CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Item 2 and 3 **MAS/38 CRD/2**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON METHODS OF ANALYSIS SAMPLING

38th Session
Budapest, Hungary, 8 -12 May 2017

REPORT OF THE PWG ON ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING

The PWG met on Sunday, 7 May 2017. The PWG considered matters referred for action by CCMAS identified in CX/MAS 17/38/2 (rev) and matters for endorsement in CX/MAS 17/38/3.

The PWG had the following discussion and made recommendations presented in the Appendix.

COMMITTEE ON PROCESSED FRUITS AND VEGETABLES (CCPFV28)

Methods of analysis for quick frozen vegetables

Method for Free Fatty Acids is not recommended for endorsement because the methods suggested are for fats and oils and not for foods. A method for fat extraction is necessary prior to the use of the suggested methods.

The PWG did not recommend endorsement of the sampling plans since the values in the table did not correspond to those recommended in the *General Guidelines on Sampling* (CAC/GL 50-2004). It was unclear whether the attributes sampling plan actually applied to attributes and not to characteristics that might be described as variable and requested CCPFV to reconsider the values in line with CAC/GL 50-2004.
Based on the request of CCPFV, CCMAS will need to develop plans for review at CCMAS39.

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA20)

Methods of analysis for laver products

The PWG recommends endorsement of the method for moisture AOAC 925.45B as Type IV.

There was discussion concerning the methods (AOCS Cd 3d-63, EN ISO 660, NMKL 38). All of these methods are identical and identified as Type I for other commodities in CODEX STAN 234. However these methods are appropriate for extracts and not for the determination of acid values in solid foods.

The regional standard does contain an extraction method, but there is no validation data available for this procedure. Based on the provisions listed, the lack of data on the extraction method, the current Typing of the analytical methods, the PWG could not reach consensus on the methods and Typing.

One suggestion was to divide the extraction and analytical method, but without a provision for extraction this decision was not considered appropriate by all PWG participants.

Sampling plan

The PWG did not recommend endorse the sampling plans since the values in the table did not correspond to those recommended in the *General Guidelines on Sampling* (CAC/GL 50-2004). It was unclear whether the attributes sampling plan actually applied to attributes and not to characteristics that might be described as variable. In conjunction with the development of other sampling plans, CCMAS will need to develop plans for review at CCMAS39.

COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU38)

Methods of analysis for infant formula

The PWG agreed to recommend the retyping of AOAC 967.21 to Type III.

Chromium, molybdenum, selenium
The PWG recommended endorsement of the new methods for chromium, molybdenum and selenium (AOAC 2011.19 | ISO 20649 | IDF 235) as Type II and retained and retyped (where necessary) the older methods as Type III. Concerns of Japan were noted, specifically that the older methods do not meet the sensitivity requirements necessary for the determination of analytes at the minimum levels stated in CODEX STAN 72-1981.

Vitamin B12, Myo-inositol and Vitamin E
The PWG noted the communication from CCNFSDU on the methods for Vitamin B12, Myo-inositol, Vitamin E and also noted that these methods had been endorsed at CCMAS37 and no further action was needed.

Provision for 'total fatty acid profile'
The PWG noted that with respect to the request from CCMAS37 to CCNFSDU on harmonizing the provision associated with method AOAC 2012.13, CCNFSDU responded with "The Committee (CCNFSDU) requested that the provision be retained as "total fatty acid" profile to maintain consistency with the term used in CODEX STAN 72-1981. Based on the interventions of a number of delegates the PWG agreed to again request, with a clearer description of the problem, that CCNFSDU reconsider harmonization of the provision and that the method would not be forwarded to the CAC for adoption until CCMAS received a response to this request.

TFA
PWG considered the request from CCNFSDU on the methods trans fat. The 'additional information' for determination of TFA provided in the background information document for the PWG was introduced followed by discussion about range of TFA compounds to be quantitated. To respond to CCNFSDU in a coherent and unified manner may require the development of a discussion paper, but as an initial step CCMAS would provide this 'Additional information' table along with the journal reference where the data was sourced. Then if further information is required by CCNFSDU then CCMAS can initiate the discussion paper on the topic.
Reference:

FAO/WHO COORDINATING COMMITTEE FOR AFRICA (CCAFRICA22)

Methods of analysis for unrefined shea butter

Methods were recommended for endorsement with the exception of the Lead, Arsenic and Iron because

there are no provisions in the standard. There were discussions about the use of General Methods for these provisions, but methods were not reviewed, typed or endorsed.

COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH3)

Methods of analysis and sampling plans for cumin

Moisture
With respect to Moisture for Cumin and Thyme, there had been no feedback from CCSCH regarding whether the provision should be 'water ' or 'moisture'; thus we have to assume it is to be retained as Moisture and therefore Type I, and delegations suggested that in this case the ISO-939 be reinstated. This recommendation received no objections.

Extraneous matter / foreign matter
There was a query about the 'Extraneous matter' and 'Foreign matter' having the same method, and clarification provided that as a 'visual inspection' the both contaminant are identified and measured at the same time.

Apart from the 'Moisture'' and 'Mammalian excreta' provision all other provision methods had been recommended for endorsement at CCMAS37 thus no further consideration was required. With the 'Mammalian excreta'; the method 'Visual examination (for whole)' and 'Enzymatic Detection method (For ground)' provided were recommended for both to be a Type IV method, as the Phosphatase was not necessarily only selective to 'mammalian excreta' and could be derived from other microbiological contamination and the method had only been validated in ground pepper.

Black, white and green pepper

With respect to black, white and green pepper the multiple principles for single ISO methods (959-1 and 959-2) were discussed.

For nonvolatile ether extract the AOAC method was removed because it is a dichloromethane extract so does not meet the provision.

Where we have 2 Type I methods, there was confirmation that the methods are identical.

The PWG did not recommend endorsement of the sampling plans since the values in the table did not correspond to those recommended in the *General Guidelines on Sampling* (CAC/GL 50-2004). It was unclear whether the attributes sampling plan actually applied to attributes and not to characteristics that might be described as variable. In conjunction with the development of other sampling plans, CCMAS will need to develop plans for review at CCMAS39.

COMMITTEE ON FATS AND OILS (CCFO25)

Method of analysis for fish oils

Three methods identical methods for p-anisidine are listed as Type I.
The NMR method for phospholipids was Typed as IV, because the validation is only on Krill Oil.
Three methods for triglycerides were listed and there was a question if they were appropriate for the provision. It was confirmed that they are correct, but a Type II method was not selected by the PWG. A confirmation is needed that the European Pharmacopeia method is referenced correctly.

Validation studies for the amended version of AOAC 963.15

The PWG reviewed CX/MAS 17/38/3 Add. 1, which reports on the multilaboratory validation data for the extension of AOAC 963.15 to the analysis of tempe. The validation was accepted.

Appendix

COMMITTEE ON PROCESSED FRUITS AND VEGETABLES (CCPFV28)

Methods of analysis for quick frozen vegetables

Product	Provision	Method	Principle	Type
Quick frozen fruits and vegetables	Thawing procedure	Method CAC/RM 32 to be placed in STAN 234	Thawing	I
Quick frozen fruits and vegetables: Vegetables	Cooking procedure	Method CAC/RM 33 to be placed in CODEX STAN 234	Cooking	I
Quick frozen fruits and vegetables (non-glazed)	Net weight	AOAC 963.26	Weighing	I
Quick frozen peas	Solids, alcohol insoluble	Method CAC/RM 35 to be placed in STAN 234	Gravimetry	I
Quick frozen green and wax beans	Tough strings	Method CAC/RM 39 to be placed in STAN 234	Stretching	I
Quick frozen fruits and vegetables: Berries, Whole kernel corn and Corn- on-the-cob	Soluble solids, total	AOAC 932.12	Refractometry	I
Quick frozen fruits and vegetables: Berries, leek and carrot	Mineral impurities	AOAC 971.33	Gravimetry	I
Quick frozen fruits and vegetables: Peaches and berries	Drained fruit/drained berries	AOAC 953.15	Draining	I
Quick frozen spinach	Dry matter, Sodium chloride-free	Move to STAN 234	Weighing	I
Quick frozen French fried potatoes	Moisture	AOAC 984.25	Gravimetry (convection oven)	I
Quick frozen French fried potatoes	Free fatty acid	~~ISO 660:2009; or AOCS Cd 3d-63 (09)~~	~~Titrimetry~~	~~I~~

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA20)

Methods of analysis for laver products

Provision	Method	Principle	Type
Moisture content	AOAC 925.45B	Gravimetry, drying at atmospheric pressure	IV
Acid value	AOCS Cd 3d-63 EN ISO 660 NMKL 38	Titrimetry	Typing not determined, See text

COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU38)

Methods of analysis for infant formula

Provision	Method	Principle	Type
Vitamin C	**AOAC 2012.22 | ISO/DIS 20635**	**HPLC-UV**	**II**
Chromium, selenium, molybdenum	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	**II**

Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	AOAC 2011.19 | ISO 20649 |IDF 235	ICP-MS	**II**
Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	EN 14082	Graphite furnace atomic absorption after dry ashing	**III**
Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	EN 14083	Graphite furnace AAS after pressure digestion	III
Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	AOAC 2006.03	ICP emission spectroscopy	III
Infant formula	Molybdenum (Section B of CODEX STAN 72-1981 only)	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	**II**
Infant formula	Molybdenum (Section B of CODEX STAN 72-1981 only)	EN 14083	Graphite furnace AAS after pressure digestion	**III**
Infant formula	Molybdenum (Section B of CODEX STAN 72-1981 only)	AOAC 2006.03	ICP emission spectroscopy	III
Infant formula	Selenium	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	**II**
Infant formula	Selenium	AOAC 996.16 or AOAC 996.17	Continuous hydride generation Flame atomic absorption spectrometry (HGAAS)	III
Infant formula	Selenium	EN 14627	Hydride generation atomic absorption spectrometry (HGAAS)	**III**
Infant formula	Selenium	AOAC 2006.03	ICP emission spectroscopy	III

FAO/WHO COORDINATING COMMITTEE FOR AFRICA (CCAFRICA22)

Methods of analysis for unrefined shea butter

Provision	Method	**Type**
Moisture content	~~AOAC 920.116 for dairy butter~~ ~~IUPAC 2.60~~ EN ISO 662~~:1998~~	I
Free fatty acid content: acid value and acidity	EN ISO 660~~:1996~~ ~~IUPAC 2.201~~ AOCS Cd 3d-63	I
Relative density	~~IUPAC 2.101~~ AOCS Cc 10c-95, EN ISO 6883	I
Saponification value	EN ISO 3657~~:1998 (revised 1992)~~ ~~IUPAC 2.202~~ AOCS Cd 3d-25	I
Iodine value	AOAC 993.20, EN ISO 3961~~:1999~~ AOCS Cd 1d-92 NMKL 39	I
Peroxide value	AOCS Cd 8b-90 ~~IUPAC 2501~~ EN ISO 3960~~: 2005~~ NMKL 158	I
Unsaponifiable matter	EN ISO 3596~~-1: 1996~~ ~~IUPAC 2.401~~ AOCS Ca 6a-40	I
Insoluble impurities content	EN ISO 663 ~~IUPAC 2604~~ AOCS Ca 3a-46	I
Melting point	EN ISO 6321~~:2002~~ AOCS Cc 3b-92	I
~~Lead~~	~~ISO 12193:1994~~ ~~AOAC 972.25~~ ~~AOAC 994.02~~ ~~IUPAC 2632 AOCS Ca 18c-91~~	
~~Arsenic~~	~~AOAC 952.13~~ ~~IUPAC 3136~~	

~~Iron~~	~~ISO 8294: 1994~~ ~~AOAC 990.05~~ ~~IUPAC 2631 AOCS Ca 18b-91~~	

COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH3)

Methods of analysis and sampling plans for cumin

Provision	Method	Principle	Type
Moisture	ISO 939~~:1980 - ISO 760:1978/ISO - AOAC 2001.12~~	Distillation	I
Total ash	ISO 928~~:1997~~	Gravimetry	I
Acid-insoluble ash	ISO 930~~:1997~~	Gravimetry	I
Volatile oils	EN ISO 6571~~:2008~~	Distillation / Volumetric	I
Extraneous vegetable matter ~~material -~~	EN ISO 927~~:2009~~	Visual examination / Gravimetry	I
Foreign matter	EN ISO 927~~:2009~~	Visual examination / Gravimetry	I
Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV
Mammalian excreta	Macroanalytical procedure manual USFDA technical bulletin V.39 B (for whole)	Visual examination	IV
	AOAC 993.27 (for ground)	Enzymatic Detection method	IV
Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV

COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH3)

Methods of analysis and sampling plans for thyme

Provision	Method	Principle	Type
Moisture	ISO 939~~:1980 ISO 760:1978/ISO AOAC 2001.12~~	Distillation	I
Total ash	ISO 928~~:1997~~	Gravimetry	I
Acid-insoluble ash	ISO 930~~:1997~~	Gravimetry	I
Volatile oils	EN ISO 6571~~:2008~~	Distillation / Volumetric	I
Extraneous vegtable matter ~~material~~	EN ISO 927~~:2009~~	Visual examination / Gravimetry	I
Foreign matter	EN ISO 927~~:2009~~	Visual examination / Gravimetry	I
Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV
Mammalian excreta	Macroanalytical procedure manual USFDA technical bulletin V.39 B (for whole)	Visual examination	IV
	AOAC 993.27 (for ground)	Enzymatic Detection method	IV
Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV

Methods of analysis and sampling plans for black, white and green pepper

Provision	**Method**	**Principle**	**Type**
Bulk density	ISO 959-1 Annex B~~:1998~~ (black) ISO 959-2 Annex A~~:1998~~ (white)	Gravimetry	IV
Light berries	ISO 959-1 Annex A (black)	Flotation	IV
Extraneous vegetable matter and foreign matter	EN ISO 927~~:2009~~	Visual examination	IV
Black berries	Physical separation and weighing ISO 959-2~~:1998~~	Visual examination	IV
Broken berries	Physical separation and weighing ISO 959-2~~:1998~~	Visual examination	IV
Mouldy berries	Macroanalytical procedure manual USFDA technical bulletin V.39 B	Visual examination	IV
Insect ~~defiled berries~~ damage	Macroanalytical procedure manual USFDA technical bulletin V.39 B	Visual examination	IV
Pinheads or broken berries	Physical separation and weighing ISO959-1~~:1998~~	Visual examination	IV
Mammalian ~~and/ or other~~ excreta	Macroanalytical procedure manual USFDA technical bulletin V.39 B (For Pepper Whole)	Visual examination(For whole pepper)	IV
Mammalian ~~and/ or other~~ excreta	AOAC 993.27 (for ground pepper)	Enzymatic Detection method (For ground pepper)	I
Moisture content	~~AOAC 986.21~~ ISO 939~~:1980~~	Distillation	I
Total ash	~~AOAC 941.12~~ ISO 928~~:1997~~	Gravimetry	I
Non-volatile ether extract	~~AOAC 940.29~~ ISO 1108	Soxhlet extraction	I
Volatile oils	~~AOAC 962.17~~ EN ISO 6571~~:2008~~	Distillation	I
Piperine content	~~AOAC 987.07~~ ISO 5564	Spectrophotometry	I
Acid- Insoluble ash	AOAC 941.12	Gravimetry	I

	ISO 930:~~1997~~		
Crude Fiber	AOAC 920.169 ISO 5498	Gravimetry	I

COMMITTEE ON FATS AND OILS (CCFO25)

Method of analysis for fish oils

Provisions	Method	Principle	Type
P-Anisidine value	European Pharmacopeia 2.5.36, AOCS Cd 18-90 ISO 3960	Spectrophotometry	I
Phospholipids	USP-FCC10 2S (Krill oil): Phosopholipids, Nuclear Magnetic Resonance, Appendix IIC	NMR Spectroscopy	IV
Triglycerides	USP 40-NF35 (Omega-3 Acid Triglycerides): Content of oligomers and partial glyceride;	HPLC-RI	III
	European Pharmacopoeia 01/2008/1352 (Omega3 acid triglycerides): Oligomers and partial glycerides	HPLC-RI	III
	AOCS Cd 11d-96	HPLC-ELSD	III

Validation studies for the amended version of AOAC 963.15

	Provisions	Method	Principle	Type
Tempe	Lipid Content	~~AOAC 983.23~~ AOAC 963.15	Gravimetry (Soxhlet Extraction)	I