

CODEX ALIMENTARIUS COMMISSION



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Agenda Item 3

CRD02

ORIGINAL LANGUAGE ONLY

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

43rd Session
Budapest, Hungary

13 – 18 May 2024

(Prepared by the Chair of the Virtual Working Group on endorsement of methods of analysis and sampling plans for provisions in Codex standards)

REPORT OF THE WORKING GROUP ON ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PLANS FOR PROVISIONS IN CODEX STANDARDS

CODEX COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH7)

1. The PWG on methods endorsement met virtually May 7 to discuss the methods of analysis for provisions in different spices and culinary herb standards including Methods of analysis for provisions in the Draft Standard for spices derived from dried or dehydrated fruits and berries – small cardamom, 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, and Methods of analysis for provisions in the Draft Standard for dried or dehydrated roots, rhizomes and bulbs – turmeric.
2. There were discussions on the chosen terminology for 'principles' of the methods but that a final decision was to be made after the CCMAS plenary had discussed the proposed names and format of method principles in CXS 234 (CX/MAS 24/43/10).
3. There were also discussions on whether to retain some type IV methods where a type I method was endorsed, but that decision was also postponed until the CCMAS plenary made a decision on the proposal to include both type I and IV in CXS 234 (CX/MAS 24/43/12).
4. The PWG also recommends to ask CCSCH if the provision **Curcuminoids content on dry basis (Colouring power)** should be changed to "Colouring Power" since the method does not measure curcuminoids.
5. The PWG recommends:
 - Endorsement of the methods and at the type indicated where consensus was reached (appendix I, tables 1-3)
 - Discussion during the PWG meeting and plenary on the remaining questions captured in the "status" column of appendix I.
 - Refer a question to CCSCH regarding the provision name Curcuminoids content on dry basis (Colouring power) and whether the provision should be changed to "colouring power" since the method does not measure curcuminoids.

CODEX COMMITTEE ON FATS AND OILS (CCFO28)

6. The PWG on methods endorsement met virtually on May 8 to discuss 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).
7. The PWG recommends:
 - Endorsement of the methods and at the type where consensus was reached (Appendix II tables 1-2);
 - Discussing the outstanding issues listed in the "status" column of all testing methods submitted by CCFO, in particular on (i) the methods pertaining to 1,2-diglycerides (DAGs) and pyropheophytin (PPP), as the revised Standard developed by CCFO lacks provisions for these; (ii) evaluation of the necessity of incorporating Type IV methods when Type I methods are already available.

Appendix I - Methods of analysis for provisions in different spices and culinary herb standards

Table 1: 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	Status (E = endorsed; NE = not endorsed)
Moisture	ISO 939	Distillation	I	<u>E</u>
Total Ash, on dry basis	ISO 939 and ISO 928	Distillation and Gravimetry	I	<u>E not consensus</u> <u>Review principle: Calculation from moisture and ash at 550</u> <u>Note: Delegation rec ASTA 3.0 to replace ISO 928, can't list 2 Type I methods</u>
Acid Insoluble Ash, on dry basis	ISO 939 and ISO 930	Distillation and Gravimetry	I	<u>E</u> <u>Calculation from moisture and ash at 550</u>
Volatile Oil on dry basis	ISO 939 and ISO 6571	Distillation followed by Volumetry	I	<u>E not consensus</u> <u>Review principle: Calculation from moisture and</u> <u>Note: Delegation rec to replace with AOAC 962.17, can't list 2 Type I methods.</u>
Extraneous Matter	ISO 927	Visual Examination followed by Gravimetry	I	<u>E</u>
Foreign Matter	ISO 927	Visual Examination followed by Gravimetry	I	<u>E</u>
Insect defiled/infested	ISO 927	Visual Examination followed by Gravimetry	I	<u>E</u>
Immature and shrivelled capsules	ISO 882-1 <u>and ISO 927</u>	Visual Examination followed by Gravimetry	I	<u>E</u>

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
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 (for whole)	Visual Examination followed by Gravimetry	IV	E
Mould visible	ISO 927	Visual Examination followed by Gravimetry	I	E
Empty and malformed capsules	ISO 882-1	Visual Examination followed by Gravimetry	I	E
Whole insect Live/dead	ISO 927 (For whole)	Visual examination followed by Gravimetry	I	E
Whole insect live/dead	AOAC 975.49 (For powdered/pieces)	Flotation followed by gravimetry	I	E
Light seeds	IS 1907 and IS 1797*	Visual examination followed by Gravimetry	III	Would ISO 927 be an option in place of 1797?

*IS 1907 and 1797 is a descriptions and an method of analysis based on Indian standard.

Table 2: 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

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
Moisture	ISO 939	Distillation	I	<u>E</u>
Total ash on dry basis	ISO 939 and ISO 928	Distillation and gravimetry.	I	<u>E not consensus</u> <u>Note: Delegation rec ASTA 3.0 to replace ISO 928, can't list 2 Type I methods</u>
Acid- insoluble on dry basis	ISO 939 and ISO 930	Distillation and gravimetry.	I	<u>E</u>
Volatile oils on dry basis	ISO 939 and ISO 6571	Distillation followed by volumetry.	I	<u>E not consensus</u> <u>Note: Delegation rec to replace with AOAC 962.17, can't list 2 Type I methods.</u>
Extraneous matter	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>
Foreign matter	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>
Mould visible	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>
Mammalian and other excreta	MPM V-8 Spices, Condiments, Flavors and Crude Drugs MPM: V-8. Spices, Condiments, Flavors, and Crude Drugs FDA (<u>whole spice</u>)	Visual examination followed by gravimetry	<u>IV</u>	<u>E</u>
Whole dead insects and live insects	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>
Insect fragments	ISO 927 (<u>whole spices</u>)	Visual examination followed by gravimetry	I	<u>E</u>
<u>Insect fragments</u>	AOAC 975.49 (For powdered/pieces)	Flotation method	I	<u>E</u>
Insect defiled	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>
Rodent hair	AOAC 965.40	Flotation	I	<u>E</u> <u>There was a question if both AOAC methods for insect fragments and rodent hair are needed or can one work for both provisions?</u>

Table 3: Methods of analysis for provisions in the Draft Standard for dried or dehydrated roots, rhizomes and bulbs – turmeric

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
Moisture	ISO 939	Distillation	I	<u>E</u>
Total Ash on dry basis	ISO 939 and ISO 928	Distillation and gravimetry	I	<u>E</u>
Acid Insoluble Ash on dry basis	ISO 939 and ISO 930	Distillation and gravimetry	I	<u>E</u>
<u>Curcuminoids content on dry basis (Colouring power)</u>	<u>ISO 939 and ISO 2825 and ISO 5566</u>	Spectrophotometry	I	<u>E</u> <u>Calculation from moisture from Curcuminoid content spectrophotometry and distillation. Rec a change to provision name to reflect what is being measured. This will need to go back to CCSCH.</u> <u>General question about adulteration in spices.</u>
Extraneous Matter	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>
Foreign Matter	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>
Insect defiled.	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>
Whole insects Live /dead	ISO 927 (for whole)	Visual Examination followed by gravimetry	I	<u>E</u>
<u>Whole insects Live /dead</u>	AOAC 975.49 (For powdered/ pieces)	Floation followed by gravimetry	<u>I</u>	<u>E</u>
Mammalian or/and Other excreta	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices <u>(whole)</u> https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs	Visual examination followed by gravimetry	<u>IV</u>	<u>E</u>
Mould visible	ISO 927	Visual examination followed by gravimetry	I	<u>E</u>

Appendix II - Methods of analysis referred by CCFO

Table 1: Methods of analysis for provisions in the revised *Standard for Olive Oils and Olive Pomace oils*

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
Absorbency in ultra-violet	COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91	Absorption in ultra-violet	I	<u>Methods E, but these should be separated into 3 provisions. Decision on that separation to be made later.</u>
Acidity, free (acid value)	ISO 660 (<u>section 9.1</u>) / AOCS Cd 3d-63 / COI/T.20/Doc. No 34	Titrimetry	I	<u>E</u> <u>Noted that these methods contain toluene in a specific situation</u>
<u>Acidity, free (acid value)</u>	<u>AOCS Ca 5a-40</u>	<u>Titrimetry</u>	<u>IV</u>	<u>Does not use toluene as the others do, this will be pending decision on Type I and Type IV in CXS 234</u>
Alpha-tocopherol	ISO 9936	HPLC (UV or fluorescence)	II	<u>E</u>
<u>Alpha-tocopherol</u>	AOCS Ce 8-89	<u>HPLC (UV or fluorescence)</u>	III	<u>E</u>
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	<u>E</u>

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
1,2 Diglycerides <u>no provision as of yet, see para 83 and 84</u>	COI /T.20/Doc. No 32	Gas chromatography (FID)	II	<u>Retain in 33 until the work is completed or in a CCFO working document or footnote as to status.</u> <u>Method fit for purpose, discussion on how to retain is needed.</u>
<u>1,2 Diglycerides no provision as of yet, see para 83 and 84</u>	ISO 29822	<u>Gas chromatography (FID)</u>	III	<u>The typing of these methods should be reviewed based on guidance and discussed at PWG and/or plenary</u>
Erythrodil + uvaol	COI/T.20/Doc. No 26	Separation and gas chromatography (FID)	II	<u>E</u>
Fatty acid composition	COI/T.20/Doc. No 33	Gas chromatography (FID) of methyl esters	II	<u>E</u>
<u>Fatty acid composition</u>	AOCS Ce 2-66 and AOCS Ch 2-91 / Ce 1h-05	<u>Gas chromatography (FID) of methyl esters</u>	III	<u>E</u>
<u>Fatty acid composition</u>	ISO 12966-2 and ISO 12966-4	<u>Gas chromatography (FID) of methyl esters</u>	III	<u>E</u>
2-glyceryl monopalmitate percentage	COI/T.20/Doc. No 23	<u>Hydrolysis and derivatization</u> Gas chromatography (FID)	II	<u>E</u>
Fatty acid ethyl ester content	COI/T.20/Doc. No 28	Gas chromatography (FID)	II	<u>E</u>
Halogenated solvents, traces	ISO 16035	<u>Headspace</u> Gas chromatography (ECD/FID)	II	<u>E</u>

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
Insoluble impurities in light petroleum	ISO 663	Gravimetry	I	<u>E</u>
Iodine value	ISO 3961 / AOAC 9930.20 / AOCS Cd 1d-92 / NMKL 39	Wijs-Titrimetry	I	<u>E</u>
Iron and copper	ISO 8294 / AOAC 990.05 / <u>AOCS Ca 18b-91 / NMKL 139 ?</u>	AAS	II	<u>Develop numeric criteria for review at PWG</u>
Lead	Use performance criteria*			<u>Methods or Typing not needed</u>
Moisture and volatile matter	ISO 662	Gravimetry	I	<u>E</u>
Organoleptic characteristics	COI/T.20/Doc. no. 15	<u>Sensory</u> Panel test	I	<u>E</u>
Peroxide value	ISO 3960 / AOCS Cd 8b-90 / <u>NMKL 158</u>	Titrimetry	I	<u>E</u>
<u>Peroxide value</u>	COI/T.20/Doc. No 35	<u>Titrimetry</u>	IV	<u>Decision should wait until decision on inclusion of Type I and Type IV. In this case it is the trade method and uses chloroform so is not preferred.</u>
Pyropheophytin "a" <u>no provision as of yet, see para 83 and 84</u>	ISO 29841	HPLC with UV/VIS or fluorescence detection	II	<u>Method fit for purpose, discussion on how to retain is needed.</u>
Relative density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	I	<u>E</u>
Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II	<u>E</u>

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

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry	I	<u>E</u>
4a-desmethylsterol and total sterol content	COI/T.20/Doc. No 26	Gas chromatography (FID)	II	<u>E</u>
<u>4a-desmethylsterol and total sterol content</u>	<u>ISO 12228-2 (part 2)</u>	<u>Separation Gas chromatography (FID)</u>	<u>III</u>	<u>E</u>
<u>4a-desmethylsterol and total sterol content</u>	<u>AOCS Ch 6-91</u>	<u>Separation Gas chromatography (FID)</u>	<u>III</u>	<u>E</u>
Stigmastadienes	COI/T.20/Doc. no. 11	Gas chromatography (FID)	II	<u>E</u>
<u>Stigmastadienes</u>	ISO 15788-1	<u>Gas chromatography (FID)</u>	III	<u>E</u>
<u>Stigmastadienes</u>	AOCS Cd 26-96	<u>Gas chromatography (FID)</u>	III	<u>E</u>
<u>Stigmastadienes</u>	ISO 15788-2	HPLC	III	<u>E</u>
<i>trans</i> Fatty acids content	COI/T.20/Doc no. 33	Gas chromatography (FID) of methyl esters	II	<u>E</u>
<u><i>trans</i> Fatty acids content</u>	ISO 12966-2 and ISO 12966-4	<u>Gas chromatography (FID) of methyl esters</u>	III	<u>E</u>
<u><i>trans</i> Fatty acids content</u>	AOCS Ce 2-66 and AOCS Ce 1h-05	<u>Gas chromatography (FID) of methyl esters</u>	III	<u>E</u>
Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53 / <u>AOCS Ca 6a-40</u>	Gravimetry	I	<u>Check for identical methods</u>
Wax content	COI/T.20/Doc. no. 28	Gas chromatography (FID)	II	<u>E</u>
<u>Wax content</u>	AOCS Ch 8-02	<u>Gas chromatography (FID)</u>	III	<u>E</u>

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
Sampling	ISO 5555 and ISO 661			<u>E</u>
Absorbency in ultra-violet	COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91	Absorption in ultra-violet	I	<u>Repeat</u>
Acidity, free (acid value)	ISO 660 / AOCS Cd 3d-63 / COI/T.20/Doc. No 34	Titrimetry	I	<u>Repeat</u>
Alpha-tocopherol	ISO 9936	HPLC (UV or fluorescence)	II	<u>Repeat</u>
<u>Alpha-tocopherol</u>	AOCS Ce 8-89	<u>HPLC (UV or fluorescence)</u>	III	<u>Repeat</u>
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	<u>Repeat</u>
1,2 Diglycerides <u>no provision as of yet, see para 83 and 84</u>	COI /T.20/Doc._No 32	Gas chromatography (FID)	II	<u>Repeat</u>
<u>1,2 Diglycerides no provision as of yet, see para 83 and 84</u>	ISO 29822	<u>Gas chromatography (FID)</u>	III	<u>Repeat</u>
Erythrodiol + uvaol	COI/T.20/Doc. No 26	Separation and gas chromatography (FID)	II	<u>Repeat</u>
Fatty acid composition	COI/T.20/Doc. No 33	Gas chromatography (FID) of methyl esters	II	<u>Repeat</u>
	AOCS Ce 2-66 and AOCS Ch 2-91 / Ce 1h-05		III	<u>Repeat</u>

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
	ISO 12966-2 and ISO 12966-4		III	Repeat
2-glyceryl monopalmitate percentage	COI/T.20/Doc. No 23	Gas chromatography (FID)	II	Repeat
Fatty acid ethyl ester content	COI/T.20/Doc. No 28	Gas chromatography (FID)	II	Repeat
Halogenated solvents, traces	ISO 16035	Gas chromatography (FID)	II	Repeat
Insoluble impurities in light petroleum	ISO 663	Gravimetry	I	Repeat
Iodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Wijs-Titrimetry	I	Repeat
Iron and copper	ISO 8294 / AOAC 990.05	AAS	II	Repeat
Lead	Use performance criteria*			Repeat
Moisture and volatile matter	ISO 662	Gravimetry	I	Repeat
Organoleptic characteristics	COI/T.20/Doc. no. 15	Panel test	I	Repeat
Peroxide value	ISO 3960 / AOCS Cd 8b-90	Titrimetry	I	Repeat
	COI/T.20/Doc. No 35		IV	Repeat
Pyropheophytin "a"	ISO 29841	HPLC with UV/VIS or fluorescence detection	II	Repeat

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

Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
Relative density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	I	Repeat
Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II	Repeat
Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry	I	Repeat
4a-desmethylsterol and total sterol content	COI/T.20/Doc. No 26	Gas chromatography (FID)	II	Repeat
				Repeat
Stigmastadienes	COI/T.20/Doc. no. 11	Gas chromatography (FID)	II	Repeat
	ISO 15788-1		III	Repeat
	AOCS Cd 26-96		III	Repeat
	ISO 15788-2	HPLC	III	Repeat
<i>trans</i> Fatty acids content	COI/T.20/Doc no. 33	Gas chromatography (FID) of methyl esters	II	Repeat
	ISO 12966-2 and ISO 12966-4		III	Repeat
	AOCS Ce 2-66 and AOCS Ce 1h-05		III	Repeat
Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53	Gravimetry	I	Repeat
Wax content	COI/T.20/Doc. no. 28	Gas chromatography (FID)	II	Repeat
	AOCS Ch 8-02		III	Repeat
Sampling	ISO 5555 and ISO 661			Repeat

Table 2: Methods of analysis for provisions in the revised *Standard for Fish Oils* (CXS 329-2017).

Commodity	Provision	Method	Principle	Type	Status (E = endorsed; NE = not endorsed)
Fish oil <u>Calanus Oil</u>	Wax content	AOCS Ch 8-02	Gas Chromatography	IV	<u>E</u> <u>Collaborative study being planned and that validation data will be presented when complete.</u>