CODEX ALIMENTARIUS COMMISSION E

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World Health Organization

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Agenda Item 3 **CX/MAS 24/43/3**

April 2024

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

43rd Session

Budapest, Hungary

13 – 18 May 2024

ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PLANS FOR PROVISIONS IN CODEX STANDARDS

1. This document contains the methods of analysis (Appendices I and II) proposed by the following Committees:
   - Codex Committee on Spices and Culinary Herbs (CCSCH) (REP24/SCH) [1]
     - Methods of analysis for provisions in different spices and culinary herbs standards: the *Standards for dried or dehydrated ginger* (CXS 343-2021), *Cloves* (CXS 344-2021), *Basil* (CXS 345-2021), *Saffron* (CXS 351-2021), *Chilli Pepper and Paprika* (CXS 353-2022);.
     - Methods of analysis for provisions in the draft Standard for spices derived from dried or dehydrated fruits and berries – small cardamom (submitted for adoption at Step 8 by CAC47).
     - Methods of analysis for provisions in the draft Standard for spices derived from dried or dehydrated fruits and berries – allspice, juniper berry and star anise (submitted for adoption at Step 8 by CAC47).
     - Methods of analysis for provisions in the draft Standard for dried or dehydrated roots, rhizomes and bulbs – turmeric (submitted for adoption at Step 5/8 by CAC47).
   - Codex Committee on Fats and Oils (CCFO) (REP24/FO) [2]
     - Revised methods of analysis for provisions in the revised *Standard for Olive Oils and Olive Pomace Oils* (CXS 33-1981) (submitted for adoption at Step 5/8 by CAC47).
     - Methods of analysis for provisions in the revised *Standard for Fish Oils* (CXS 329-2017) (submitted for adoption at Step 5/8 by CAC47).

CODEX COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH7)

Methods of analysis for provisions in different spices and culinary herbs standards

2. CCSCH7 agreed to forward the respective answers to CCMAS to support the endorsement decisions on the various methods in the different spices and culinary herbs standards (Appendix I, Part A).[3]

3. CCMAS **is invited to consider and endorse** the methods of analysis based on replies from CCSCH7 in Appendix I, Part B.

Draft Standard for spices derived from dried or dehydrated fruits and berries – small cardamom, Draft Standard for spices derived from dried or dehydrated fruits and berries -allspice, juniper berry and star anise, and Draft Standard for dried or dehydrated roots, rhizomes and bulbs – turmeric

4. CCMAS **is invited to endorse** the methods of analysis in Appendix I, Part B.

CODEX COMMITTEE ON FATS AND OILS (CCFO28)

Methods of analysis for provisions in the revised Standard for Olive Oils and Olive Pomace oils (CXS 33-1981) and the revised Standard for Fish Oils (CXS 329-2017)

5. CCMAS **is invited to endorse** the methods of analysis in Appendix II.

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1 REP24/SCH, paras 12, 30 (ii), 48 (ii), 70 (ii), 85 (ii) and Appendices II Part B, III, IV, V, and VI

2 REP24/FO, paras 85 (ii), 103 (ii) and Appendix IX Section 8 and Section 3, and Appendix X

3 For background on the discussions in CCMAS42 and referral to CCSCH, refer to REP23/MAS, paras 23 - 24

APPENDIX I
Part A

CODEX COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH7)

Methods of analysis for provisions in different Spices and Culinary Herbs Standards

REPLIES FROM CCSCH7 TO THE QUESTIONS FROM CCMAS42

	Questions/clarifications from CCMAS	Justification/recommendations
	Standard for Dried Roots, Rhizomes, and Bulbs – Dried or Dehydrated Ginger **(CXS 343-2021);** ***Standard for Dried Floral Parts – Cloves*** **(CXS 344-2021) and** ***Standard for Dried Basil*** **(CXS 345-2021)**	
1	ISO 927 is identified as a Type I whole dead insects, but only as a Type IV for live insects. Is there a reason for this difference in tying?	ISO 927 analytical method for live insects is also a direct method based on visual examination followed by gravimetry and is also a Type I method. *It is recommended to correct the typing of the method for the analysis of live insects to Type I.*
2	MPM V8 is listed as a Type IV for Mammalian/Other Excreta, however ISO 927 appears to capture this category and is identified as a Type I at other parts of the table, is there a reason for selecting a Type IV for this provision?	In this context, ISO 927 includes the method for rodent excreta only. As the provision is for mammalian excreta and other excreta, MPM V-8 is a more suitable method of analysis for mammalian excreta and other excreta. CCSCH has typed MPM V-8 as Type I method since excreta is defined by the method used (visual in this case). *It is recommended to correct the typing of MPM V-8 method to Type I.*
	Draft Standard for Dried Floral Parts – Saffron	
1	The taste strength, aroma strength, colouring strength provisions uses the ISO 3632-2 and is listed as Type IV. As this ISO standard is specific to saffron, is there a reason it is listed as a Type IV and not a Type I?	Taste strength, aroma strength, and colouring strength provisions of saffron are defined by the ISO 3632-2 method. Hence CCSCH has typed this as a Type I method. *It is recommended to correct the typing of this method to Type I.*
	Standard for Dried or Dehydrated Chilli Pepper and Paprika **(CXS 353-2022)**	
3.	For the provision Live Insect there are 2 methods listed and both identified as Type I. Are these methods identical? If not, one must be endorsed as the Type I method and the other removed.	ISO 927 analytical method for live insect is a Type 1 method. Hence AOAC 960.51 may be removed. *It is recommended to remove the AOAC 960.51method.*
	Draft Standard for dried small cardamom and draft Standard for spices derived from dried fruits and berries (Part A – allspice, juniper berry and star anise)	
1	There are Type I and Type IV methods listed for the provisions "whole dead insects" and "insect fragments". While listing both Type I and Type IV is allowed, there should be a compelling reason for the listing. Would it be possible to explain the reasoning for this request?	The first method ISO 927 (Type I) is applicable to whole dead insects in whole spices. AOAC 975.49 (Type I) is "Light filth in spices and condiments", which would be applicable to insect fragments for dried allspice, juniper berries, and star anise – in ground/small piece forms. Both these methods are required to analyze these two forms and two provisions. ISO 927 and AOAC 975.49 are complimentary methods for testing different styles of spices (whole and ground), and both are Type I methods. *CCSCH has concluded that both methods are required for*

		the provision and also revised the typing of the method AOAC 975.49 to Type I in the Methods of Analysis table.
2	There are parenthetical comments in the provision for 'filth' and 'light filth', which says list all the filth here – for example – mammalian excreta? It is unclear if this is text should have been removed.	This text has evolved since then and has been removed in the revised draft standards.
	Comparison between different CCSCH standards	
1.	In the *Standard for Dried Roots, Rhizomes and Bulbs – Dried or Dehydrated Ginger* (CXS 343-2021) ISO 927 is a Type IV for 'mammalian / other excreta', but in the *Standard for Dried Seeds – Nutmeg* (CXS 352-2022) ISO 927 is listed as a Type I for this same provision. Is there a reason for the different typing of the same method for the same provision?	In the *Standard for Dried Roots, Rhizomes and Bulbs – Dried or Dehydrated Ginger* (CXS 343-2021), MPM-V8 method has been recommended for the analysis of mammalian/other excreta (not ISO 927 method, which is a method for rodent excreta only) Based on the discussion in the committee, MPM V-8 is classified as a Type I method since this method is the one designated reference method and other Type I methods do not apply. *It is recommended to correct the typing of MPM V-8 method to Type I.*
2	In some standards the provision is listed as 'mould visible' and in others it is listed as 'visible mould', is there a significance to this difference or could a single name for the provision be used consistently across standards.	'Mould visible' and 'visible mould' imply the same provision. For consistency, the CCSCH standards would use the terminology given in respective references based on the criteria and methods of analysis.
3	Across standards, there are some differences in provision groups. One example, in the draft Standard for dried small cardamom the provision is 'whole insect live / dead', while in the *Standard for Dried Roots, Rhizomes and Bulbs – Dried or Dehydrated Ginger* (CXS 343-2021), the provisions are listed separately as 'whole dead insects' and 'live insect'. Are these intentional?	Based on the nature of the spice, and references available for that provision, the committee may combine the two provisions or list it separately. Spices and culinary herbs are very large and diverse group of plant products sometimes requiring separate provisions.

Appendix I
Part B

Methods of analysis for provisions in the Draft Standard for spices derived from dried or dehydrated fruits and berries – small cardamom

Provision	Method	Principle	Type
Moisture	ISO 939	Distillation	I
Total Ash, on dry basis	ISO 939 and ISO 928	Distillation and Gravimetry	I
Acid Insoluble Ash, on dry basis	ISO 939 and ISO 930	Distillation and Gravimetry	I
Volatile Oil on dry basis	ISO 939 and ISO 6571	Distillation followed by Volumetry	I
Extraneous Matter	ISO 927	Visual Examination followed by Gravimetry	I
Foreign Matter	ISO 927	Visual Examination followed by Gravimetry	I
Insect defiled/infested	ISO 927	Visual Examination followed by Gravimetry	I
Immature and shrivelled capsules	ISO 882-1	Visual Examination followed by Gravimetry	I
Mammalian or/and Other excreta	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs	Visual Examination followed by gravimetry	I
Mould visible	ISO 927	Visual Examination followed by gravimetry	I
Empty and malformed capsules	ISO 882-1	Visual Examination followed by gravimetry	I
Whole insect live/dead	ISO 927 (For whole)	Visual examination followed by gravimetry	I
	AOAC 975.49 (For powdered/pieces)	Floatation followed by gravimetry	I
Light seeds	IS 1907*	Visual examination followed by gravimetry	III

*IS 1907 is a method of analysis based on Indian standard.

Methods of analysis for provisions in the Draft Standard for spices derived from dried or dehydrated fruits and berries -allspice, juniper berry and star anise

Product Name	Provision	Method	Principles	Type
Allspice Juniper berries Star anise	Moisture	ISO 939	Distillation	I
	Total ash on dry basis	ISO 939 and ISO 928	Distillation and gravimetry.	I
	Acid- insoluble on dry basis	ISO 939 and ISO 930	Distillation and gravimetry	I
	Volatile oils on dry basis	ISO 939 and ISO 6571	Distillation followed by volumetry	I
	Extraneous matter	ISO 927	Visual examination followed by gravimetry	I
	Foreign matter	ISO 927	Visual examination followed by gravimetry	I
	Mould visible	ISO 927	Visual examination followed by gravimetry	I
	Mammalian and other excreta	MPM V-8 Spices, Condiments, Flavors and Crude Drugs MPM: V-8. Spices, Condiments, Flavors, and Crude Drugs | FDA	Visual examination followed by gravimetry	I
	Whole dead insects and live insects	ISO 927	Visual examination	I
	Insect fragments	ISO 927	Visual examination	I
		AOAC 975.49 (For powdered/pieces)	Flotation method	I
	Insect defiled	ISO 927	Visual examination followed by gravimetry	I
	Rodent hair	AOAC 965.40	Flotation	I

Methods of analysis for provisions in the Draft Standard for dried or dehydrated roots, rhizomes and bulbs - turmeric

Parameter	Method	Principle	Type
Moisture	ISO 939	Distillation	I
Total Ash on dry basis	ISO 939 and ISO 928	Distillation and gravimetry	I
Acid Insoluble Ash on dry basis	ISO 939 and ISO 930	Distillation and gravimetry	I
Curcuminoids content on dry basis (Colouring power)	ISO 2825 and ISO 5566	Spectrophotometry	I
Extraneous Matter	ISO 927	Visual examination followed by gravimetry	I
Foreign Matter	ISO 927	Visual examination followed by gravimetry	I
Insect defiled.	ISO 927	Visual examination followed by gravimetry	I
Whole insects Live /dead	ISO 927 (for whole) AOAC 975.49 (For powdered/ pieces)	Visual Examination followed by gravimetry Floatation followed by gravimetry	I
Mammalian or/and Other excreta	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs	Visual examination followed by gravimetry	I
Mould visible	ISO 927	Visual examination followed by gravimetry	I

APPENDIX II

CODEX COMMITTEE ON FATS AND OILS (CCFO28)

Methods of analysis and sampling for provisions in Proposed draft revision to the Standard for Olive Oils and Olive Pomace Oils (CXS 33-1981)

8. METHODS OF ANALYSIS AND SAMPLING

Fats and oils and related products	Provision	Method(s)	Principle	Type
Olive oils and olive pomace oils	Absorbency in ultra-violet	COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91	Absorption in ultra-violet	I
Olive oils and olive pomace oils	Acidity, free (acid value)	ISO 660 / AOCS Cd 3d-63 / COI/T.20/Doc. No 34	Titrimetry	I
Olive oils and olive pomace oils	Alpha-tocopherol	ISO 9936	HPLC (UV or fluorescence)	II
		AOCS Ce 8-89		III
Olive oils and olive pomace oils	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 and COI/T.20/Doc. No 33	Analysis of triglycerides by HPLC and fatty acids by GC followed by calculation	I
Olive oils and olive pomace oils	1,2 Diglycerides	COI/T.20/Doc. No 32[1]	Gas chromatography (FID)	II
		ISO 29822[1]		III
Olive oils and olive pomace oils	Erythrodiol + uvaol	COI/T.20/Doc. No 26	Separation and gas chromatography (FID)	II
Olive oils and olive pomace oils	Fatty acid composition	COI/T.20/Doc. No 33	Gas chromatography (FID) of methyl esters	II
		AOCS Ce 2-66 and AOCS Ch 2-91 / Ce 1h-05		III
		ISO 12966-2 and ISO 12966-4		III
Olive oils and olive pomace oils	2-glyceryl monopalmitate percentage	COI/T.20/Doc. No 23	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Fatty acid ethyl ester content	COI/T.20/Doc. No 28	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Halogenated solvents, traces	ISO 16035	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Insoluble impurities in light petroleum	ISO 663	Gravimetry	I
Olive oils and olive pomace oils	Iodine value	ISO 3961 / AOAC 9930.20 / AOCS Cd 1d-92 / NMKL 39	Wijs-Titrimetry	I

[1] This method is retained pending review in CCFO29 and CCFO30. For background, refer to REP24/FO paras 83 and 84.

Fats and oils and related products	Provision	Method(s)	Principle	Type
Olive oils and olive pomace oils	Iron and copper	ISO 8294 / AOAC 990.05	AAS	II
Olive oils and olive pomace oils	Lead	Use performance criteria*		
Olive oils and olive pomace oils	Moisture and volatile matter	ISO 662	Gravimetry	I
Olive oils and olive pomace oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	Panel test	I
Olive oils and olive pomace oils	Peroxide value	ISO 3960 / AOCS Cd 8b-90	Titrimetry	I
		COI/T.20/Doc. No 35		IV
Olive oils and olive pomace oils	Pyropheophytin "a"	ISO 29841[1]	HPLC with UV/VIS or fluorescence detection	II
Olive oils and olive pomace oils	Relative density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	I
Olive oils and olive pomace oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
Olive oils and olive pomace oils	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry	I
Olive oils and olive pomace oils	4α-desmethylsterol and total sterol content	COI/T.20/Doc. No 26	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Stigmastadienes	COI/T.20/Doc. no. 11	Gas chromatography (FID)	II
		ISO 15788-1		III
		AOCS Cd 26-96		III
		ISO 15788-2	HPLC	III
Olive oils and olive pomace oils	*trans* Fatty acids content	COI/T.20/Doc no. 33	Gas chromatography (FID) of methyl esters	II
		ISO 12966-2 and ISO 12966-4		III
		AOCS Ce 2-66 and AOCS Ce 1h-05		III
Olive oils and olive pomace oils	Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53	Gravimetry	I
Olive oils and olive pomace oils	Wax content	COI/T.20/Doc. no. 28	Gas chromatography (FID)	II
		AOCS Ch 8-02		III

* ISO 12193; AOAC 994.02; and AOCS Ca 18c-91 are currently listed in CXS 234.

Fats and oils and related products	Provision	Method(s)	Principle	Type
Olive oils and olive pomace oils	Sampling	ISO 5555 and ISO 661		

OTHER QUALITY AND COMPOSITION FACTORS

3. METHODS OF ANALYSIS AND SAMPLING

Fats and oils and related products	Provision	Method(s)	Principle	Type
Olive oils and olive pomace oils	Absorbency in ultra-violet	COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91	Absorption in ultra-violet	I
Olive oils and olive pomace oils	Acidity, free (acid value)	ISO 660 / AOCS Cd 3d-63 / COI/T.20/Doc. No 34	Titrimetry	I
Olive oils and olive pomace oils	Alpha-tocopherol	ISO 9936	HPLC (UV or fluorescence)	II
		AOCS Ce 8-89		III
Olive oils and olive pomace oils	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 and COI/T.20/Doc. No 33	Analysis of triglycerides by HPLC and fatty acids by GC followed by calculation	I
Olive oils and olive pomace oils	1,2 Diglycerides	COI /T.20/Doc.No 32[1]	Gas chromatography (FID)	II
		ISO 29822[1]		III
Olive oils and olive pomace oils	Erythrodiol + uvaol	COI/T.20/Doc. No 26	Separation and gas chromatography (FID)	II
Olive oils and olive pomace oils	Fatty acid composition	COI/T.20/Doc. No 33	Gas chromatography (FID) of methyl esters	II
		AOCS Ce 2-66 and AOCS Ch 2-91 / Ce 1h-05		III
		ISO 12966-2 and ISO 12966-4		III
Olive oils and olive pomace oils	2-glyceryl monopalmitate percentage	COI/T.20/Doc. No 23	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Fatty acid ethyl ester content	COI/T.20/Doc. No 28	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Halogenated solvents, traces	ISO 16035	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Insoluble impurities in light petroleum	ISO 663	Gravimetry	I
Olive oils and olive pomace oils	Iodine value	ISO 3961 / AOAC 9930.20 / AOCS Cd 1d-92 / NMKL 39	Wijs-Titrimetry	I

[1] This method is retained pending review in CCFO29 and CCFO30. For background, refer to REP24/FO paras 83 and 84.

Fats and oils and related products	Provision	Method(s)	Principle	Type
Olive oils and olive pomace oils	Iron and copper	ISO 8294 / AOAC 990.05	AAS	II
Olive oils and olive pomace oils	Lead	Use performance criteria*		
Olive oils and olive pomace oils	Moisture and volatile matter	ISO 662	Gravimetry	I
Olive oils and olive pomace oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	Panel test	I
Olive oils and olive pomace oils	Peroxide value	ISO 3960 / AOCS Cd 8b-90	Titrimetry	I
		COI/T.20/Doc. No 35		IV
Olive oils and olive pomace oils	Pyropheophytin “a”	ISO 29841[3]	HPLC with UV/VIS or fluorescence detection	II
Olive oils and olive pomace oils	Relative density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	I
Olive oils and olive pomace oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
Olive oils and olive pomace oils	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry	I
Olive oils and olive pomace oils	4α-desmethylsterol and total sterol content	COI/T.20/Doc. No 26	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Stigmastadienes	COI/T.20/Doc. no. 11	Gas chromatography (FID)	II
		ISO 15788-1		III
		AOCS Cd 26-96		III
		ISO 15788-2	HPLC	III
Olive oils and olive pomace oils	*trans* Fatty acids content	COI/T.20/Doc no. 33	Gas chromatography (FID) of methyl esters	II
		ISO 12966-2 and ISO 12966-4		III
		AOCS Ce 2-66 and AOCS Ce 1h-05		III
Olive oils and olive pomace oils	Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53	Gravimetry	I
Olive oils and olive pomace oils	Wax content	COI/T.20/Doc. no. 28	Gas chromatography (FID)	II
		AOCS Ch 8-02		III
Olive oils and olive pomace oils	Sampling	ISO 5555 and ISO 661		

* ISO 12193; AOAC 994.02; and AOCS Ca 18c-91 are currently listed in CXS 234.

Methods of analysis and sampling for provisions in Proposed draft amendment/revision of the Standard for fish oils (CXS 329-2017) : inclusion of calanus oil

8. METHODS OF ANALYSIS AND SAMPLING

Commodity	Provision	Method	Principle	Type
Fish oil	Wax content	AOCS Ch 8-02	Gas Chromatography	IV