0.005	0.0028	0.001	0.002	44	80-110	ISO 17294-2:2003 ISO 15586:2003 ***EPA 200.8***	ICP-MS GF-AAS ***ICP-MS***
Arsenic	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2:2003 ISO 15586:2003 ISO 11969:1996 ***EPA 200.8***	ICP-MS GF-AAS AAS-hydride ***ICP-MS***
Barium	0.7	0.35	0.07	0.14	34	95-105	ISO 11885:2007 ISO 17294-2:2003 ***EPA 200.8***	ICP-OES ICP-MS ***ICP-MS***
Borate	5	3.1	0.5	1	25	97-103	ISO 9390:1990 ISO 11885:2007 ISO 17294-2:2003 ***EPA 200.8***	Spectrophotometry ICP-MS ICP-MS ***ICP-MS***
Cadmium	0.003	0.0017	0.0006	0.0012	44	80-110	ISO 11885:2007 ISO 17294-2:2003 ISO 15586:2003 ISO 5961:1994 ***EPA 200.8***	ICP-OES ICP-MS GF-AAS AAS ***ICP-MS***
Chromium	0.05	0.028	0.01	0.02	44	90-107	ISO 11885:2007 ISO 17294-2:2003 ISO 15586:2003 ISO 18412:2005 (Cr VI) ISO 23913:2006 (Cr VI) ISO 9174:1998 ***EPA 200.8***	ICP-OES ICP-MS GF-AAS Photometric CIA, AAS spectrophotometry ***ICP-MS***
Copper	1	0.52	0.1	0.2	32	97-103	ISO 11885:2007 ISO 17294-2:2003	ICP-OES ICP-MS

Provision	**ML (mg/L)**	**Min Applicable (mg/L)**	**LOD (mg/L)**	**LOQ (mg/L)**	**Precision RSDR (%) Not more than**	**Recovery (%)**	**Suggested method meeting the criteria**	**Principle**
							ISO 15586:2003 ISO 8288:1986 ***EPA 200.8***	GF-AAS AAS ***ICP-MS***
Cyanide	0.07	0.039	0.014	0.028	44	90-107	ISO 14403:2002 ISO 6703-1:1998 ***APHA 4500***	CFA Photometric, trometric ***Colometric***
Fluoride	1.0	0.52	0.1	0.2	32	97-103	ISO 10304-1:2007 ISO 10359-1:1994 (dissolved fluoride) ISO 10359-2:1994 (inorganic bound) ***APHA 4110 B***	HPLC Electrochemical probe Digestion, distillation ***Ion chromatography***
Lead	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2:2003 ISO 15586:2003 ISO 8288:1986 ***EPA 200.8***	ICP-MS GF-AAS AAS ***ICP-MS***
Manganese	0.4	0.18	0.04	0.08	37	95-105	ISO 11885:2007 ISO 17294-2:2003 ISO 15586:2003 ***EPA 200.8***	ICP-OES ICP-MS GF-AAS ***ICP-MS***
Mercury	0.001	0.00056	0.0002	0.0004	44	80-110	EN 1483:2007 ISO 17852:2006 ISO 5666:1999 ISO 16590:2000 ***EPA 200.8***	AAS – Enrichment by amalgamation (II) AFS AAS after tin (II) chloride reduction Enrichment by amalgamation (II) ***ICP-MS***
Nickel	0.02	0.011	0.004	0.008	44	90-107	ISO 17294-2:2003 ISO 15586:2003 ***EPA 200.8***	ICP-MS GF-AAS ***ICP-MS***
Nitrate	50	37	5	10	18	98-102	ISO 10304-1:2007 ISO 13395:1996	HPLC CFA, FIA, Spectromphotometry

Provision	**ML (mg/L)**	**Min Applicable (mg/L)**	**LOD (mg/L)**	**LOQ (mg/L)**	**Precision RSDR (%) Not more than**	**Recovery (%)**	**Suggested method meeting the criteria**	**Principle**
							ISO 7890-3:1988 ***APHA 4500***	Spectrophotometry ***CFA***
Nitrite	0.1	0.03	0.01	0.02	44	95-105	ISO 10304-1:2007 ISO 13395:1996 ISO 6777:1984 ***APHA 4500***	HPLC CFA, FIA, Spectromphotometry Spectrophotometry ***CFA***
Selenium	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2:2003 ISO 15586:2003 ISO 9965:1993 ***EPA 200.8***	ICP-MS GF-AAS AAS (Hydride) ***ICP-MS***
Surface active agents	-	0.1 – 5.0 mg/L 0.25 – 0.8 mg/L 0.05 – 5.0 mg/L	0.05 m/L			19 10 <44	ISO 16265:2009 ***APHA 4500***	CFA ***CFA***
Mineral Oil (hydrocarbon index)	-	> 0.1 mg/L				<41	ISO 9377-2:2000 ***EPA 8015***	GC ***GC***
PCB		> 10 ng/L >15 ng/L				27-79 <20	ISO 9377-2:2000 AOAC 990.16 ***EPA 1613***	GC ECD GC ECD ***HRGC_HRMS***
Pesticide (organochloride	-	>10 ng/L > 15 ng/L				27-79 <20	ISO 6468 :1996 AOAC 990.16 ***EPA 508.1***	GC ECD GC ECD ***GC ECD***
PAHs	-	0.005 ug/L 0.04 ug/L 0.005 ug/L				<10 <18 <19	ISO 17993:2004 ISO 7981-1:2005 ISO 7981-2:2005 ***EPA 550.1***	HPLC ECD TLC HPLC ***HPLC***

Performance characteristic obtained from APHA and EPA methods :

No.	Provision	LOD (mg/L)	Recovery %	Additional method suggested	Principle
1	Antimony	0.0004	99-101	EPA 200.8	ICP-MS
2	Arsenic (As)	0.0014	99-103	EPA 200.8	ICP-MS
3	Barium (Ba)	0.0008	96	EPA 200.8	ICP-MS
4	Borate	0.002	78	EPA 200.8	ICP-MS
5	Cadmium (Cd)	0.0005	97-102	EPA 200.8	ICP-MS
6	Chromium (Cr)	0.0009	99-111	EPA 200.8	ICP-MS
7	Copper (Cu)	0.00009	93-95	EPA 200.8	ICP-MS
8	Cyanide (as CN^-)	0.02	93	APHA 4500 CN^-	Colorimetric
9	Fluoride (as F^-)	0.03	96-102	APHA 4110 B	Ion Chromatography with Chemical Suppression of Eluent Conductivity
10	Lead (Pb)	0.0006	97-99	EPA 200.8	ICP-MS
11	Manganese (Mn)	0.0001	95-97	EPA 200.8	ICP-MS
12	Mercury (Hg)	0.0001	90	EPA 200.8	ICP-MS
13	Nickel	0.0005	95-100	EPA 200.8	ICP-MS
14	Nitrates (as NO3-)	0.01	97-101	APHA 4500	Continuous-flow analytical (CFA)
15	Nitrites (as NO2-)	0.004	97-101	APHA 4500	Continuous-flow analytical (CFA)
16	Selenium (Se)	0.0079	93-99	EPA 200.8	ICP-MS
17	Surface active agents	0.003	97-104	APHA 4500	CFA
18	Mineral Oil (hydrocarbon index)	0.1	117	EPA 8015	GC
19	PCB	4.4 pg/L	25-197	EPA 1613	HRGC-HRMS
20	Pesticide (organochlorine)	0.0054	105	EPA Method 508.1; APHA 6630	GC-ECD
21	PAHs	0.0049 ug/L	86-99	EPA Method 550.1	HPLC

F. MILK AND MILK PRODUCTS

Update to the current list of recommended IDF/ISO methods in the section Milk and Milk products of Codex Stan 234

Proposed changes are shown in **~~bold strikethrough~~** for deletion and **bold underlined** for additions.

The table below includes the modifications adopted in 2008, and 2010 (Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008 and Appendix II Alinorm 10/33/23 CCMAS 2010).

Products	Provisions	Method	Principle	Type
Milk products	Iron	**ISO 6732|IDF 103:2010** **~~IDF 103A:1986 / ISO 6732:1985~~**	Photometry (bathophenanthroline)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF)[1]	**ISO 6731|IDF 21:2010** **~~IDF 21B:1987/ISO 6731:1989~~** and ISO 1737| IDF 13:2008	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	MSNF[1]	**ISO 6731|IDF 21:2010** **~~IDF 21B:1987/ISO 6731:1989~~** and ISO 1737| IDF 13:2008	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	I
Blend of sweetened condensed skimmed milk and vegetable fat	Milk solids-not-fat (MSNF)[1]	**ISO 6734|IDF 15:2010** **~~IDF 15B:1991 / ISO 6734:1989~~**	Calculation from total solids content, fat content and sugar content	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	MSNF[1]	**ISO 6734|IDF 15:2010** **~~IDF 15B:1991 / ISO 6734:1989~~**	Calculation from total solids content, fat content and sugar content	IV
Cream	Solids	**ISO 6731|IDF 21:2010** **~~IDF 21B:1987 / ISO 6731:1989~~**	Gravimetry (drying at 102 °C)	I
Edible casein products	pH	**ISO 5546|IDF 115:2010** **~~IDF 115A:1989 / ISO 5546:1979~~**	Electrometry	IV
Evaporated milks	Solids, total	**ISO 6731|IDF 21:2010** **~~IDF 21B:1987 / ISO 6731:1989~~**	Gravimetry (drying at 102 °C)	I
Milk powders and cream powders	Acidity, titratable	**ISO 6091|IDF 86:2010** **~~IDF 86:1981 / ISO 6091:1980~~**	Titrimetry, titration to pH 8.4	I
Milk fat products (anhydrous milk fat)	Peroxide value	ISO 3976|IDF 74:2006	Photometry	I
Sweetened Condensed Milks	Solids	**ISO 6734/IDF 15:2010** **~~IDF 15B:1991 / ISO 6734:1989~~**	Gravimetry, drying at 102 °C	I

Products	Provisions	Method	Principle	Type
Whey cheeses by coagulation	Milk fat in dry matter		**Calculation from fat content and dry matter content**	I
		ISO 1735|IDF 5:2004 and ISO 5534|IDF 4:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff) Gravimetry, drying at 102 °C	~~**IV**~~ ~~**IV**~~
			~~**Calculation from fat content and dry matter content**~~	

G. SUGARS AND HONEY

As the Committee on Sugars was adjourned *sine die* in 2001, the questions from CCMAS to the committee remained pending. CCMAS is therefore invited to review these methods to decide on their status.

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Honey	Fructose and Glucose (sum of both)	Harmonised method of the EHC, Apidologie, Special Issue 28, 1997, Chapter 1.7..2	HPLC	The Commodity Committee is requested to verify that a collaborative study has been performed on this method.	II	TE
Honey	Sucrose content	Harmonised method of the EHC, Apidologie, Special Issue 28, 1997, Chapter 1.7.2	HPLC	The Commodity Committee is requested to verify that a collaborative study has been performed on this method.	II	TE
Honey	Electrical conductivity	Harmonised method of the EHC, Apidologie, Special Issue 28, 1997, Chapter 1.2		The Commodity Committee is requested to verify that a collaborative study has been performed on this method.	I	TE

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note	Type	Status
Honey	Diastase activity	Phadebas – Harmonised method of the EHC	Enzyme	The Commodity Committee is requested to verify that the reagents for the method are available, and a collaborative study has been performed on this method and to provide a method reference.	III	TE
Honey	Hydroxymethylfurfural	Harmonised method of the EHC	HPLC	The Commodity Committee is requested to verify that a collaborative study has been performed on this method and to provide a method reference.	III	TE

ANNEX I

SAMPLING PLANS FOR AFLATOXIN CONTAMINATION IN READY-TO-EAT TREENUTS AND TREENUTS DESTINED FOR FURTHER PROCESSING: ALMONDS, HAZELNUTS, PISTACHIOS AND SHELLED BRAZIL NUTS

(This document includes the sections with actual changes only.)

SAMPLING PLAN DESIGN CONSIDERATIONS

1. Importers may commercially classify treenuts as either "ready-to-eat" (RTE) or "destined for further processing" (DFP). As a result, maximum levels and sampling plans are proposed for both commercial types of treenuts. Maximum levels need to be defined for treenuts destined for further processing and ready-to-eat treenuts before a final decision can be made about a sampling plan design.

2. Treenuts can be marketed either as inshell or shelled nuts. For example, pistachios are predominately marketed as inshell nuts while almonds are predominately marketed as shelled nuts.

3. Sampling statistics, shown in Annex I, are based upon the uncertainty and aflatoxin distribution among laboratory samples of shelled nuts. Because the shelled nut count per kg is different for each of the treenuts, the laboratory sample size is expressed in number of nuts for statistical purposes. However, the shelled nut count per kg for each treenut, shown in Annex I, can be used to convert laboratory sample size from number of nuts to mass and vice versa.

4. Uncertainty estimates associated with sampling, sample preparation, and analysis, shown in Annex I, and the negative binomial distribution[1],[2],[3] are used to calculate operating characteristic (OC) curves that describe the performance of the proposed aflatoxin-sampling plans (Annex II).

5. In Annex I, the analytical variance reflects a reproducibility relative standard deviation of 22%, which is suggested by Thompson and is based upon Food Analysis Performance Assessment Scheme (FAPAS) data[2]. A relative standard deviation of 22% is considered by FAPAS as an appropriate measure of the best agreement that can be reliably obtained between laboratories. An analytical uncertainty of 22% is larger than the within laboratory variation measured in the sampling studies for the four treenuts. The within laboratory analytical uncertainty for almonds, hazelnuts and pistachios can be found at the website http://www5.bae.ncsu.edu/usda/www/ResearchActDocs/treenutwg.html and for Brazil nuts in the CONFORCAST[3].

6. The issue of correcting the analytical test result for recovery is not addressed in this document. However, Table 2 specifies several performance criteria for analytical methods including suggestions for the range of acceptable recovery rates.

AFLATOXIN TEST PROCEDURE AND MAXIMUM LEVELS

7. An aflatoxin-sampling plan is defined by an aflatoxin test procedure and a maximum level. A value for the proposed maximum level and the aflatoxin test procedure are given below in this section.

8. The maximum levels for total aflatoxins in treenuts (almonds, hazelnuts, pistachios and shelled Brazil nuts) "ready-to-eat" and "destined for further processing" are 10 and 15 µg/kg, respectively.

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[1] Whitaker, T., Dickens, J., Monroe, R., and Wiser, E. 1972. Comparison of the negative binomial distribution of aflatoxin in shelled peanuts to the negative binomial distribution. J. American Oil Chemists' Society, 49:590-593.

[2] Thompson, M. 2000. Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing. J. Royal Society of Chemistry, 125:385-386.

[3] CONFORCAST. Ferramentas Analíticas para Capacitação do Brasil na Garantia da Conformidade da Castanha-Do-Brasil (Bertholletia Excelsa) quanto ao Perigo aflatoxina. Projeto nº 1.265/05, Aprovado pela FINEP na Chamada Pública, "Ação Transversal - TIB - 06/2005 - Linha 1". MAPA. Minist~erio da Agricultura, pecuária e do Abasteciento. Secretaria de Defesa Agropecuária - DAS, Departamento de Inspeção de Produtos de Origem Vegetal – DIPOV. Coordenação-Geral de Apoio Laboratorial – CGAL, Laboratório Nacional Agropecuário – LANAGRO/MG, United States Department of Agriculture (Thomas Whitaker and Andy Slate).

9. Choice of the number and size of the laboratory sample is a compromise between minimizing risks (false positives and false negatives) and costs related to sampling and restricting trade. For simplicity, it is recommended that the proposed aflatoxin sampling plans use a 20 kg aggregate sample for all four treenuts.

10. The two sampling plans (RTE and DFP) have been designed for enforcement and controls concerning total aflatoxins in bulk consignments (lots) of treenuts traded in the export market.

Treenuts destined for further processing

Maximum level – 15 µg/kg total aflatoxins

Number of laboratory samples – 1

Laboratory sample size - 20 kg
Almonds – shelled nuts
Hazelnuts – shelled nuts
Pistachios – inshell nuts (equivalent to about 10kg shelled nuts that is calculated on the basis of the actual edible portion in the sample)
Brazil nuts – shelled nuts

Sample preparation – sample shall be finely ground and mixed thoroughly using a process, e.g., dry grind with a vertical cutter mixer type mill, that has been demonstrated to provide the lowest sample preparation variance. Preferably, Brazil nuts should be ground as slurry.

Analytical method – performance based (see Table 2)

Decision rule – If the aflatoxin test result is less than or equal to 15 µg/kg total aflatoxins, then accept the lot. Otherwise, reject the lot.

The operating characteristic curve describing the performance of the sampling plan for the three treenuts destined for further processing is shown in Annex II.

Ready-to-eat treenuts

Maximum level – 10 µg/kg total aflatoxins

Number of laboratory samples – 2

Laboratory sample size - 10 kg
Almonds – shelled nuts
Hazelnuts – shelled nuts
Pistachios – inshell nuts (equivalent to about 5 kg shelled nuts per test sample that is calculated on the basis of the actual edible portion in the sample)
Brazil nuts – shelled nuts

Sample preparation – sample shall be finely ground and mixed thoroughly using a process, e.g., dry grind with a vertical cutter mixer type mill, that has been demonstrated to provide the lowest sample preparation variance. Preferably, Brazil nuts should be ground as slurry.

Analytical method – performance based (see Table 2)

Decision rule – If the aflatoxin test result is less than or equal to 10 µg/kg total aflatoxin in both test samples, then accept the lot. Otherwise, reject the lot.

The operating characteristic curve describing the performance of the sampling plan for the four ready-to-eat treenuts is shown in Annex II.

11. To assist member countries implement these two Codex sampling plans, sample selection methods, sample preparation methods, and analytical methods required to quantify aflatoxin in laboratory samples taken from bulk treenut lots are described in the following sections.

ANNEX II

Proposed Sampling Plans

(Draft Standard for Certain Canned Vegetables and Draft Standard for Jams and Jellies)

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

ANNEX III
SAMPLING PLANS FOR CHILI SAUCE

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