Agenda and Background Information

Physical Working Group on Endorsement CCMAS38

7 May 2017, Budapest, Hungary

This document is intended to provide both an agenda for the Physical Working Group on Endorsement and some additional background information on the items before the committee. It was developed in hope of reducing the back and forth reading between multiple documents. It closely follows CX/MAS 17/38/2-Rev and CX/MAS 17/38/3, but groups items by committee and not by Matters Referred or Endorsement. It recreates many, but not all, of the tables in CX/MAS 17/38/2-Rev and 17/38/3, therefore it should be used in conjunction with these other documents. The BACKGROUND information is taken from previous Committee Reports or Agenda Items and the source is stated prior to presentation of the information. Additional information is also presented when the Chairs felt it would be helpful in the discussion.

From CX/MAS 17/38/2-Rev Matters Referred

COMMITTEE ON PROCESSED FRUITS AND VEGETABLES (CCPFV28)

Standard for Ginseng Products – sampling plans7

10. CCPFV28 reconsidered the sampling plan in view of the request from CCMAS368 and was of the opinion that the chemical and physical characteristics were quality factors for which an attribute sampling plan would be appropriate. The characteristics were either conforming or non-conforming in relation to the limit noted.

11. CCPFV28 agreed to retain the current sampling plans in the Standard for Ginseng Products

(CODEX STAN 321-2015) and that, should a variable sampling plan be required, CCMAS could develop a suitable proposal which would meet the requirements of the *Guidelines on Sampling* (CAC/GL 50-2004).

12. The Committee **is invited to endorse** the sampling plan (CX/MAS 17/38/2-Rev Appendix I). This matter will be considered by the PWG on methods of analysis and sampling (endorsement working group).

BACKGROUND

From REP15/MAS

Committee on Processed Fruits and Vegetables (CCPFV)

The Committee:

- agreed to replace the CAC/RM 46-1972 (method for fill of glass containers) with ISO 8106 (Glass containers – determination of capacity by gravimetric methods) and to apply this change to all relevant standards on processed fruits and vegetables (para. 12); and

- did not endorse the sampling plans in the Standard for Ginseng and Ginseng Products (para.16).

16. The Committee did not 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 and requested CCPFV to reconsider the values in line with CAC/GL 50-2004.

Methods of analysis for quick frozen vegetables

13. CCPFV considered a proposed list of methods of analysis for quick frozen vegetables with the possible replacement of Codex Recommended Methods (CAC/RMs) and agreed to:

- recommend AOAC 940.28b and IUPAC 2.201 as methods for determination of free fatty acid in quick frozen French fried potatoes;
- replace CAC/RM with more updated internationally validated methods; and
- to request CCMAS to assist in the identification of equivalent internationally validated methods for other CAC/RMs that the Committee could not identify at its present session.

CODEX COMMITTEE ON PROCESSED FRUITS AND VEGETABLES (CCPFV28)

NOTE: The Committee agreed to forward the proposed draft annexes for certain quick frozen vegetables (for inclusion in the *Standard for Quick Frozen Vegetables* (CODEX STAN 320-2015) to the Commission for adoption at Step 5/8.

2. The Committee **is invited to endorse** the methods of analysis and sampling plan in CX/MAS 17/38/3 Appendix I.

From CX/MAS 17/38/2-Rev Matters Referred

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

Methods for trans fatty acids

15. CCNSDU38 agreed to request CCMAS to review if the three methods (see *CX/MAS 16/37/2* Appendix II) are applicable to determine TFA as defined in both the *Guidelines on Nutrition Labelling* (CAC/GL 2-1985)12 and the WHO definition – *at least one double bond in the trans configuration – at the level of 1 g per 100 g of fat*. .

16. The Committee **is invited to consider** the request from CCNFSDU. This matter will be considered by the PWG on methods of analysis and sampling (endorsement working group).

BACKGROUND

From CCMAS 2015

From CX/MAS 15/36/2

Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU)

Trans fatty acids

19. CCNFSDU36 agreed to request advice from CCMAS on the lowest level of TFAs that current analytical methods can accurately detect as well as consistently reproduce.

From REP15/MAS

Committee on Nutrition and Foods for Special Dietary Uses

The Committee:

- noted that it would be difficult to provide information on the lowest level of TFAs that current analytical methods can accurately determine as well as consistently reproduce as the levels would depend on the matrix of the product (para. 31);

Trans fatty acids

30. The Committee considered the request from CCNFSDU on the lowest level of TFAs that current analytical methods can accurately determine as well as consistently reproduce.

31. The Committee noted that it would be difficult to provide such information to CCNFSDU, as the levels obtained would depend on the matrix of the product. It would be more appropriate for CCNFSDU to provide CCMAS with the levels for total TFA and the matrix to which the level applies. The Committee also pointed out that it would not be possible to establish a single level for TFA for all foods, but that CCNFSDU would have to develop separate levels for different commodities.

32. The Committee noted that in-depth analysis in some matrices had been carried out by ISO, IDF and AOAC, and gave some results as summarized in CRD 16. The method would be published by end of 2015.

33. The Observer from AOCS reiterated its concern previously expressed at CCNFSDU, that low levels of *trans* fatty acids cannot be routinely determined by the average laboratory with any high degree of reproducibility. This situation might lead to confusion in the marketplace and in general trade where products might be deemed to be *"trans-free"* by one laboratory and above the threshold for this claim in another.

Additional Information: Determination of TFA in Collaborative Studies for each method/matrix

Product	Method		
	ISO 16958/IDF 231/ AOAC 2012.13	AOCS Ce 1h-05 and AOAC 996.06	AOCS Ce 1j-07 and Ce 2b-11/Ce 2c-11
Dairy and ruminant products/fats	**TFA Range: 0.17–5.06 g/100 g (n=5)**: • Cheese (extracted fat), 5.06 g/100 g • Butter, 4.24 g/100 g • Cream, 1.62 g/100 g • Milk powder, 1.03 g/100 g • Liquid milk, 0.17 g/100 g	Not validated	**TFA Range: 0.32–7.27% of total fatty acids (n=5):** • Cheese powder, 7.27% • Anhydrous milk fat, 5.11% • Butter, 2.49% • Evaporated milk, 0.33% • Yogurt, 0.32%
Adult nutritionals	**TFA Range: 0.006–0.010 g/100 g (n=3):** • High protein RTF, 0.009 g/100 g • High fat RTF, 0.010 g/100 g • Milk-based powder, 0.006 g/100 g	Not validated	Not validated
Infant formula	**TFA Range: 0.010–0.073 g/100 g (n=4):** • Milk-based powder, 0.073 g/100 g • Milk-based RTF, 0.027 g/100 g • Milk-based powder, 0.012 g/100 g • Soy-based powder, 0.010 g/100 g	Samples unknown	**TFA Range: 0.15% of total fatty acids (n=1)** • DHA/EPA-fortified infant formula, 0.15%
Samples containing vegetable oils	Not validated	**TFA Range: 0.06–45.01% of total fatty acids (n=10):** • Vegetable shortening, 45.01% • Canola oil, 26.27% and 26.55% • Margarine, 11.62% • Hydrogenated lard, 1.00% • Lard, 0.90% • Sunflower oil, 0.17% • Coconut oil, 0.10% and 0.11% • Cocoa butter, 0.06%	Not validated
Samples containing marine oils or other oils with long chain polyunsaturated fatty acids	Not validated	Not validated	**TFA Range: 0.00–0.68% of total fatty acids (n=2):** • Encapsulated DHA/EPA, 0.68% • DHA/EPA-fortified orange juice, 0.00%
Samples with unknown fat sources	Not validated	Not validated	**TFA Range: 0.00–0.68% of total fatty acids (n=14):** • Tallow, 7.14% • Chocolate-cake mix, 0.90% • Whole-egg powder, 0.43% • Frozen cheese pizza, 0.37% • Extruded dog food, 0.31% • Creamy ranch-dressing, 0.24% • Potato chips, 0.22%

			• Peanut butter, 0.06% • Oatmeal cookie, 0.05% • Canned cat food, 0.05% • Full-fat soy flour flakes, 0.02% • Dry cereal fortified with flax, 0.00% • Horse feed, 0.00% • Gamebird feed, 0.00%

From CX/MAS 17/38/2-Rev Matters Referred

Methods of analysis for provisions in the Standard for Infant Formula and Formulas for Special Medical Purposes intended for Infants (CODEX STAN 72-1981)

17. CCNSFDU38 considered the matters referred from CCMAS37 and took the following decisions

chromium, selenium and molybdenum: *review of criteria* .

18. CCNFSDU38 agreed to:

i. inform CCMAS that it did not support using of criteria approach because:

a. A general or single conversion factor to convert ug/100kCal to ug/g should not be used, as the energy density of infant formula varies across products; and

b. None of the current methods in CODEX STAN 234-19991, nor the newer methods AOAC 2011.19| ISO 20649 | IDF 235 meet the criteria

ii. CCNFSDU38 agreed to request that CCMAS reconsider the method for chromium, selenium and molybdenum as Type II in light of published validation data measuring the minimum level for chromium, selenium and molybdenum in CODEX STAN 72-1981;

iii. Inform CCMAS that other methods for chromium, selenium and molybdenum other than the AOAC method were still fit for purpose and to reconsider their classification, if necessary.

BACKGROUND

From REP16/MAS

Chromium, selenium and molybdenum

30. The Committee did not endorse the methods as Type II as proposed by CCNSFDU as there were concerns that these methods (requiring expensive instrumentation) were recommended for dispute settlement. The current methods in CODEX STAN 234-1999 were considered by some delegations as equally suitable for use. It was clarified that the newer methods had been extensively validated specifically for infant formula, were more sensitive, precise and necessary for use to ensure the nutritional safety of the products. In order to provide flexibility to countries in the selection of methods, it was agreed to recommend numeric values for method criteria for the determination of chromium, selenium, and molybdenum for consideration by CCNFSDU.

31. The Committee noted that the method criteria developed (Appendix II) indicated that none of the current methods in CODEX STAN 234-1999, nor the newer proposed methods would meet the criteria, although the newer AOAC/ISO/IDF methods were closest to meeting the performance criteria. The Committee agreed to request CCNFSDU to review the numeric values for method criteria, specifically the minimum limit in column 2, and to inform CCMAS whether it had interpreted the limits in the related provisions correctly. If the values are correct then CCNFSDU should note that none of the methods (newly endorsed or existing) meet the numeric values for method criteria. If the values are incorrect then CCNFSDU should provide CCMAS advice on the correct values and how to proceed.

32. While CCNFSDU reviews the numeric values for method criteria, CCMAS has endorsed the proposed methods as Type III and maintained the typing of the existing methods in CODEX STAN 234-1999.

Current in CODEX STAN 234-1999 (2016)

Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	EN 14082	Graphite furnace atomic absorption after dry ashing	II
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	Chromium (Section B of CODEX STAN 72-1981 only)	AOAC 2011.19 | ISO 20649 |IDF 235	ICP-MS	III
Infant formula	Molybdenum (Section B of CODEX STAN 72-1981 only)	EN 14083	Graphite furnace AAS after pressure digestion	II
Infant formula	Molybdenum (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	III
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)	II
Infant formula	Selenium	AOAC 2006.03	ICP emission spectroscopy	III
Infant formula	Selenium	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	III

From CX/MAS 17/38/2-Rev Matters Referred

Methods of analysis for provisions in the Standard for Infant Formula and Formulas for Special Medical Purposes intended for Infants (CODEX STAN 72-1981)13***14***

Vitamin B12

19. CCNFSDU38 confirmed that the existing method, AOAC 989.23, is fit for purpose.

Total fatty acid profile

20. CCNSDU38 agreed to inform CCMAS that the current method, AOAC 996.06, is fit for purpose and agreed with its classification as Type III. Method AOAC 2012.13 endorsed by CCMAS should be sent to CAC for adoption. The Committee requested that the provision be retained as "total fatty acid" profile to maintain consistency with the term used in CODEX STAN 72-1981.

Myo-inositol and Vitamin E

21. CCNFSDU38 confirmed that the definition and scope of the methods harmonize and should be sent to CAC for adoption.

22. The Committee **is invited to consider** the information and to take the appropriate action. These replies will be considered by the PWG on methods of analysis and sampling (endorsement working group).

BACKGROUND

From REP16/MAS

Determination of Vitamin B12

33. The Committee endorsed the method as Type II, and agreed to request CCNFSDU to clarify whether the existing method in CODEX STAN 234-1999 is still fit for purpose, and if so, this method would become Type III.

Determination of myo-inositol

35. The Committee agreed to request CCNSFDU to confirm that the AOAC 2011.18 and ISO 20637 determine the forms to be measured according to CODEX STAN 72-1981 for myo-inositol. The AOAC 2011.18 and ISO 20637 determine free and bound myo-inositol as phosphatidylinositol, but it is unclear if this is the definition (inclusion of free and bound) in CODEX STAN 72-1981. Provided that the definition and the scope of the methods harmonize, CCMAS recommended endorsement of AOAC 2011.18 and ISO 20637 as Type II. **(It does not need to come back for re-endorsement by CCMAS.)** *Bold added*

Determination of Vitamin E

37. The Committee agreed to request CCNSFDU to confirm that the scope of AOAC 2012.10 and ISO 20633 is in line with the provision for the isomers of Vitamin E in the CODEX STAN 72-1981. The methods do not discriminate both d and dl-alpha-tocopherol, neither do the currently endorsed methods (AOAC 992.03 and EN 12822) and Vitamin E is listed in the *Advisory lists of nutrient compounds for use in foods for special dietary uses intended for infants and young children* (CAC/GL GL10-1979), with sources listed as D-alpha-Tocopherol, DL-alpha-Tocopherol, D-alpha-Tocopheryl acetate, DL-alpha-Tocopheryl acetate, D-alpha-Tocopheryl acid succinate, DL-alpha-Tocopheryl acid succinate, DL-alpha-Tocopheryl polyethylene glycol 1000 succinate. However in CODEX STAN 72-1981 the footnote only refers to d-alpha-tocopherol. Provided that the provision and the scope of the methods harmonize, CCMAS recommends endorsement of AOAC 2012.10 and ISO 20633 as Type II. **(It does not need to come back for re-endorsement by CCMAS.)** *Bold added*

Determination of fatty acid profile

38. The Committee noted that the provisions in CODEX STAN 72-1981 are total fat, linoleic acid, and α-linolenic acid and that the scope of the AOAC 2012.13 and ISO 16958|IDF 231 are appropriate for those provisions. The Committee recommended to change the wording of the provision (Appendix II), endorsed the method as Type II and further recommended the existing method (AOAC 996.06) be changed to Type III.

From CX/MAS 17/38/3 Endorsement

Methods of analysis for provisions in the Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants (CODEX STAN 72-1981)

4. The Committee agreed to submit the method for determination of Vitamin C in infant formula for endorsement and inclusion in the *Recommended Methods of Analysis and Sampling* (CODEX STAN 234-1999) as this method reflected the most recent scientific methods of analysis for nutrients in infant formula and were fully validated for these products (Appendix V, Part I). CCMAS is requested to remove or reclassify methods that are not validated for infant formula in CODEX STAN 234-1999 that might be replaced by the aforementioned method.

Background:

Multilaboratory validation: Campos-Gimenez et al. Vitamin C in Infant Formula and Adult/Pediatric Nutritional Formula by Ultra-Performance Liquid Chromatography with Ultraviolet Detection: First Action 2012.22. J AOAC International Vol. 96, No. 5, 2013.

From CODEX STAN 72-1981 (2016)

Vitamin C[15] Unit	Minimum	Maximum	GUL
mg/100 kcal	10	-	70[16]
mg/100 kJ	2.5	-	17[16]

15) Expressed as ascorbic acid
16) This GUL has been set to account for possible high losses over shelf-life in liquid formulas; for powdered products lower upper levels should be aimed for.

From CX/MAS 17/38/2-Rev Matters Referred and CX/MAS 17/38/3 Endorsement

COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH3)

Methods of analysis for cumin and thyme15

23. CCSCH agreed to request CCMAS to propose alternative equivalent analytical methods that could be used to those already typed. *(See CX/MAS 17/38/3 Appendix V for methods of analysis and sampling plans)*

From CX/MAS 17/38/3 Endorsement

Methods of analysis for cumin5 and thyme6 and BWG pepper

NOTE: The Committee agreed to forward the draft standards for cumin and thyme to the Commission for adoption at Step 8; and the proposed draft Standard for Black, White and Green (BWG) Pepper for adoption at Step 5/8; and the methods of analysis and sampling plans for endorsement and inclusion in CODEX STAN 234-1999.

7. The methods of analysis for cumin and thyme were previously considered by CCMAS37 and proposals made for consideration by CCSCH.

8. The Committee **is invited to endorse** the methods of analysis and sampling plans in CX/MAS 17/38/3 Appendix V. Noting the 'Mammalian Excreta' and 'Mould Damage' provision methods for Cumin; and the 'Mammalian Excreta' provision method were not considered at 37CCMAS

BACKGROUND

From REP16/MAS

Committee on Spices and Culinary Herbs (CCSCH)

Proposed draft Standards for Cumin and Thyme

Determination of Moisture

26. An editorial correction was made to the suggested ISO method: ISO 939:1980. However, the Committee recommended the removal of ISO 939:1980 due to the complexity of the method and use of hazardous reagents. The Committee agreed to recommend the Karl Fischer titration methods: AOAC 2001.12 and ISO 760: 1978, but delayed typing the method. It is unclear if the provision should be water or moisture. If the provision is water then both the AOAC and ISO methods may be listed in the standard, with one designated as Type II and one as Type III. CCSCH should recognize that the ISO method has not been collaboratively studied, while the AOAC method is collaboratively studied, but not for cumin. If the two methods provide equivalent results, CCSCH should recommend which of the two methods should be considered Type II. If the provision should be moisture as listed, then only one method may appear in the standard as Type I, and CCSCH should recommend which method.

Determination of total ash, acid insoluble ash, volatile oils, extraneous matter and foreign matter

27. The Committee agreed to recommend the ISO methods as Type I recognizing that there can only be one Type I method. In cases where alternative methods were proposed by CCSCH (i.e. AOAC methods and/or the ASTA methods), these were deleted, as it could not be confirmed that they were identical to the ISO methods. The Committee agreed to recommend to change the provision “extraneous vegetable material” to “extraneous matter” for harmonization with the corresponding ISO method.

Determination of Insect Damage (for cumin and thyme) and mould damage (for thyme)

28. The Committee agreed to recommend endorsement of the methods as Type IV as the methods were not collaboratively studied.

BACKGROUND
From REP16/MAS Appendix II
A. SPICES AND CULINARY HERBS

STANDARD FOR CUMIN – METHOD OF ANALYSIS

Commodity	Provision	Method	Principle	Type
Cumin	Moisture	ISO 760:1978 AOAC 2001.12	Titration	To be determined
Cumin	Total ash	ISO 928:1997	Gravimetry	I
Cumin	Acid-insoluble ash	ISO 930:1997	Gravimetry	I
Cumin	Volatile oils	ISO 6571:2008	Distillation / Volumetric	I
Cumin	Extraneous matter	ISO 927:2009	Visual examination / Gravimetry	I
Cumin	Foreign matter	ISO 927:2009	Visual examination / Gravimetry	I
Cumin	Insect damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)	Visual examination	IV

STANDARD FOR DRIED THYME - METHODS OF ANALYSIS

Commodity	Provision	Method	Principle	Type
Dried Thyme	Moisture	ISO 760:1978 AOAC 2001.12	Titration	To be determined
Dried Thyme	Total ash	ISO 928:1997	Gravimetry	I
Dried Thyme	Acid-insoluble ash	ISO 930:1997	Gravimetry	I
Dried Thyme	Volatile oils	ISO 6571:2008	Distillation / Volumetric	I
Dried Thyme	Extraneous matter	ISO 927:2009	Visual examination / Gravimetry	I
Dried Thyme	Foreign matter	ISO 927:2009	Visual examination / Gravimetry	I
Dried Thyme	Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)	Visual examination	IV
Dried Thyme	Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)	Visual examination	IV

From CX/MAS 17/38/3

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

Provision	Method	Principle	Type
Bulk density	ISO 959-1:1998 ISO 959-2:1998	Separation by density	IV
Light berries	ISO 959-1:1998	Flotation	IV
Extraneous vegetable matter and foreign matter	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	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	i) Macroanalytical procedure manual USFDA technical bulletin V.39 B (For Pepper Whole) ii) AOAC 993.27 (for ground pepper)	Visual examination(For whole pepper) Enzymatic Detection method (For ground pepper)	IV III
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 ISO 6571:2008	Distillation	I
Piperine content	AOAC 987.07 ISO 5564	Spectrophotometry	I
Acid- Insoluble ash	AOAC 941.12 ISO930:1997	Gravimetry	I
Crude Fiber	AOAC 920.169 ISO 5498	Gravimetry	I

From CX/MAS 17/38/3 Endorsement

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA20)

NOTE: The Committee agreed to forward the proposed draft Standard for Laver products for adoption at Step 5/8 and the methods of analysis and sampling plans.

3. The Committee **is invited to endorse** the methods of analysis and sampling plans in CX/MAS 17/38/3 Appendix II.

Provision	Method	Principle	Type
Moisture content	AOAC 925.45	Gravimetry, drying at atmospheric pressure	IV
Acid value	AOCS Cd 3d-63	Titrimetry	I

From CX/MAS 17/38/3 Endorsement

FAO/WHO COORDINATING COMMITTEE FOR AFRICA (CCAFRICA22)4

Method of Analysis for shea butter

NOTE: The Committee agreed to forward the proposed draft Standard for Unrefined Shea Butter to the Commission for adoption at Step 5/8 and the methods of analysis for endorsement and inclusion in CODEX STAN 234-1999.

6. The Committee **is invited to endorse** the methods of analysis in CX/MAS 17/38/3 Appendix IV.

Methods of analysis for unrefined shea butter

Provision	Method
Moisture content	AOAC 920.116 IUPAC 2.60 ISO 662:1998
Free fatty acid content: acid value and acidity	ISO 660:1996 IUPAC 2.201
Relative density	IUPAC 2.101
Saponification value	ISO 3657:1998 (revised 1992) IUPAC 2.202
Iodine value	AOAC 925.56 ISO 3961:1999
Peroxide value	AOCS cd. 8b – 90 IUPAC 2501 ISO 3960: 2005
Unsaponifiable matter	ISO 3596-1: 1996 IUPAC 2.401
Insoluble impurities content	ISO 663: 2000 IUPAC 2604
Melting point	ISO 6321:2002
Lead	ISO 12193:1994 AOAC 972.25 AOAC 994.02 IUPAC 2632
Arsenic	AOAC 952.13 IUPAC 3136
Iron	ISO 8294: 1994 AOAC 990.05 IUPAC 2631

From CX/MAS 17/38/3 Endorsement

COMMITTEE ON FATS AND OILS (CCFO25)

Methods of analysis for fish oils

NOTE: The Committee agreed to forward the draft Standard for Fish Oils for adoption at Step 8 and the methods of analysis and sampling plans for inclusion in CODEX STAN 234. CCMAS36 already endorsed the sampling plan and several methods of analysis for fish oils except for the method for determination of phospholipids pending clarification from CCFO9. The reply from CCFO is provided in CX/MAS 17/38/2-Rev.

9. The Committee **is invited to endorse** the methods of analysis in CX/MAS 17/38/3 Appendix VI.

Provisions	Method
P-Anisidine value	European Pharmacopeia 2.5.36
Phospholipids	USP-FCC10 1S (Krill oil): Content of total phospholipids by qualitative and quantitative NMR Analysis
Triglycerides	USP 38 (Omega-3 Acid Triglycerides): Content of oligomers and partial glyceride; European Pharmacopoeia 01/2008/1352 (Omega3 acid triglycerides): Oligomers and partial glycerides; AOCS Cd 11d-96 (Mono- and diglycerides determination by HPLC-ELSD)

BACKGROUND

FROM REP15/MAS

Standard for Fish Oils

Determination of fatty acid composition

21. The Committee endorsed the methods and noted that ISO 5508 would be superseded by ISO 12966-2, but that this method was maintained because it was still effective and could be used until it was withdrawn.

Determination of arsenic and lead

22. The Committee recommended that criteria be developed once the ML for arsenic and lead were finalised. The Committee noted that a similar approach taken for the performance criteria for natural mineral waters provisions could be taken, i.e. provide performance criteria and examples of methods that meet the criteria for inclusion in CODEX STAN 234.

Determination of acid value

23. In addition to the proposal of the working group, the Committee also agreed to endorse the NMKL 38 and the European Pharmacopoeia 2.5.1 as Type I methods, as these methods were identical to the AOCS methods.

Determination of Peroxide Value

24. The Committee noted that European Pharmacopoeia 2.5.5 had two parts, which required the use of different reagents and endorsed the European Pharmacopoeia 2.5.5 (Part B iso-octane as solvent), while maintaining the recommendation of the working group not to endorse the EP method that used chloroform as a reagent. The Committee also endorsed the NMKL 158 as it was applicable and identical to the other methods endorsed.

Determination of Vitamin D

25. The Committee considered whether to endorse the European Pharmacopoeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29, which only determined Vitamin D3, while other methods determined D2 and D3. The committee was informed that for fish oils Vitamin D3 was the analyte of concern. It was noted that either Vitamin D3 or Vitamin D2 were determined, but could not be carried out together, and therefore all three methods submitted were fit for purpose and were endorsed.

Determination of Phospholipids

26. The Committee noted that the provision in the Standard referred to phospholipids and that there were currently no methods for the determination of phospholipids, but for phosphorous. The Committee therefore did not endorse the method, as a conversion factor was needed to convert the phosphorous to phospholipids. The Committee agreed to request CCFO to establish a conversion factor for inclusion in the Standard or to indicate in the Standard that the provision applied to phospholipids expressed as phosphorous before the methods could be endorsed.

From REP17/FO-Rev

Section 8 Methods of Analysis

Determination of phospholipids

24. The Committee noted that conversion factors for the determination of phospholipids from phosphorus are being used in practice. However, the PWG was unable to recommend a single suitable conversion factor for fish oils and had instead recommended an NMR-based method for the determination of phospholipids.

25. AOCS informed the Committee that they were considering the validation of the method which might be adopted in the near future. The observer also proposed to look into the matter of conversion factors or to request CCMAS to recommend such a factor to allow the methods previously submitted for endorsement7 to be used.

26. The Codex Secretariat recalled that CCMAS had previously indicated that identification of conversion factors was within the domain of commodity committees, but that a request could still be made if this would facilitate the endorsement of the previously submitted methods.

Determination of p-anisidine and determination of triglycerides

27. The Committee noted a proposal of an Observer for an additional method for the determination of anisidine, the European Pharmacopeia 2.5.36; and the proposal of the PWG for methods of analysis for determination of triglycerides following the addition of a provision for triglycerides in the standard.

Conclusion

28. The Committee agreed to:

(i) Forward the draft standard for Fish Oils (Appendix III) to CAC40 for adoption at Step 8;

(ii) Send the labelling provisions for endorsement by CCFL;

(iii) Send the methods of analysis for endorsement by CCMAS, along with clarification on phospholipids; and a request for CCMAS to consider a factor for the conversion of phosphorus to phospholipids.

Addendum to Agenda Item 3 (CX/MAS 17/38/3-Add.1)

Consideration of Validation Data for amended version of AOAC 963.15, submitted by Indonesia.

BACKGROUND

FROM REP15/MAS

Regional Standard for Tempe

39. The Committee noted that CCASIA had agreed to replace the method of analysis for lipid content with ISO 1211|IDF 1:2010 as proposed by CCMAS in order to replace AOAC 983.23 which used chloroform as a reagent. It was pointed out that the scope of the ISO 1211|IDF 1:2010 did not include solid foods, such as tempe and that IDF and ISO did not intend to carry out work to extend the scope at this stage. The Committee agreed to retain the current method AOAC 983.23 for lipid determination in tempe and to request information from, in particular countries in the Asia region as to the applicability of the methods to tempe and whether this method had been tested on tempe products.

From REP16/MAS

Determination of lipid content (Regional *Standard for Tempe*)

41. The Committee, based on the information received, reconfirmed AOAC 983.23 for determination of lipids in tempe. The Delegation of Indonesia informed the Committee that they were using an amended version of the soxhlet extraction method for determination of fat in cocoa products. The Committee encouraged Indonesia to carry out validation studies for this method in tempe products.