CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

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Agenda Item 3 **CRD03**

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

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

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

Methods of Analysis referred by Codex Committee on Spices and Culinary Herbs (CCSCH) (REP24/SCH)

The PWG reviewed the methods of analysis referred by CCSCH in a virtual working group on May 7 and recommendations are recorded in CRD02. These include methods of analysis for provisions in the Draft Standard for spices derived from dried or dehydrated fruits and berries – small cardamom, spices derived from dried or dehydrated fruits and berries -allspice, juniper berry and star anise, and dried or dehydrated roots, rhizomes and bulbs – turmeric.

Questions remaining from PWG to CCMAS:

Cardamom

- Replace ISO 939 (moisture) with ASTA 3.0 (moisture)? CCSCH referred ISO 939 as method of choice. Both are type I and are not identical so CCMAS should decide.
- Replace ISO 6571 with AOAC 962.17 (volatile oil)? CCSCH referred ISO 6571 as method of choice. Both are type I and not identical so CCMAS should decide.
- Cardamom light seeds – I.S. 1907 (defines a light seed) and references I.S. 1797 (method to determine the amount of light seeds). Would ISO 927 which is endorsed for other provisions be appropriate in place of I.S. 1797?

Allspice, Juniper Berry, and Star Anise

- Replace ISO 939 (moisture) with ASTA 3.0 (moisture)? CCSCH referred ISO 939 as method of choice. Both are type I and are not identical so CCMAS should decide.
- Replace ISO 6571 with AOAC 962.17 (volatile oil)? CCSCH referred ISO 6571 as method of choice. Both are type I and not identical so CCMAS should decide.

The PWG recommends:

- Endorsement of the methods and at the type indicated (appendix I, tables 1-3)
- Not endorsing the methods for “Curcuminoids content on dry basis (Colouring power)” and returning the following question to CCSCH
    - The method ISO 5566 measures the absorbance of light at 425 nm and does not measure curcuminoids. Should the provision name be changed to “Colouring Power”?

The PWG also reviewed the methods of analysis referred by CCSCH including Methods of analysis for provisions in the *Standard for Dried Roots, Rhizomes and Bulbs – Dried or Dehydrated ginger* (CXS 343-2021), *Standard for Dried Floral Parts-Cloves* (CXS 344-2021), *Standard for Dried Leaves - Dried Basil* (CXS 345-2021), *Standard for Dried Floral Parts – Saffron* (CXS 351-2021), *Standards for Dried Seeds – Nutmeg* (CXS 352-2022), *Standard for Dried or Dehydrated Chilli Pepper and Paprika* (CXS 353-2022).

There was some discussion on keeping the method choice and typing consistent among the different commodities, e.g. endorsement of a method across the different commodities for a single provision when fit for purpose.

The PWG recommends:

- Endorsement of the methods and at the type indicated (appendix I, tables 4-9)
- Not endorsing AOAC 993.27 for 'Other Excreta' in Dried Floral Parts – Saffron. The method measures 'fecal particles/10 g portion' but the provision in CXS 351 is 'mg/kg.' The AOAC 993.27 measurand is different than the provision.
- Not endorsing FDA MPM V-8 as a type IV method for insect sample in dried floral parts – Cloves as ISO 927 is a type I method for that provision.
- Not endorsing ASTA 21.3 for the provision "Pungency, scoville heat units" as there was a question whether the technique of chromatography made this a rational method which is not allowed to co-exist with a type I method. It was also unclear whether ASTA 21.3 measured scoville heat units or whether it measured the chemical compounds capsicums and their oleoresins.
- Returning the following questions to CCSCH regarding the provisions method endorsement between ISO 927 and FDA MPM V-8:
    - To increase method consistency, ISO 927 could be endorsed for many provisions in spices rather than FDA MPM V-8. ISO 927 includes rodent excreta, other excreta (including insect and bird excreta), whole insects and parts (dead or alive, including mites and psocids), larvae, mouldy material (all seeds or leaves exhibiting mould), and insect defiled products) in the spice commodities under review. Would CCSCH consider ISO 927 as appropriate for the provisions listed to assist CCMAS in promoting consistency among endorsed methods?
    - Would CCSCH provide an equation to convert "fecal particles/10 g portion" into "mg excreta/kg", and if none is available, is another method suggested? Alternatively, would it be appropriate to change the provision name to "fecal particles/10 g portion?
    - ASTA 21.3 appears to measure capsicums and their oleoresins. Are the measurands of ASTA 21.3 in agreement with the provision "pungency, scoville heat units" since that is a sensory provision?

Methods of Analysis referred by Codex Committee on Fats and Oils (CCFO) (REP24/FO)

The PWG reviewed the methods of analysis referred by CCFO in a virtual working group on May 8 and recommendations are recorded in CRD02. These include methods of analysis for provisions in the Revised Standard for Olive Oils and Olive Pomace Oils (CXS 33-1981) and in the Revised Standard for Fish Oils (CXS 329-2017). There was discussion on endorsing alternative type IV methods that did not use toluene when a type I method was appropriate. The methods endorsed for alpha-tocopherol were retained as part of the food additive standard with a maximum use level even though there was no provision in the revised standard for alpha-tocopherol.

There was discussion on the best way to retain methods for 1,2 diglycerides (DAGs) and pyropheophytin (PPP). The revised standard does not contain these provisions, but CCFO confirmed the need to generate data for olive oil and olive pomace oil produced in different geographical and climatic regions that would support the further consideration of these parameters by CCFO30. While CCFO acknowledged that methods should only be forwarded to CCMAS when there was an associated provision, Members strongly recommended that these methods be included in the standard to promote the use of these specific methods in generating comparative data. The table referred to CCMAS contained a footnote stating: "The methods (DAGs and PPP) are retained pending review in CCFO29 and CCFO30. CCFO28 endorsed the ISO and IOC methods for DAGs, and the ISO method for PPP, and agreed to forward the list of methods to CCMAS with the explanation for the exceptional circumstances related to the inclusion of methods of analysis for DAGs and PPP in the standard.

The PWG found that the methods for 1,2 digylcerides and pyropheophytin 'a' were fit for purpose but did not reach consensus on the best way to retain this information.

The PWG also reviewed the sampling plan for olive oils and olive pomace oils, and the addition of 'wax content' in the proposed draft amendment/revision of the standard for fish oils (CXS 329-2017). The PWG also developed numeric performance criteria for the provisions iron (Fe) and copper (Cu) in olive oils and olive pomace oils. The performance criteria for lead (Pb) in olive oils and olive pomace oils was previously developed as that commodity falls within the Edible Oils group.

Questions remaining from PWG to CCMAS:

Acidity, free (acid value)

- Is the use of toluene an exceptional situation that would justify endorsing AOCS Ca 5a-40 as a type IV method when there already exists an endorsed type I method?

Peroxide value

- COI/T.20/Doc. No 35 was suggested as a type IV method when other identical methods for peroxide value have been endorsed. Does CCMAS find this an exceptional situation that would justify the co-listing of a type IV method when there already exists an endorsed type I method?

1,2 diglycerides and Pyropheophytin "a"

- Should CCMAS retain the typing suggested by CCFO while their work is ongoing. Would a footnote to the table indicating that the provision levels are undefined and the methods are endorsed pending the ongoing work of CCFO be appropriate?

The PWG recommends:

- Endorsement of the methods and at the type indicated and revocation or retyping of the current methods for olive oil and olive pomace oils (appendix II, table 1)
- Endorsement of the numeric performance criteria for lead, iron, and copper (appendix II, table 2)
- Endorsement of the method for wax content in calanus oil (appendix II, table 3). The method was endorsed as type IV pending the completion of a collaborative study and those validation data would be presented to CCMAS upon study completion.

Methods of Analysis and Sampling Plans referred by Codex Committee on Contaminants in Foods (CCCF)

Endorsement of the Sampling Plan for Methylmercury in Fish (CX/MAS 24/43/3 Add.1)

The PWG considered the sampling plan sent by the Codex Committee on Contaminants in Foods for methylmercury in fish (REP24/CF). There was discussion on the definition of Decision Rule (appendix I of CX/MAS 24/43/3 Add. 1). CCMAS agreed to revise and clarify the definition.

The PWG also agreed to integrate the numeric performance criteria for Orange Roughy and Pink cusk-eel into the existing numeric performance criteria table for methyl mercury in fish in CXS 234. The performance criteria for Orange Roughy and Pink cusk-eel are found in Appendix VI Table 4 under the Fish and Fishery Products workable package method review.

The PWG recommends:

- Informing CCCF of the revised definition of "Decision Rule" as follows:
    - "The lot is accepted if the test result is less than or equal to the Codex maximum level (ML); otherwise, the lot is rejected."
- Integrating Annex I from CX/MAS 24/43/3 Add. 1 into the numeric performance criteria table developed in the fish and fishery product workable package.
- Endorsing the Sampling Plan (Appendix I and Annex II) with a revised definition of "Decision Rule."

Examples of methods that can meet numeric performance criteria for aflatoxins in certain cereals and cereal-based products including foods for infants and young children

The PWG reviewed the responses to CL 2024/08-MAS of example methods that could meet the performance criteria for aflatoxins in certain cereals and cereal based products. The PWG considered the methods proposed (MAS43/CRD05): EN 17641:2022 and EN 15851:2010. EN 17641:2022 was found to meet the performance criteria whereas EN 15851:2010 does not quantify all aflatoxin isomers. The PWG noted that the CCCF sampling plan for aflatoxins required 25 g test portion size, while EN 17641:2022 used 5 g test portion size due to the cost of the isotopically labeled internal standards. The PWG agreed to recommend EN 17641:2022 as an example method along with a footnote to the performance criteria table.

The PWG recommends:

- Including EN 17641:2022 as an example method in the numeric performance criteria table with the following footnote:
    - "The sampling plan specifies a test portion size of 25.0 g and EN 17641:2022 uses a test portion size of 5.0 g. No alternative method was found that meets the performance criteria, therefore EN 17641:2022 is included as an example method as it is the best possible method at this time. The smaller test portion size may introduce additional variation in the test result."
- The PWG also recommends notifying CCCF of the footnote as a matter for information.

Review of methods of analysis for contaminants: performance criteria for lead and cadmium in foods

The PWG reviewed the suggested methods that would meet the performance criteria for lead (Pb) and cadmium (Cd) in foods. The PWG prepared a table including performance criteria and methods that meet those criteria.

The PWG recommends:

- Endorsement of the example methods in Appendix III.

Methods of Analysis and Sampling Plans referred by Codex Committee on Food Hygiene (CCFH)

The PWG considered the list of methods from CXS 230 with the additional information contained in a response to CL 2024/08-MAS contained in CRD05. The information in CRD 05 showed that the methods are still in use and fit for purpose except EN 13783 (Difference between total microorganism count and viable microorganism count) and EN 13784 (Detection of DNA fragmentation presumptive to irradiation treatment) which both have been withdrawn. In addition, the footnote to EN 1785 should be removed.

The PWG recommends:

- Endorsement of the methods in appendix IV and inclusion in CXS 234
- Proposal for the removal of methods and revocation of CXS 230

Review of CXS 234 – Workable packages for processed fruits and vegetables, cereals, pulses and legumes, fish and fishery products, and fruit juices

Establishment of Numeric Performance Criteria from the Processed Fruits and Vegetable Package

The PWG reviewed the numeric performance criteria developed after the review of processed fruits and vegetable methods at CCMAS 42 (2023). The PWG reviewed these criteria in a virtual working group on May 7 and recommendations are recorded in CRD02. Of note, the numeric performance criteria were not developed for some calcium provisions in preserved tomatoes, canned citrus fruits, certain canned vegetables, and citrus marmalade.

The PWG recommends:

- Endorsement of the numeric performance criteria in appendix V and inclusion in CXS 234
- Retained typing for the methods for calcium in preserved tomatoes, canned citrus fruits, certain canned vegetables, and citrus marmalade

Review of Methods of Analysis in CXS 234 Cereals, Pulses, and Legumes Workable Package

The PWG considered the work and comments of the electronic working group on the Cereals, Pulses, and Legumes workable package (CX/MAS 24/43/4). There was extensive discussion on the ash provision for degermed maize (corn) meal and maize (corn) grits, Pearl millet flour, Sorghum flour, Sorghum grains, Vegetable protein products, Wheat flour, Wheat protein products including wheat gluten, Whole and decorticated pearl millet grains, Whole maize (corn) meal, and Couscous. The discussion was between the most appropriate ashing temperature, either 550 ℃ or 900℃. It was not decided which temperature was originally intended when the provision was specified, and that the two temperatures would yield different results. It was also stated that the amount of salt in the commodity would affect the result with commodities that contain salt will be most appropriate at 550℃, and products that do not contain salt will require 900℃ ashing temperature.

The EWG proposed two provisions for Ash to separate the temperature requirements: Ash-550℃ and Ash-900℃. With the exception of the ash methods, most of the methods were recommended for endorsement without comment which is indicated in the comment column in appendix VI. The Particle size method required a special note because additional sieves are required before CCMAS can endorse it (gari and edible cassava flour).

The ash methods have been highlighted in bold with the corresponding temperatures and a note explaining what resulted in the need for two temperatures has been included as a footnote to the Appendix VI.

The PWG recommends:

- Endorsement of the methods in appendix VI
- Referring a question back to CAC on whether CCMAS can have assistance in determining the original intent for ashing temperature, and if not, would two provisions at 550℃ and 900℃ be acceptable?

Review of Methods of Analysis in CXS 234 Fish and Fish Products Workable Package

The PWG considered the work and comments of the electronic working group on the fish and fish products workable package (CX/MAS 24/43/5). The PWG discussed the availability of methods that could achieve the numeric performance criteria for the OA group and AZA group of toxin analogues. It was stated that the practical recovery range should be 70-130% rather than 80-120% and the PWG agreed. At such time when a new method that can achieve better recovery is available, it will be added to the examples of application methods that meet the criteria.

The numeric performance criteria from the methylmercury sampling plan (CX/MAS 24/43/3 Add.1) with the additional two fish species (orange roughy and pink cusk-eel) were integrated into appendix VII table 4.

The PWG recommends:

- Endorsement of methods and performance criteria in appendix VII

Methods of analysis for determination of moisture content in dried milk

The PWG reviewed and discussed the validation data submitted in support of drying at 102 ℃. The PWG did not reach consensus and it was agreed to continue discussion during the plenary session and hold an in session work group on the matter.

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
Moisture	ISO 939	Distillation	I	Q to CCMAS – replace ISO 939 w ASTA 3.0?
Total Ash, on dry basis	ISO 939 and ISO 928	Distillation and Gravimetry	I	Endorsed
Acid Insoluble Ash, on dry basis	ISO 939 and ISO 930	Distillation and Gravimetry	I	Endorsed
Volatile Oil on dry basis	ISO 939 and ISO 6571	Distillation followed by Volumetry	I	Q to CCMAS - replace IS 6571 with AOAC 962.17?
Extraneous Matter	ISO 927	Visual Examination followed by Gravimetry	I	Endorsed
Foreign Matter	ISO 927	Visual Examination followed by Gravimetry	I	Endorsed
Insect defiled/infested	ISO 927	Visual Examination followed by Gravimetry	I	Endorsed
Immature and shrivelled capsules	ISO 882-1 **and ISO 927**	Visual Examination followed by Gravimetry	I	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	~~I~~ **IV**	Endorsed
Mould visible	ISO 927	Visual Examination followed by Gravimetry	I	Endorsed
Empty and malformed capsules	ISO 882-1	Visual Examination ~~followed by gravimetry~~	I	Endorsed
Whole insect Live/dead	ISO 927 (For whole)	Visual examination followed by Gravimetry	I	Endorsed
Whole insect live/dead	AOAC 975.49 (For powdered/pieces)	Flotation ~~followed by gravimetry~~	I	Endorsed
Light seeds	IS 1907 **and IS 1797**	Visual examination followed by Gravimetry	**I** ~~III~~	Q to CCMAS - Would ISO 927 be an option in place of 1797?

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
Moisture	ISO 939	Distillation	I	Q to CCMAS – ISO 939 or ASTA 3.0?
Total ash on dry basis	ISO 939 and ISO 928	Distillation and gravimetry.	I	Endorsed
Acid- insoluble on dry basis	ISO 939 and ISO 930	Distillation and gravimetry.	I	Endorsed
Volatile oils on dry basis	ISO 939 and ISO 6571	Distillation followed by volumetry.	I	Q to CCMAS - IS 6571 or AOAC 962.17?
Extraneous matter	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Foreign matter	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Mould visible	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Mammalian and other excreta	MPM V-8 Spices, Condiments, Flavors and Crude Drugs MPM: V-8. Spices, Condiments, Flavors, and Crude Drugs | FDA **(whole spice)**	Visual examination followed by gravimetry	~~I~~ **IV**	Endorsed
Whole dead insects and live insects	ISO 927	Visual examination **followed by gravimetry**	I	Endorsed
Insect fragments	ISO 927 **(whole spices)**	Visual examination **followed by gravimetry**	I	Endorsed
Insect fragments	AOAC 975.49 (For powdered/pieces)	Flotation method	I	Endorsed
Insect defiled	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Rodent hair	AOAC 965.40	Flotation	I	Endorsed Are both AOAC methods for insect fragments and rodent hair needed or can one work for both provisions?

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
Moisture	ISO 939	Distillation	I	Endorsed
Total Ash on dry basis	ISO 939 and ISO 928	Distillation and gravimetry	I	Endorsed
Acid Insoluble Ash on dry basis	ISO 939 and ISO 930	Distillation and gravimetry	I	Endorsed
~~Curcuminoids content on dry basis (~~Colouring power~~)~~	**ISO 939 and** ISO 2825 and ISO 5566	Spectrophotometry	I	Not Endorsed Q to CCSCH: The method ISO 5566 measures the absorbance of light at 425 nm and does not measure curcuminoids. Should the provision name be changed to "Colouring Power"?
Extraneous Matter	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Foreign Matter	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Insect defiled.	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Whole insects Live /dead	ISO 927 (for whole)	Visual Examination followed by gravimetry	I	Endorsed
Whole insects Live /dead	AOAC 975.49 (For powdered/ pieces)	Floatation followed by gravimetry	I	Endorsed
Mammalian or/and Other excreta	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices **(whole)** https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs	Visual examination followed by gravimetry	~~I~~ **IV**	Endorsed
Mould visible	ISO 927	Visual examination followed by gravimetry	I	Endorsed

Table 4 - Methods of analysis for provisions in the *Standard for Dried Roots, Rhizomes and Bulbs – Dried or Dehydrated ginger* (CXS 343-2021)

Provision	Method	Principle	Type[1]	STATUS
Moisture	ISO 939	Distillation	I	Endorsed
Total Ash ~~on dry basis~~ **(dry weight basis)**	ISO 939 and ISO 928 **(ashing at 600ºC)**	**Calculation from moisture and ash**, Distillation and Gravimetry	I	Endorsed
Acid Insoluble Ash ~~on dry Basis~~ **(dry weight basis)**	ISO 939 and ISO 930	**Calculation from moisture and ash,** Distillation and Gravimetry	I	Endorsed
Volatile Oil ~~on dry basis~~ **(dry weight basis)**	ISO 939 and ISO 6571	**Calculation from moisture and volatile oils,** Distillation ~~followed by Volumetry~~ **and Distillation**	I	Endorsed
Extraneous Matter	ISO 927	Visual **examination followed by** Gravimetry	I	Endorsed
Foreign Matter	ISO 927	Visual **examination followed by** Gravimetry	I	Endorsed
Insect Damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices	Visual **examination followed by** Gravimetry	IV	Endorsed
Whole dead insect	ISO 927	Visual **examination followed by** Gravimetry	I	Endorsed
Mammalian/ Other Excreta	~~MPM V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices (For whole)~~ **Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (for whole) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32**	Visual examination followed by Gravimetry	~~I~~ IV	Endorsed as type IV
Mould visible	~~Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices~~ **ISO 927**	Visual examination **followed by Gravimetry**	~~IV~~ I	Endorsed as type I
Live Insect	ISO 927 ~~AOAC 960.51~~	Visual examination	~~IV~~ **I** ~~IV~~	Endorsed as type I
Calcium (as oxide) on dry basis	**ISO 939 and ISO 928 (ashing at 600ºC) and** ISO 1003 – Annex A	~~Chemical reaction followed by gravimetry~~ **Calculation from moisture and ash, and titrimetry**	IV	Endorsed
Sulphur dioxide ~~SO_2~~	**AOAC 990.28** ~~AOAC 963.20~~	~~Colorimeter~~ **Distillation followed by titrimetry**	~~II~~ **IV**	II

Table 5 - Methods of analysis for provisions in the *Standard for Dried Floral Parts-Cloves* (CXS 344-2021)

Provision	Method	Principle	Type	STATUS
Moisture	**ISO 939** ~~ASTA 2.0~~	Distillation	I	Endorsed
Volatile Oil ~~on dry basis~~ **(dry weight basis)**	**ISO 939 and** ISO 6571	**Calculation from moisture and volatile oils,** Distillation ~~followed by Volumetry~~ **and Distillation**	I	Endorsed
Total Ash ~~on dry basis~~ **(dry weight basis)**	**ISO 939 and** ISO 928	**Calculation from moisture and ash,** Distillation and Gravimetry	I	Endorsed
Acid Insoluble Ash ~~on dry Basis~~ **(dry weight basis)**	**ISO 939** and ISO 930	**Calculation from moisture and ash,** Distillation and Gravimetry	I	Endorsed
Extraneous matter	ISO 927	Visual examination **followed by** Gravimetry	I	Endorsed
Foreign matter	ISO 927	Visual examination **followed by** Gravimetry	I	Endorsed
Insect damage	ISO 927	Visual examination **followed by** Gravimetry	I	Endorsed as type I
	~~Method V-8 Spices, Condiments, Flavors and Crude Drugs~~	~~Visual Examination~~	~~IV~~	Not endorsed
Insects/Excreta/Insect fragments	ISO 927	Visual examination **followed by** Gravimetry	~~IV~~ **I**	Endorsed as type I
Crude fibre	ISO 5498	Gravimetry	I	Endorsed
Mould visible	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) (for whole) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32	Visual examination **followed by** Gravimetry	IV	Endorsed, but PWG report sends Q back to CCSCH on whether ISO 927 would be a better type I method
Live insect	ISO 927	Visual **examination**	~~IV~~ **I**	Endorsed
Mammalian or/and Other excreta	MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V 32) (For whole) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32	Visual Examination	IV	Endorsed

Table 6 - Methods of analysis for provisions in the *Standard for Dried Leaves - Dried Basil* (CXS 345-2021)

Provision	Method	Principle	Type	STATUS
Moisture	ISO 939	Distillation	I	Endorsed
Total Ash **(dry weight basis)**	**ISO 939 and** ISO 928	**Calculation from moisture and ash, Distillation and** Gravimetry	I	Endorsed
Acid Insoluble Ash **(dry weight basis)**	ISO 928 and ISO 930	**Calculation from moisture and ash, Distillation and** Gravimetry	I	Endorsed
Volatile Oil **(dry weight basis)**	**ISO 939 and** ISO 6571	**Calculation from moisture and volatile oils,** Distillation ~~followed by Volumetry~~ **and Distillation**	I	Endorsed
Extraneous Matter	ISO 927	Visual Examination followed by ~~Volumetry~~ **Gravimetry**	I	Endorsed
Foreign Matter	ISO 927	Visual Examination followed by ~~Volumetry~~ **Gravimetry**	I	Endorsed
Insect Damage	~~Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)~~ **Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) (whole leaves) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32**	Visual Examination	IV	Endorsed
Insects/Excreta/Insect Fragments	~~Method appropriate for particular spice from AOAC Chapter 16, subchapter 14~~ **ISO 927**	Visual Examination followed by Gravimetry	~~IV~~ **I**	Endorsed as type I
Mould damage	~~Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)~~ **Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) (for whole leaves)**	Visual examination (for whole)	IV	Endorsed

Provision	Method	Principle	Type	STATUS
	https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32			
Mammalian Excreta, And Other Excreta	~~Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B)(For whole)~~ **MPM V-8 Spices, Condiments, Flavours and Crude Drugs** **A. General methods for spices herbs and botanicals (V 32) (For whole leaves)** **https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32**	Visual Examination	~~I~~ **IV**	Endorsed as a type IV

Table 7 - Methods of analysis for provisions in the *Standard for Dried Floral Parts – Saffron* (CXS 351-2021)

Provision	Method	Principle	Type	STATUS
Moisture	ISO 3632-2	Gravimetry **(drying at 103ºC)**	I	Endorsed
Total Ash **(dry weight basis)**	ISO 3632-2 and ISO 928	**Calculation from moisture and ash (ash at 550ºC), Gravimetry and** Gravimetry	I	Endorsed
Acid Insoluble Ash **(dry weight basis)**	ISO 3632-2 **and ISO 928** and ISO 930	**Calculation from moisture and ash, Gravimetry and** Gravimetry	I	Endorsed
Soluble extract in cold water **(dry weight basis)**	ISO 3632-2 and ISO 941	**Calculation from moisture and Soluble extract, Gravimetry and** Extraction	I	Endorsed
Taste strength (expressed as picrocrocin) $A^{1\%}_{1cm}$ 257 nm	ISO 3632-2	Absorbance	~~IV~~ **I**	Endorsed as type I
Aroma strength (expressed as safranal) $A\ ^{1\%}_{1cm}$ 330 nm	ISO 3632-2	Absorbance	~~IV~~ **I**	Endorsed as type I
Coloring strength (expressed as crocin) $A\ ^{1\%}_{1cm}$ 440 nm	ISO 3632-2	Absorbance	~~IV~~ **I**	Endorsed as type I
Extraneous Matter	ISO 3632-2	Visual Examination followed by Gravimetry	I	Endorsed
Foreign Matter	ISO 3632-2	Visual Examination followed by Gravimetry	I	Endorsed
Insect Damage	ISO 927	Visual Examination followed by Gravimetry	I	Endorsed
Whole dead Insects /Insect Fragments	ISO 927	Visual examination **followed by** Gravimetry	I	Endorsed
Visible mould	~~Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macro analytical Procedure Manual, FDATechnical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/Laoratory Methods/ucm084394.htm#v-32~~ **ISO 927**	Visual Examination followed by Gravimetry	I	Endorsed
Mammalian Excreta	~~Macro analytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole)~~ **MPM V-8 Spices, Condiments, Flavors and Crude Drugs MPM: V-8. Spices, Condiments, Flavors, and Crude Drugs | FDA (whole spice)**	Visual Examination followed by Gravimetry	~~I~~ **IV**	Endorsed

Provision	Method	Principle	Type	STATUS
Other Excreta	AOAC 993.27 (For Ground)	**Calculation from** Enzymatic Detection Method **and conversion of 'fecal particles counts/10g' to 'mg excreta/kg'**	IV	Not endorsed
Rodent filth	ISO 927	Visual Examination **followed by Gravimetry**	I	Endorsed

Table 8 - Methods of analysis for provisions in the *Standard for Dried Seeds - Nutmeg* (CXS 352-2022)

Provision	Method	Principle	Type	OUTCOME
Moisture content	ISO 939	Distillation	I	Endorsed
Total ash **(dry weight basis)**	ISO 939 and ISO 928	**Calculation from moisture and ash (ash at 550ºC),** Distillation **and** Gravimetry	I	Endorsed
Acid-insoluble ash **(dry weight basis)**	ISO 939 and ISO 930	**Calculation from moisture and ash,** Distillation **and** Gravimetry	I	Endorsed
Water-insoluble ash **(dry weight basis)**	ISO 939 and ISO 929	**Calculation from moisture and ash,** Distillation **and** Gravimetry	I	Endorsed
Volatile oil content **(dry weight basis)**	ISO 939 and ISO 6571	**Calculation from moisture and volatile oils,** Distillation **and** Distillation	I	Endorsed
Extraneous matter	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Foreign matter	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Visible mould	ISO 927	Visual examination followed by gravimetry	I	Endorsed
Insect defiled/infested	~~MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V 32)~~ **ISO 927**	Visual Examination followed by gravimetry	I	Endorsed
Dead insect, insect fragments, rodent contamination	ISO 927	Visual Examination **followed by Gravimetry**	I	Endorsed
Live insect	ISO 927	Visual Examination	I	Endorsed
Mammalian and or other excreta	~~Macroanalytical Procedure Manual (MPM) USFDA technical bulletin V.41~~ **Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.41 (For whole and broken) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32**	Visual examination followed by gravimetry	I	Endorsed
Piece of mace	ISO 927	Visual examination followed by gravimetry	I	Endorsed

Table 9 - Methods of analysis for provisions in the *Standard for Dried or Dehydrated Chilli Pepper and Paprika* (CXS 353-2022)

Provision	**Method[1]**	**Principle**	**Type[2]**	**OUTCOME**
Moisture	ISO 939	Distillation	I	Endorsed
Total Ash **(dry weight basis)**	ISO 939 and ISO 928	**Calculation from moisture and ash (ash at 550ºC),** Distillation **and** Gravimetry	I	Endorsed
Acid-insoluble ash **(dry weight basis)**	ISO 939 and ISO 930	**Calculation from moisture and ash,** Distillation **and** Gravimetry	I	Endorsed
~~Pungency, Scoville Heat units~~	~~ASTA 21.3~~	~~Chromatography~~		Not Endorsed
Pungency, Scoville Heat units	ISO 3513	Sensory evaluation	I	Endorsed
Colour value	ISO 7541	Spectrophotometry	IV	Endorsed
Mammalian excreta	ISO 927	Visual examination followed by Gravimetry (whole)	I	Endorsed
Mould damage **(for whole chilli peppers)**	MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V.32)	Visual Examination followed by Gravimetry	I	Endorsed
Mould damage **(for ground)**	AOAC 945.94	Visual Examination **(Howard Mould Count)**	I	Endorsed
Insect Damage **(for whole chilli peppers)**	MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V.32)	Visual Examination followed by Gravimetry	I	Endorsed
Extraneous matter	ISO 927	Visual Examination followed by Gravimetry	I	Endorsed
Foreign matter	ISO 927	Visual Examination followed by Gravimetry	I	Endorsed
Live insect	ISO 927 ~~/ AOAC 960.51~~	Visual Examination	I	Endorsed
Insect filth	ISO 927	Visual Examination	I	Endorsed
Insect fragments	ISO 927	Visual examination	I	Endorsed
Rodent hair (Ground chilli)	AOAC 978.22	Microscopic examination	I	Endorsed
Rodent hair (Ground paprika)	**AOAC 977.25 B**	**Microscopic examination**	**I**	Endorsed

Appendix II – Methods of analysis for Olive Oils and Olive Pomace Oils

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

Provision	Method	Principle	Type	Status
Absorbency in ultra-violet	COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91	Absorption in ultra-violet	I	Methods Endorsed, but may need to be separated
Acidity, free (acid value)	ISO 660 **(section 9.1)**/ AOCS Cd 3d-63 / COI/T.20/Doc. No 34	Titrimetry	I	Endorsed These methods use toluene in a specific situation
	AOCS Ca 5a-40	**Titrimetry**	**IV**	Method does not use toluene. Is this a justifiable reason to endorse as type IV?
Alpha-tocopherol	ISO 9936	HPLC (UV or fluorescence)	II	Endorsed
Alpha-tocopherol	AOCS Ce 8-89	HPLC (UV or fluorescence)	III	Endorsed
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	Endorsed
~~Difference between the actual and theoretical ECN 42 triglyceride content~~	~~AOCS Ce 5b-89~~	~~Analysis of triglycerides of HPLC and calculation~~	~~I~~	Revoked and replaced with methods above
1,2 Diglycerides	COI /T.20/Doc. No 32	Gas chromatography (FID)	II	Method fit for purpose, discussion on how to retain is needed.
	ISO 29822	**Gas chromatography (FID)**	III	The typing of these methods should be reviewed based on guidance and discussed at PWG and/or plenary
Erythrodiol + uvaol	COI/T.20/Doc. No 26	Separation and gas chromatography (FID)	II	Endorsed
Erythrodiol + uvaol	COI/T.20/Doc.no. 30	Gas chromatography		Revoke or retype to type III?
Fatty acid composition	COI/T.20/Doc. No 33	Gas chromatography (FID) of methyl esters	II	Endorsed
	AOCS Ce 2-66 and AOCS Ch 2-91 / Ce 1h-05	**Gas chromatography (FID) of methyl esters**	III	Endorsed

Provision	Method	Principle	Type	Status
	ISO 12966-2 and ISO 12966-4	**Gas chromatography (FID) of methyl esters**	III	Endorsed
2-glyceryl monopalmitate percentage	COI/T.20/Doc. No 23	**Hydrolysis and derivatization** Gas chromatography (FID)	II	Endorsed
Fatty acid ethyl ester content	COI/T.20/Doc. No 28	Gas chromatography (FID)	II	Endorsed
Halogenated solvents, traces	ISO 16035	**Headspace** Gas chromatography (**ECD**~~FID~~)	II	Endorsed
Halogenated solvents, traces	COI/T.20/Doc. no. 8	Gas chromatography		Revoke or retype to type III?
Insoluble impurities in light petroleum	ISO 663	Gravimetry	I	Endorsed
Iodine value	ISO 3961 / AOAC 993~~0~~.20 / AOCS Cd 1d-92 / NMKL 39	Wijs-Titrimetry	I	Endorsed
Iron and copper	~~ISO 8294 / AOAC 990.05 / AOCS Ca 18b-91~~	~~AAS~~	~~II~~	Performance Criteria
Lead				Performance Criteria
Moisture and volatile matter	ISO 662	Gravimetry	I	Endorsed
Organoleptic characteristics	COI/T.20/Doc. no. 15	**Sensory** Panel test	I	Endorsed
Peroxide value	ISO 3960 / AOCS Cd 8b-90 **/ NMKL 158**	Titrimetry	I	Endorsed
	COI/T.20/Doc. No 35	**Titrimetry**	**IV**	Uses chloroform so is not preferred. Is this a justifiable reason to endorse as type IV?
Pyropheophytin “a”	ISO 29841	HPLC with UV/VIS or fluorescence detection	II	Method fit for purpose, discussion on how to retain is needed.
Relative density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	I	Endorsed
~~Relative density~~	~~IUPAC 2.101, with the appropriate conversion factor.~~	~~Pycnometry~~	~~I~~	Revoke due to a type I method
Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II	Endorsed
Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry	I	Endorsed
4α-desmethylsterol and total sterol content	COI/T.20/Doc. No 26	Gas chromatography (FID)	II	Endorsed
	ISO 12228-2 (part 2)	**Separation Gas chromatography (FID)**	**III**	Endorsed
	AOCS Ch 6-91	**Separation Gas chromatography (FID)**	**III**	Endorsed
~~Sterol composition and total sterols~~	~~COI/T.20/Doc. no. 30 | ISO 12228-2 or AOAC Ch 6-91~~	~~Gas chromatography~~	~~II~~	Replaced and typed as above
Stigmastadienes	COI/T.20/Doc. no. 11	Gas chromatography (FID)	II	Endorsed
	ISO 15788-1	Gas chromatography (FID)	III	Endorsed
	AOCS Cd 26-96	Gas chromatography (FID)	III	Endorsed

Provision	Method	Principle	Type	Status
	ISO 15788-2	HPLC	III	Endorsed
trans Fatty acids content	COI/T.20/Doc no. 33	Gas chromatography (FID) of methyl esters	II	Endorsed
	ISO 12966-2 and ISO 12966-4	Gas chromatography (FID) of methyl esters	III	Endorsed
	AOCS Ce 2-66 and AOCS Ce 1h-05	Gas chromatography (FID) of methyl esters	III	Endorsed
	COI/T.20/Doc no. 17 or ISO 15304 or AOCS Ch 2a-94	Gas chromatography of methyl esters		Revoke or retype to type III?
Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53 **/ AOCS Ca 6a-40**	Gravimetry	I	Needs to be checked for identical method
~~Unsaponifiable matter~~	~~ISO 18609~~	~~Gravimetry~~		Revoke due to type I
Wax content	COI/T.20/Doc. no. 28	Gas chromatography (FID)	II	Endorsed
	AOCS Ch 8-02	Gas chromatography (FID)	III	Endorsed
	COI/T.20/Doc. no. 18	Gas chromatography		Revoke or retype to type III?
Sampling	ISO 5555 and ISO 661			Endorsed

Appendix II – Methods of analysis for Olive Oils and Olive Pomace Oils

Table 2 - Numeric Performance Criteria for Fe and Cu in Olive Oils and Olive Pomace Oils (CXS 33-1981)

Commodity	Provision	ML (mg/kg)	Method Performance Criteria					
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSD_R) (%) No more than	Recovery (%)	Example of Methods that meet the criteria
Edible Oils	Lead	0.08	0.045 - 0.115	0.016	0.032	44	60 -115	NMKL 186, ISO 12193, AOAC 994.02, AOCS Ca 18c-91
Olive oils and olive pomace oils	Iron	3.0	1.8 – 4.2	0.3	0.6	27	80 – 110	ISO 8294 AOAC 990.05 AOCS Ca 17a-18
Olive oils and olive pomace oils	Copper	0.1	0.03 – 0.17	0.01	0.02	44	80 – 110	ISO 8294 AOAC 990.05 AOCS Ca 17a-18

n.b. Edible Oils includes olive oil and olive pomace oil

Appendix II – Methods of analysis for Olive Oils and Olive Pomace Oils

Table 3 - PROPOSED DRAFT AMENDMENT/REVISION OF THE STANDARD FOR FISH OILS (CXS 329-2017): INCLUSION OF CALANUS OIL

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

Appendix III: CL 2024/08 - MAS Review of methods of analysis for Performance Criteria for Lead and Cadmium in Foods

Explanation to the Table:

- ~~Strikethrough~~ text is text recommended to be deleted.
- The columns: “Comments by” are meant for assistance in the CCMAS discussions and will not be included in CXS 234.
- Text in brackets [ ] is to be considered as comments and not to be included in CXS 234
- Member country/member abbreviations where required Colombia (Co.); Uruguay (Uru.), European Union (EU), Saudi Arabia (S.Ar.), Bazil (Br.), Peru (Pe), Philippines (Ph.), Indonesia (Ind.).

Red text – comments based on CX/MAS 24/43/3 Add.3, CRD05, and consolidation of

- ‘Performance criteria for lead in butter, edible casein and whey powders (developed by CCMAS41, adopted by CAC44 and included in CXS234) and
- CL 2024/08-MAS ‘Appendix II - Analytical methods for lead for review’

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Natural mineral waters	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%	EPA 200.8, ISO 17294-2, EN 17851, EN 14083	ICP-MS, ICP-MS, ICP-MS, GF-AAS
Infant formula, formula for special medical purposes intended for infants and follow-up formula	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851.	ICP-MS, ICP-MS, ICP-MS, ICP-MS.
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, GF-AAS ICP-MS.

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Secondary milk products (including Butter, edible casein products and whey powders)	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083, AOAC 999.11 (edible casein), ~~AOAC 972.25~~ ~~ISO/TS 6733~~ ~~IDF/RM 133,~~	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS AAS or GF-AAS, ~~AAS~~ ~~Spectroph-otometry~~ ~~(1,5-~~ ~~diphenylthiocarbazone)~~
Fruit juices, except juices exclusively from berries and other small fruits	lead	0.03	0.017 - 0.043	0.006	0.012	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, GF-AAS, ICP-MS
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%	EN 15763, EN 17851. NMKL 161	ICP-MS, ICP-MS., ICP- MS
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%	EN 15763, EN 17851, NMKL 161 EN 14083,	ICP-MS, ICP-MS, ICP-MS GF-AAS.

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	EN 15763, EN 17851, NMKL 161, EN 14083,	ICP-MS, ICP-MS, ICP-MS GF-AAS.
Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161, EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Edible fats and oils (including Fats and Oils (all); Named Vegetable Oils; Olive Oils and Olive Pomace Oils)	lead	0.08	0.045 - 0.115	0.016	0.032	44	60-115%	AOAC 994.02, AOCS Ca 18c-91, ISO 12193, EN 17851, NMKL 186 . NMKL 161	GF-AAS, GF-AAS, GF-AAS, ICP-MS, GF-AAS, ICP-MS.
Berries and other small fruits, except cranberry, currant, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161, EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Bulb vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Canned fruits	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Canned vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Fruits, except cranberry, currants, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Legume vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Meat and fat of poultry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 161 EN 14083	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2 AOAC 2013.06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083	ICP-MS, ICP-MS, ICP-MS GF-AAS
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS
Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	EN 15763, OIV-MA-AS323-07.	ICP-MS, ICP-MS
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%	EN 15763, OIV-MA-AS323-07.	ICP-MS, ICP-MS.
Pig, edible offal of	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS
Cattle, edible offal of	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS
Cranberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS. ICP-MS GF-AAS.
Currants	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Elderberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Wine (wine and fortified / liqueur wine) made from grapes harvested	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	EN 15763, OIV-MA-AS323-07.	ICP-MS, ICP-MS.

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Fish	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	AOAC 999.11, FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083	GF-AAS, ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS
Fresh farmed mushrooms (common mushrooms (*Agaricus bisporous*), shiitake mushrooms (*Lentinula edodes*), and oyster mushrooms (*Pleurotus ostreatus*))	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS GF-AAS.
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS
Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083	ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS
Table olives	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 161 EN 14083, AOAC 999.11 NMKL 139	ICP-MS ICP-MS, ICP-MS ICP-MS, ICP-MS, GF-AAS, AAS (Flame Absorption)
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%	~~EUsalt/AS 013~~, EUsalt/AS 015, EN 17851, EN 14083.	~~Flame AAS,~~ ICP-OES, ICP-MS, GF-AAS.
Natural mineral waters	cadmium	0.003	0.0017-0.0043	0.0006	0.0012	44	40-120%	EPA 200.8, ISO 17294-2, EN 17851, EN 14083.	ICP-MS, ICP-MS, ICP-MS, GF-AAS.
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Bulb vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%	FDA Method 4.7 Ver.1.2 AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	ISO 23637, EN 17851, NMKL 161 EN 14083.	GF-AAS ICP-MS, ICP-MS GF-AAS.
Legume vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS GF-AAS.

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS.
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	ISO 23637, EN 17851, NMKL 161 EN 14083	GF-AAS, ICP-MS, ICP-MS GF-AAS
Chocolate containing or declaring < 30% total cocoa solids on a dry matter basis	cadmium	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, GF-AAS

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	ISO 23637 EN 17851, NMKL 161 EN 14083.	GF-AAS ICP-MS, ICP-MS, GF-AAS.
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%	EUsalt/AS 015, EN 17851, EN 14083.	ICP-OES, ICP-MS, GF-AAS.
Chocolate containing or declaring >30% to <50% total cocoa solids on a dry matter basis	cadmium	0.7	0.35 - 1.05	0.07	0.14	34	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, GF-AAS
Chocolate containing or declaring ≥50% to <70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table chocolate	cadmium	0.8	0.40 - 1.20	0.08	0.16	33	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, GF-AAS

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Chocolate containing or declaring ≥70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table	cadmium	0.9	0.46 - 1.34	0.09	0.18	33	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, GF-AAS
Cephalopods	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, GF-AAS.
Marine bivalve molluscs (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%	EN 15763, EN 17851, NMKL 161 EN 14083.	ICP-MS, ICP-MS, ICP-MS, GF-AAS.

Appendix IV: General Methods for the Detection of Irradiated Foods for review and inclusion in CXS 234

Commodity	Provision	Method	Principle	Type
Food containing fat **(e.g. raw meat and chicken, cheese, fruits)**	Detection of irradiated food – **Detection of radiation-induced hydrocarbons**	EN 1784	Gas chromatographic analysis of hydrocarbons	II
Food containing fat **(e.g. raw meat and chicken, liquid whole egg)**	Detection of irradiated food – **Detection of radiation-induced 2-alkylcyclobutanones**	EN 1785	Gas chromatographic mass spectrometric analysis of 2-alkylcyclobutanones	III
Food containing bone	Detection of irradiated food – **Radiation induced Electron Spin Resonance (ESR) signal attributed to hydroxyapatite (principle component of bones)**	EN 1786	ESR spectroscopy	II
Food containing cellulose **(e.g. nuts and spices)**	Detection of irradiated food **– Radiation induced Electron Spin Resonance (ESR) signal attributed to crystalline cellulose**	EN 1787	ESR spectroscopy	II
Food containing silicate minerals **(e.g. herbs, spices, their mixtures, and shrimps)**	Detection of irradiated foods – **Thermoluminescence glow ratio used to indicate the irradiation treatment of the food**	EN 1788	Thermoluminescence	II
Food containing silicate minerals **(e.g. shellfish, herbs, spices, seasonings)**	Detection of irradiated foods – **Measurement of photostimulated luminescence intensity**	EN 13751	Photostimulated luminescence	III
Food containing crystalline sugar **(e.g. dried fruits and raisins)**	Detection of irradiated food **- Radiation induced Electron Spin Resonance (ESR) signal attributed to crystalline sugar**	EN 13708	ESR spectroscopy	II

Appendix V: Performance criteria for selected processed fruits and vegetables

Commodity	Provision	ML (mg/kg)	Method performance criteria					
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria
Jams, jellies, and marmalades	Benzoic Acid	1000	830 – 1170	100	200	11.3	95 – 105	ISO 5518, NMKL 124, AOAC 983.16
Pickled cucumbers	Benzoic Acid	1000	830 – 1170	100	200	11.3	95 – 105	NMKL 124, AOAC 983.16
Mango chutney	Benzoic Acid	250	197 – 302	25	50	13.9	90 – 107	ISO 5518, NMKL 124, AOAC 983.16
Coconut milk and coconut cream	Benzoic Acid	1000	830 – 1170	100	200	11.3	95 – 105	ISO 5518, NMKL 124, AOAC 983.16
Canned strawberries	Calcium	350	280 – 420	35	70	13.2	90 – 107	AOAC 968.31
Pickled cucumbers	Calcium	250	197 – 302	25	50	13.9	90 – 107	AOAC 968.31
~~Preserved tomatoes~~	~~Calcium~~							Rec retain current methods in CXS 234
~~Canned citrus fruits~~	~~Calcium~~							Rec retain current methods in CXS 234
~~Certain canned vegetables~~	~~Calcium~~							Rec retain current methods in CXS 234
~~Citrus Marmalade~~	~~Calcium~~							Rec retain current methods in CXS 234
Jams, Jellies, and Marmalades	Sorbates	1000	830 – 1170	100	200	11.3	95 – 105	NMKL 124, AOAC 983.16
Pickled Cucumbers	Sorbates	1000	830 – 1170	100	200	11.3	95 – 105	NMKL 124, AOAC 983.16
Processed Fruits and Vegetables	Tin	250	197 – 302	25	50	13.9	90 – 107	AOAC 980.19, NMKL 126, NMKL 191
Table Olives	Tin	250	197 – 302	25	50	13.9	90 – 107	NMKL 190, EN 15764, NMKL 126, NMKL 191

Appendix VI: Review of the workable package of methods for cereals, pulses and legumes

Outcomes of the PWG Discussion for Round 3 of the EWG-CPL

Cereals, Pulses and Legumes and Derived Products							
Commodity	**Provision**	**Codex Standard**	**Method**	**Principle**	**Type**	**Committee**	**Comments**
Degermed maize (corn) meal and maize (corn) grits	**Ash-550**	CXS 155-1985 (2019)	ISO 2171 and ICC 110/1	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	**Further discussion is required[1]**
	Ash-900		ISO 2171 / ICC 104/1 and ICC 110/1	Calculation from moisture and Gravimetry (incineration at 900°C)	I		
Durum wheat semolina and durum wheat flour	**Ash-550**	CXS 178-1991 (2019)	ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	**Further discussion is required[1]**
Gari	Particle size	CXS 151-1989 (2019)	ICC Recommendation 207	Sieving	I	CCCPL	**Recommended for endorsement, however, method requires inclusion of 1250 µm aperture sieve prior to endorsement**
Edible Cassava flour	Particle size	CXS 176-1989 (2019)	ICC Recommendation 207	Sieving	I	CCCPL	**Recommended for endorsement, however, method requires inclusion of 600 and 1200 µm aperture sieves prior to endorsement**
Pearl millet flour	**Ash-550**	CXS 170-1989 (2019)	AOAC 923.03 and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	**Further discussion is required[1]**

Cereals, Pulses and Legumes and Derived Products							
Pearl millet flour	Colour	CXS 170-1989 (2019)	ISO 16624	Diffuse reflectance Colorimetry (specific colour grader)	I	CCCPL	Recommended for endorsement
Quinoa	Moisture	CXS 333-2019 (2020)	ISO 712	Gravimetry (oven drying)	I	CCCPL	Recommended for endorsement
Quinoa	Protein	CXS 333-2019 (2020)	ISO 1871	Titrimetry (Kjeldahl digestion)	IV	CCCPL	Recommended for endorsement
Sorghum flour	**Ash-550**	CXS 173-1989 (2019)	ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	**Further discussion is required[1]**
	Ash-900		ISO 2171 / ICC 104/1 and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (incineration at 900°C)	I		
Sorghum flour	Colour	CXS 173-1989 (2019)	ISO 16624	Diffuse reflectance Colorimetry (specific colour grader)	I	CCCPL	Recommended for endorsement
Sorghum grains	**Ash-550**	CXS 172-1989 (2019)	ISO 2171and ISO 6540	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	**Further discussion is required[1]**
	Ash-900		ISO 2171 / ICC 104/1 and ISO 6540	Calculation from moisture and Gravimetry (incineration at 900°C)	I		

Cereals, Pulses and Legumes and Derived Products							
Soy protein products	**Ash-550**	CXS 175-1989 (2019)	ISO 2171and AOAC 925.09	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCVP	**Further discussion is required[1]**
Soy protein products	Fat	CXS 175-1989 (2019)	ISO 734	Gravimetry (extraction)	I	CCVP	Recommended for endorsement
Soy protein products	Crude Protein; excluding added vitamins, minerals, amino acids and food additives	CXS 175-1989 (2019)	AOCS Ba 4f-00 AACCI 46.30 ISO 16634-1	Gravimetry (Combustion)	IV IV IV	CCVP	Recommended for endorsement
Vegetable protein products	**Ash-550**	CXS 174-1989 (2019)	ISO 2171 and AOAC 925.09	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCVP	**Further discussion is required[1]**
Vegetable protein products	Fat	CXS 174-1989 (2019)	ISO 734	Gravimetry (extraction)	I	CCVP	Recommended for endorsement
Vegetable protein products	Crude Protein; excluding added vitamins, minerals, amino acids and food additives	CXS 174-1989 (2019)	AOCS Ba 4f-00 AACCI 46.30 ISO 16634-1	Gravimetry (Combustion)	IV IV IV	CCVP	Recommended for endorsement
Wheat flour	**Ash-550**	CXS 152-1985 (2019)	ISO 2171	Gravimetry (incineration at 550°C)	I	CCCPL	**Further discussion is required[1]**
	Ash-900		ISO 2171 / ICC 104/1	Gravimetry (incineration at 900°C)	I		

Cereals, Pulses and Legumes and Derived Products							
Wheat protein products including wheat gluten	**Ash-550**	CXS 163-1987 (2001)	ISO 2171 and AOAC 925.09	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCVP	**Further discussion is required[1]**
Whole and decorticated pearl millet grains	**Ash-550**	CXS 169-1989 (2019)	AOAC 923.03 and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	**Further discussion is required[1]**
Whole maize (corn) meal	**Ash-550**	CXS 154-1985 (2019)	ISO 2171 and ICC 110/1	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	**Further discussion is required[1]**
	Ash-900		ISO 2171 / ICC 104/1 and ICC 110/1	Calculation from moisture and Gravimetry (incineration at 900°C)	I		
Couscous	**Ash-550 or Ash-900**	CXS 202-1995	ISO 2171	Calculation from moisture and Gravimetry (incineration at 550°C or 900°C)	I	CCCPL	**Further discussion is required[1]**

[1]Recommended methods identified for Ash provision in several Commodity Standards include two temperatures for Ash (550°C, 900°C). Ash methods are Type I and only one Type I method for a single provision is in agreement with the Procedural Manual. This means including both temperatures under the provision "Ash" would not meet Procedural Manual requirements. As a result, two provisions for Ash have been proposed, to separate the temperature requirements to be consistent with Commodity Standards. Those commodities with only one temperature will need to have a consistent provision name (e.g., Pearl Millet flour – Ash-550°C, AOAC 923.03 is the only method identified, which is performed at 550°C, but "Ash" alone is inconsistent with the proposed Ash provisions)

Appendix VII: Working Package for Fish and Fishery Products – review of CXS 234

Explanation to the Table:

- ~~Strikethrough~~ text is text recommended to be deleted.
- **Bold** text is text recommended to be included.

Table 1 – Review of Methods and Method Typing

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
1	~~Fish and fishery products~~	~~Histamine~~	~~AOAC 977.13~~	~~Fluorimetry~~	~~II~~		Recommend deletion as method performance criteria applies, see below for Histamine.
2	~~Fish and fishery products~~	~~Mercury~~	~~AOAC 977.15~~	~~Flameless atomic absorption spectrophotometry~~	~~III~~		Recommend deletion as method performance criteria applies for mercury. **AOAC 977.15 for determination of mercury is included for the screening for methyl mercury, which is the provision in CCFP standards**
3	Fish and fishery products: canned products	Drained weight	Described in the Standard	Weighing	I	CXS 3-1981 CXS 37-1991 CXS 70-1981 CXS 90-1981 CXS 94-1981 CXS 119-1981	Recommend unchanged.
4	Fish and fishery products: canned products	Net weight	Described in the Standard	Weighing	I	CXS 3-1981 CXS 37-1991 CXS 70-1981 CXS 90-1981 CXS 94-1981 CXS 119-1981	Recommend unchanged.
5	~~Boiled dried salted anchovies~~	~~Sodium Chloride (chloride expressed as sodium chloride)~~	~~AOAC 937.09~~	~~Titrimetry~~	~~II~~	CXS 236-2003	Recommend deletion as method performance criteria is suggested for sodium chloride in this document, see below.

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
6	~~Canned shrimps or prawns~~	~~Size, determination of~~	~~Described in the Standard~~	~~Number per 100 g~~	~~I~~	CXS 37-1991	Recommend deletion of this entry and replacing it with the subsequent row to ensure consistency in both sensory and physical determinations for all fish and fishery products.
7	**Fish and fishery products**	**Sensory and Physical Determinations**	**Described in the Standard**	**Sensory analysis, Visual inspections, Counting**	**I**	All the CCFFP standards	Recommend inclusion of this entry as the provision is given in CCFFP standards, but not listed in the CXS 234.
8	Fish sauce	Total nitrogen	~~AOAC 940.25~~ **AOAC 978.02**	~~digestion~~ **Titrimetry (Kjeldahl digestion)**	I	CXS 302-2011	Recommend replacement of method due to the use of mercury. AOAC 940.25 (for seafood) refers to AOAC 955.04 (for fertilizers), which utilizes a mercury catalyst. Suggestion: AOAC 978.02 Nitrogen (Total) in Fertilizers, using copper sulphate or chromium metal as catalyst.
9	Fish sauce	Amino acid nitrogen	AOAC 920.04 and AOAC 920.03	determining formaldehyde titration method subtracting by ammoniacal nitrogen (magnesium oxide method)	I	CXS 302-2011	Recommend considering replacement of these methods, as AOAC 920.04 was Surplus 1970. In CXS 302: • AOAC 920.04 is referred **AOAC 2.066** • AOAC 920.03 is referred **AOAC 2.065** No method is suggested for amino acid nitrogen. The methods listed determines ammoniacal nitrogen. As there

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
							is no applicable method for the provision, we seek clarification from CCFFP for the intended use of this provision.
10	Fish sauce	pH	AOAC 981.12 The pH shall be measured in a sample of fish sauce diluted with water to 1:10 using a pH meter. The dilution of fish sauce is necessary because of the high ionic strength in the undiluted sauce.	~~Electrometry~~ **Potentiometry**	~~III~~ **IV**	CXS 302-2011	Recommend changing the Principle and Type. AOAC 981.12 is for vegetables and no validation information is included. If no validation data is available, Type IV is suggested.
11	**Fish sauce**	**pH**	**NMKL 179**	**Potentiometry**	**II**	CXS 302-2011	Recommend inclusion of this entry as NMKL 179 is validated and already in Codex as Type II. Dilutions series are described in the method.
12	~~Fish sauce~~	~~Sodium chloride~~	~~AOAC 976.18~~	~~Potentiometry~~	~~II~~	CXS 302-2011	Recommend deletion as method performance criteria is suggested for sodium chloride in this document, see below.
13	~~Fish sauce~~	~~Sodium chloride~~	~~AOAC 937.09~~	~~Titrimetry~~	~~IV~~	CXS 302-2011	Recommend deletion as method performance criteria is suggested for sodium chloride in this document, see below.
14	~~Fish sauce~~	~~Histamine~~	~~AOAC 977.13~~	~~Fluorimetry~~	~~II~~	CXS 302-2011	Recommend deletion as method performance criteria has been established, see below.

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
15	Frozen abalone (covered by glaze)	Net weight	AOAC 963.18	Gravimetry	I	CXS 312-2013	Recommend unchanged.
16	**Quick Frozen Raw Scallop Products**	**Net weight**	**AOAC 963.18**	**Gravimetry**	**I**	CXS 315-2014	Recommend inclusion of this entry, as it is described in CXS 315, but not in CXS 234.
17	**Quick Frozen Raw Scallop Products – Block Frozen Products**	**Drained weight**	**AOAC 967.13 ~~or AOAC 970.60~~ and Described in the Standard**	**Gravimetry**	I	CXS 315-2014	Recommend inclusion of this entry, as it is described in CXS 315, but not in CXS 234.
18	Frozen fish and fishery products	Thawing and cooking procedures	Described in the Standards	Thawing and heating	I	Several CCFFP standards	Recommend unchanged.
19	Quick frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Proportion of fish fillet and minced fish	AOAC 988.09	Physical separation	I	CXS 165-1989	Recommend unchanged. In CXS 165, the AOAC 988.09 is written in incorrectly (AOAC 988**,** 09)
20	Quick frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Net content of frozen fish blocks covered by glaze	Described in the Standard	Gravimetry	I	CXS 165-1989	Recommend unchanged.
21	~~Quick frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh~~	~~Sodium chloride~~	~~AOAC 971.21 (Codex general method)~~	~~Potentiometry~~	~~II~~	CXS 165-1989	Recommend deletion as this entry is not listed in CXS 165.
22	Quick frozen fish fillets	Net weight of products covered by glaze	Described in the Standard	Water spraying and sieving	I	CXS 190-1995	Recommend unchanged.
23	**Quick Frozen Fish Fillets**	**Gelatinous Condition (Determined as Moisture)**	**AOAC 983.18 and AOAC 950.46A**	**Gravimetry**	**I**	CXS 190-1995	Recommend inclusion of this entry, as it is described in CXS 190, but not in CXS 234.

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
24	Quick frozen fish sticks (fish fingers) and fish portions - breaded or in batter	Fish content (declaration)	AOAC 996.15 and calculation (described in the standard)	Gravimetry	I	CXS 166-1989	Recommend unchanged.
25	Quick frozen fish sticks (fish fingers) and fish portions - breaded or in batter	Net weight	Described in the Standard	Weighing	I	CXS 166-1989	Recommend unchanged.
26	Quick frozen fish sticks (fish fingers) and fish portions- breaded and in batter ~~(except for certain fish species with soft flesh)~~	Proportion of fish fillet and minced fish	WEFTA Method (Described in the Standard)	Gravimetry	I	CXS 166-1989	Recommend deletion of the phrase “except for certain fish species with soft flesh”, as it is not specified in CXS 166.
27	~~Quick frozen fish sticks (fish fingers) and fish portions - breaded or in batter~~	~~Sodium chloride~~	~~AOAC 971.27 (Codex general method)~~	~~Potentiometry~~	~~II~~	CXS 166-1989	Recommend deletion as this entry is not listed in CXS 166.
28	**Quick Frozen Fish Sticks (Fish Fingers), Fish Portions and Fish Fillets - Breaded or in Batter**	**Gelatinous Condition (Determined as Moisture)**	**AOAC 983.18 and AOAC 950.46A**	**Gravimetry**	**I**	CXS 166-1989	Recommend inclusion of this entry, as it is described in CXS 166, but not in CXS 234.
29	Salted Atlantic herring and salted sprat	Water content **(Determined as Moisture)**	AOAC 950.46B **a)**	~~Air drying~~ **Gravimetry**	I	CXS 244-2004	Recommend changing the Provision and Principle.
30	Salted fish **and dried salted fish** of the Gadidae family **of fishes**	Salt **content (Determined as Chloride expressed as Sodium Chloride)**	**AOAC 937.07 and Sodium Chloride see method criteria** ~~Described in CXS 167-~~**1989**	Titrimetry (Mohr) S~~alt determined as chloride expressed as sodium chloride~~	II	CXS 167-1989	Recommend changing the Commodity, Provision and Method according to the CXS 167.

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
31	Salted fish **and dried salted fish** of the Gadidae family **of fishes**	~~Salt Content~~ ~~Water content~~ **Moisture**	**AOAC 937.07 and AOAC 950.46B (airdrying a)** ~~Sampling and method Described in the Standard~~	Gravimetry, ~~titrimetric~~	I	CXS 167-1989	Recommend changing the Provision and Method. In CXS 167, AOAC 950.46B
31B	**Salted fish and dried salted fish of the Gadidae family of fishes**	**Salt saturation**	**See equation below**	**Calculation**	**I**		Included after discussion in the PWG

The % salt saturation is calculated as follows:

1. % salt in water = (%salt content / (%salt content + % moisture)) x 100%
2. % salt saturation = (% salt in water / 26.4%*) x 100%

*The solubility of sodium chloride in water is 36 g per 100 g water, and the constant is calculated as follows:

36 g sodium chloride / (100 g water + 36 g sodium chloride) x 100% = 26.4%

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
32	**Salted fish and dried salted fish of the Gadidae family of fishes**	**Water content in the whole fish**	**Described in the Standard**	**Gravimetry**	**I**	CXS 167-1989	Recommend inclusion of this entry, as it is described in CXS 167, but not in CXS 234. In CXS 234, No. 31 and No. 32 might be merged.
33	Smoked fish, smoke-flavoured fish and smoke-dried fish	Water phase salt **(salt determined as chloride expressed as sodium chloride)**	AOAC 952.08 **and** ~~AOAC 937.09~~ **Sodium Chloride see method criteria** ~~**Described** in Standard *~~ Water phase salt = (% salt × 100) / (%water + %salt)	**Gravimetry and Titrimetry (Mohr) and** Calculation	I	CXS 311-2013	Recommend changing the Provision, Method and Principle. In the column Method, the word “and” recommend added, as AOAC 952.08 and AOAC 937.09 are complementary methods for calculation of the provision.

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
34	Smoked fish, smoke-flavoured fish and smoke-dried fish	Water activity	NMKL 168 | ~~ISO 21807~~	Electrometry	III	CXS 311-2013	Recommend deletion of ISO 21807 as it is withdrawn. ISO 18787 is included in the subsequent row.
35	**Smoked fish, smoke-flavoured fish and smoke-dried fish**	**Water activity**	**ISO 18787**	**Electrometry**	**II**	CXS 311-2013	Recommend inclusion of the entry as ISO 18787 was endorsed for commodity 'Dried Meat' at CCMAS41 as a Type II method. The method is applicable for foodstuffs.
36	Sturgeon caviar	Salt content **(Determined as Chloride expressed as Sodium Chloride)**	**AOAC 937.07 and Method performance criteria** Described in CXS 167-1989	Titrimetry (Mohr) ~~Salt determined as chloride expressed as sodium chloride~~	II	CXS 291-2010	Recommend changing the Provision, Method and Principle. Sample prep. according to AOAC 937.07.
37	Live and raw bivalve molluscs	Paralytic shellfish toxicity	AOAC 959.08	Mouse bioassay	IV	CXS 292-2008	Recommend unchanged. Should preferably not be used due to animal welfare, however, CCMAS35 agreed to retain as a Type IV and again during CCMAS36.
38	Live and raw bivalve molluscs	Paralytic shellfish toxicity	AOAC 2011.27	Receptor binding assay	IV	CXS 292-2008	Recommend unchanged. Should preferably not be used due to animal welfare, however, CCMAS35 agreed to retain as a Type IV and again during CCMAS36.

No.	Commodity	Provision	Method	Principle	Type	Codex Standards	Comments
39	**Crackers from marine and freshwater fish, crustacean and molluscan shellfish**	**Crude Protein**	**AOAC 2001.11**	**Titrimetry (Kjeldahl Digestion)**	**I**	CXS 222-2001	Recommend inclusion of this entry, as it is described in CXS 222, but not in CXS 234. Recommend method AOAC 2001.11 Protein (crude) in animal feed, Forage (plant tissue), Grain, and oilseed. In CXS 222, AOAC 920.87 or 960.52 are included. Both methods include mercury/mercuric oxide, replacement is required due to hazardous chemicals.

Table 2 – **Method performance criteria for histamine for fish and fishery products**

Provision	ML (mg/100 g)	Minimum applicable range (mg/100 g)	LOD (mg/100 g)	LOQ (mg/100 g)	RSDR(%)	Recovery (%)	Applicable methods that meet the criteria	Principle
Histamine	10 (average)	8 – 12	1	2	16	90 – 107	AOAC 977.13 ~~/~~ NMKL 99, NMKL 196, **ISO 19343**	Fluorometri~~c~~**y**, HPLC-UV, **HPLC-UV** **HPLC-FLD**
Histamine	20 (each unit)	16 – 24	2	4	14	90 - 107	AOAC 977.13 ~~/~~ NMKL 99, NMKL 196, **ISO 19343**	Fluorometri~~c~~**y**, HPLC-UV. **HPLC-UV** **HPLC- FLD**

Determination of biotoxins in live and raw bivalve molluscs

The method selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use.

Criteria for determination of toxin analogues by chemical methods

Methods shall meet the numerical criteria listed in Table 1 and may either meet the minimum applicable range, or LOD and LOQ criteria listed.

Table 3. Criteria for determination of toxin analogues by chemical methods

Toxin group	Toxin	Minimum applicable range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD_R) (%) No more than	Recovery (%)	Applicable methods that meet the criteria
STX group	Saxitoxin (STX)	0.05 – 0.2	0.01	0.02	44	50 – 130	AOAC 2005.06 **(HPLC-FLD),** NMKL 182 **(HPLC-FLD),** EN 14526 **(HPLC-FLD),** AOAC 2011.02 **(HPLC-FLD),** NMKL 197 **(HPLC-FLD)** **Turner et al (2020) J.AOAC Int. Vol. 103, No. 2, p533-62 (uHPLC-MSMS)**
	NEO	0.05 – 0.2	0.01	0.02	44	50 – 130	
	dcSTX	0.05 – 0.2	0.01	0.02	44	50 – 130	
	GTX1	0.05 – 0.2	0.01	0.02	44	50 – 130	
	GTX2	0.1 – 0.5	0.03	0.06	38	50 – 130	
	GTX3	0.1 – 0.5	0.03	0.06	38	50 – 130	
	GTX4	0.05 – 0.2	0.01	0.02	44	50 – 130	
	GTX5	0.1 – 0.5	0.03	0.06	38	50 – 130	
	GTX6	0.1 – 0.5	0.03	0.06	38	50 – 130	
	dcGTX2	0.1 – 0.5	0.03	0.06	38	50 – 130	
	dcGTX3	0.1 – 0.5	0.03	0.06	38	50 – 130	

Toxin group	Toxin	Minimum applicable range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD_R) (%) No more than	Recovery (%)	Applicable methods that meet the criteria
	C1	0.1 – 0.5	0.03	0.06	38	50 – 130	
	C2	0.1 – 0.5	0.03	0.06	38	50 – 130	
	C3	0.5 – 1.5	0.1	0.2	32	50 – 130	
	C4	0.1 – 0.5	0.1	0.2	32	50 – 130	
OA group	OA	0.03 – 0.2	0.01	0.02	44	~~60 – 115~~ **70-130**	**EU-harmonised SOP using HPLC-MSMS (see** reference below*) **For other methods see references ****
	DTX1	0.03 – 0.2	0.01	0.02	44	~~60 – 115~~ **70-130**	
	DTX2	0.1 – 0.5	0.03	0.06	38	~~60 – 115~~ **70-130**	
Domoic a**c**id	DA	14 – 26	2	4	20	80 – 110 **70-130**	**EN 14176 (HPLC - UV) AOAC 991.26 (HPLC-UV)**
AZA group	AZA1	0.03 – 0.2	0.01	0.02	44	~~40 – 120~~ **70-130**	**EU-harmonised SOP using HPLC-MSMS (**See reference below*) **For other methods see references ****
	AZA2	0.03 – 0.2	0.01	0.02	44	~~40 – 120~~ **70-130**	
	AZA3	0.03 – 0.2	0.01	0.02	44	~~40 – 120~~ **70-130**	

~~Reference: http://aesan.msssi.gob.es/en/CRLMB/web/procedimientos_crlmb/crlmb_standard_operating_procedures.shtml Harmonised-SOP-LCMS-OAVersion4.pdf~~

***** **https://www.aesan.gob.es/en/CRLMB/docs/docs/metodos_analiticos_de_desarrollo/EU-Harmonised-SOP-LIPO-LCMSMS_Version5.pdf**

**** H.J. van den Top, A. Gerssen, P. McCarron, H.P. van Egmond. Quantitative determination of marine lipophilic toxins in mussels, oysters and cockles using liquid chromatography-mass spectrometry: inter-laboratory validation study. Food Additives & Contaminants: Part A , 2011, Vol. 28, Iss. 12.**

Total toxicity is estimated as the sum of the molar concentrations of detected analogu**e**s multiplied by the relevant specific toxicity equivalency factors (TEFs). Internationally scientifically validated TEFs must be used. The science behind TEFs is developing. Current internationally validated TEF's ~~will be found~~ are available on the FAO website. ~~Information on TEFs could be incorporated in this standard at a future date.~~

Methods should be validated and used for the relevant toxin analogues that may contribute to total toxicity. Currently known toxin analogues to consider are listed in Table 1. Where toxin analogues that are not listed in Table 1 are determined the competent authority must assess the contribution of these analog**ue**s to total toxicity whilst conducting further investigations.

Table 4 - Method Performance Criteria for ~~methods of analysis~~ screening and for determination of methylmercury*

Commodity	Provision	ML (mg/kg)	Min. Appl. Range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (%) No more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Tuna (all species)	methylmercury*	1.2	0.64 – 1.8	0.12	0.24	31	80 - 110	EN 16801 / **NMKL 202** **AOAC 977.15**** **NMKL 186** /** **AOAC 2013.06**/** **EN 15763/****	GC-ICP/MS **AAS-flame** **ICP-MS** **ICP-MS** **ICP-MS**
Alfonsino	methylmercury*	1.5	0.82 – 2.2	0.15	0.30	30	80 - 110	AOAC 988.11 EN 16801 / NMKL 202 **AOAC 977.15**** **NMKL 186** /** **AOAC 2013.06**/** **EN 15763/****	GC-electron capture GC-ICP/MS **AAS-flamme** **ICP-MS** **ICP-MS** **ICP-MS AS-flame**
Marlin (all species)	methylmercury*	1.7	0.95 – 2.5	0.17	0.34	30	80 - 110	AOAC 988.11 EN 16801/ **NMKL 202** **AOAC 977.15**** **NMKL 186** /** **AOAC 2013.06**/** **EN 15763/****	GC-electron capture GC-ICP/MS **AAS-flame** **ICP-MS** **ICP-MS** **ICP-MS**
Shark (all species)	methylmercury*	1.6	0.88 – 2.3	0.16	0.32	30	80 - 110	AOAC 988.11 EN 16801 / **NMKL 202** **AOAC 977.15**** **NMKL 186** /** **AOAC 2013.06**/** **EN 15763/****	GC-electron capture GC-ICP/MS **AAS-flame** **ICP-MS** **ICP-MS** **ICP-MS**
Orange roughy	methylmercury*	0.8	0.40 – 1.2	0.08	0.16	33	80 - 110	AOAC 988.11 EN 16801 / **NMKL 202** **AOAC 977.15**** **NMKL 186** /** **AOAC 2013.06**/** **EN 15763/****	GC-electron capture GC-ICP/MS **AAS-flame** **ICP-MS** **ICP-MS** **ICP-MS**

Commodity	Provision	ML (mg/kg)	Min. Appl. Range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (%) No more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Pink cusk-eel	methylmercury*	1.0	0.52 – 1.5	0.10	0.20	32	80 - 110	AOAC 988.11 EN 16801 / **NMKL 202** **AOAC 977.15**** **NMKL 186** / **AOAC 2013.06**/ **EN 15763/****	GC-electron capture GC-ICP/MS **AAS-flame** **ICP-MS** **ICP-MS** **ICP-MS**

*Countries or importers may decide to use their own screening when applying the ML for methylmercury in fish by analysing total mercury in fish. If the total mercury concentration is below or equal to the ML for methylmercury, no further testing is required, and the sample is determined to be compliant with the ML. If the total mercury concentration is above the ML for methylmercury, follow-up testing shall be conducted to determine if the methylmercury concentration is above the ML. The ML also applies to fresh or frozen fish intended for further processing.

** Method applicable for determination of mercury and can be used for screening of methyl mercury, see *.

[NEW!] Method Performance Criteria for Sodium Chloride and for Salt determined as Chloride expressed as Sodium Chloride

Commodity	Provision	ML* (%)	Min. Appl. Range (%)	LOD (%)	LOQ (%)	Precision (%) No more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Boiled dried salted anchovies	**Sodium Chloride and Salt determined as Chloride expressed as Sodium Chloride,**	**15 (NaCl)** **9.1 (Cl^-)**	**13.8 – 16.2** **8.3 - 9.9**	**1.5** **0.91**	**3.0** **1.8**	**5.3** **5.7**	**98-102** **98-102**	**NMKL 178** **AOAC 971.27** AOAC 937.09	**Potentiometric titration** **Potentiometric titration** Titration
Fish Sauce	**Salt determined as Chloride expressed as Sodium Chloride,**	**≥ 20 (NaCl) Minimum limit** **≥ 12 (Cl^-)**	**≥ 18** **≥ 11**	**2.0** **1.2**	**4.0** **2.4**	**5.1** **5.5**	**98-102** **98-102**	**NMKL 178** **AOAC 971.27** AOAC 976.18 AOAC 937.09	**Potentiometric titration** **Potentiometric titration** Titration Titration

* The criteria are established for NaCl, as ML are for NaCl. For conversion into Cl, the parameters should be divided by 1.6485 as NaCl(%) = Cl(%)·1.6485

For CCMAS43: 15 % = 15 g/100 g = 150000 mg/kg (NaCl) -> 150000/1.6485 mg/kg Cl^- = 90992 mg/kg Cl^-

20% = 20g / 100g (NaCl) -> 200000 /1.6485mg/kg (Cl^-) = 121322 mg/kg Cl^-

ML ≥ 0,1 in mg/kg	LOD mg/kg	LOQ mg/kg	From mg/kg	To mg/kg	Precision RSDR (%)
150000	**15000**	**30000**	**138025,958**	**161974,042**	**5,3**
90992	9099,2	18198,4	83160,869	98823,131	5,7
200000	20000	40000	184711,100	215288,900	5,1
121322	12132,2	24264,4	111322,969	131321,031	5,5

Recovery:

Conc.	Recovery (%)
100 g/100g	98 – 102
10 g/100g	98 – 102
1 g/100g	97 – 103
1 mg/g	95 – 105
100 mg/kg	90 – 107