

# CODEX ALIMENTARIUS COMMISSION

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Food and Agriculture  
Organization of the  
United Nations



World Health  
Organization

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REP24/MAS

**JOINT FAO/WHO FOOD STANDARDS PROGRAMME  
CODEX ALIMENTARIUS COMMISSION**

**Forty-seventh Session  
25-30 November 2024**

**REPORT OF THE 43rd SESSION OF THE  
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING**

**Budapest, Hungary  
14-18 May 2024**

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## SUMMARY AND STATUS OF WORK

Responsible Party	Purpose	Text/Topic	Code/Reference	Step(s)	Para(s)
CCEXEC86 CAC47	Adoption / Revocation / Amendments / Information	Methods of analysis / performance criteria / sampling plan for provisions in Codex standards	CXS 234-1999 CXS 193-1995	-	20(i) and (iii); 23(i); 26(i)
CCEXEC86 CAC47 Relevant commodity committees Codex Secretariat	Adoption / Information/ac tion	Inclusion of "Nitrogen to protein conversion factors" as an Annex/consider the revocation Nx values in the pertinent commodity standards	CXS 234-1999 pertinent commodity standards	-	76(i – iii and v)
CCEXEC86 CAC47 CCFH55	Revocation / Information	<i>General Methods for the Detection of Irradiated Foods</i>	CXS 231-2001	-	20(ii) and (v)
CCEXEC86 CAC47	Consideration / Advice	Provision of assistance to CCMAS on ashing temperature and/or acceptability to endorse two ash provisions for relevant cereals, pulses and legumes standards	CXS 152-1985 CXS 154-1985 CXS 155-1985 CXS 172-1989 CXS 173-1989 CXS 202-1995	-	23(ii)
CCCF18	Consideration / Action	Inclusion of an example method that would meet the numeric performance criteria for determining MLs for aflatoxins in certain cereals and cereals-based products	CXS 193-1995	-	16(i) and 20(v)
	Information / Consideration / Action	Inclusion of the numeric performance criteria for methyl mercury and total mercury in CXS 234.  To transfer all method numeric performance criteria in sampling plans in CXS 193 to CXS 234 once the review by CCCF is completed	CXS 234-1999 CXS 193-1995	-	12(iii), 16(ii) and 20(v)
	Information	Revised definition of the "decision rule" in the sampling plan for methylmercury in fish	CXS 193-1995	-	12(i ii) and 20(v)
CCFO29	Information	Retention of ISO 3596 and AOCS Ca 6b-53 for testing unsaponifiable matter in olive oils and olive pomace oils	CXS 234-1999	-	11(iii) and 20(v)
		Recommended methods of analysis for DAGs and PPP	CXS 33-1981	-	11(iii) and

Responsible Party	Purpose	Text/Topic	Code/Reference	Step(s)	Para(s)
		in olive oils and olive pomace oils for data generation and collection			20(v)
CCSCH8	Action / Reply	The test portion size and method for light seeds	Draft standard for spices derived from dried or dehydrated fruits and berries – small cardamom	-	10(iii) and 20(v)
		The method for curcuminoids content on dry basis (colouring power) and the appropriate provision name	Draft standard for dried and dehydrated roots, rhizomes and bulbs – turmeric	-	10(iv) and 20(v)
		The method for pungency, Scoville Heat Units and the appropriate provision name	CXS 353-2022	-	10(v) and 20(v)
		The method for mould visible	CXS 344-2021	-	10(vi) and 20(v)
CCFFP36	Action / Reply	To clarify the intended use of the provision for amino acid nitrogen	CXS 302-2011	-	26(ii)
CCNE12	Action / Reply	To consider the nitrogen conversion factor of 5.71 for tehena	CXS 259R-2017	-	76(iv)
Members / Observers PWG on endorsement CCMAS44	Replies / Consideration	Information for methods for protein in quinoa	CXS 234-1999	-	22(ii) and 23(iii)
EWG (Germany) PWG on endorsement CCMAS44	Review / Update	Fruit juices workable package	CXS 234-1999	-	29
EWG (Serbia, USA) PWG on endorsement CCMAS44	Review / Update	Cocoa products and chocolate workable package	CXS 234-1999	-	31(ii)
EWG (USA, Australia) PWG on endorsement CCMAS44 CCFA55	Action / information	Testing methods: nitrates and nitrites in certain food matrices	CXS 192-1995	-	44

Responsible Party	Purpose	Text/Topic	Code/Reference	Step(s)	Para(s)
EWG (USA, UK) PWG on endorsement CCMAS44 CCFL48	Action / Information	Testing methods: Precautionary allergen labelling	CXS 1-1985		50
New Zealand, Uruguay, Brazil, Australia, EU and IDF PWG on endorsement CCMAS44	Drafting / Discussion	Discussion paper on the application of the determination of moisture content in whey powder	CXS 234-1999	-	17 and 20(vii)
EWG (New Zealand / Germany) CCMAS44	Drafting / Discussion	Information document: e-book with sampling plan apps	CXG 50-2004		39(i)
		Review of sampling plans	CXG 234-1999		39(ii, iii)
EWG (Brazil, Chile) CCMAS44	Drafting / Consideration	Harmonization of names and format for principles	CXS 234-1999	-	55
Codex Secretariat	Publication	Revised Information Document " <i>Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234</i> "	-	-	76(vi) and 90(ii)

## LIST OF ABBREVIATIONS

AOAC	AOAC International (formerly known as Association of Official Agricultural Chemists)
AOCS	American Oil Chemists' Society
CAC	Codex Alimentarius Commission
CCAFRICA	FAO/WHO Coordinating Committee for Africa
CCASIA	FAO/WHO Coordinating Committee for Asia
CCNE	FAO/WHO Coordinating Committee for the Near East
CCCF	Committee on Contaminants in Foods
CCFA	Committee on Food Additives
CCFFP	Committee on Fish and Fishery Products
CCFH	Committee on Food Hygiene
CCFL	Committee on Food Labelling
CCFO	Committee on Fats and Oils
CCMAS	Committee on Methods of Analysis and Sampling
CCSCH	Committee on Spices and Culinary Herbs
CCEXEC	Executive Committee of the Codex Alimentarius Commission
CEN	European Committee for Standardization
CLs	Circular Letters
CRD	Conference room document
DAGs	1,2 diglycerides
EU	European Union
EWG	Electronic working group
FAO	Food and Agriculture Organization of the United Nations
IAM	Interagency Meeting
IDF	International Dairy Federation
IOC	International Olive Council
IS	Indian Standard
ISO	International Organization for Standardization
LOD	Limit of Determination
LOQ	Limit of Quantification
NFCISO	National Food Chain Safety Office (Hungary)
Nx	Nitrogen conversion factor
ML	Maximum level
PM	Procedural Manual
PPP	Pyropheophytin "a"
PWG	Physical working group
REU	Regional Office for Europe and Central Asia
SDO	Standards development organisations
USPC	United States Pharmacopeial Convention
VWG	Virtual working group
WG	Working group
WHO	World Health Organization

**LIST OF CONFERENCE ROOM DOCUMENTS  
(CRDs)**

<b>CRD No.</b>	<b>Agenda Item</b>	<b>Submitted by</b>
1	Division of Competence	European Union
2	3	Chair of the Virtual Working Group (USA)
3	3	Chair of the Physical Working Group (USA)
4	11	Chair of the Inter-Agency Meeting (MoniQA)
5	3, 10	European Union
6	3, 9	Philippines
7	7	Chambre de Commerce Internationale (ICC) and Association of European Coeliac Societies (AOECS)
8	10	Association of American Feed Control Officials (AAFCO), AOAC International, AACC International (C&G (AACC)), Chambre de commerce internationale (ICC), International Dairy Federation (IDF), International Fruit and Vegetable Juice Association (IFU), International Organization for Standardization (ISO), MoniQA Association, Nordic-Baltic Committee on Food Analysis (NMKL) and United States Pharmacopeial Convention (USP)
9	12	FAO
10	3	Codex Secretariat
11	3.1, 3.11, 3.2, 4.1, 4.2, 4.3, 5, 7, 8, 9, 10	Kenya
12 REV	3.3, 9	International Dairy Federation (IDF) and International Organization for Standardization (ISO)
13	3.1, 3.11, 3.2, 4.1, 4.2, 5, 7, 9, 10	Thailand
14	3.1, 3.2, 4.1, 4.2, 5, 8, 9, 10	Ghana
15	3.2, 5, 6	Singapore
16	4.2	Morocco
17	12	CCMAS Chairperson and Codex Secretariat
18	3.2, 4.1, 4.2, 5, 8, 9, 10	Nigeria
19	3.1, 4.1, 4.2, 5, 8, 9	Uruguay
20	4.2	Costa Rica
21	3	Brazil and Uruguay and supported by Argentina, Colombia, Costa Rica, Ecuador, Honduras, Panama, Paraguay and Peru
22	3.1	International Olive Oil Council (IOC)
23	4.1, 4.2, 4.3	Senegal
24	3.3	New Zealand
25	5	Cabo Verde
26	3.2	Nordic-Baltic Committee on Food Analysis (NMKL)
27	10	Codex Secretariat
28 REV	1, 2, 3.1, 3.11, 3.2, 3.3, 4.1, 4.2, 8, 9, 10	Chile
29	4.2	Jamaica



## INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling (CCMAS) held its 43rd Session, in Budapest, Hungary, from 14 to 18 May 2024, at the kind invitation of the Government of Hungary. The Session was chaired by Dr Attila Nagy, Director, National Food Chain Safety Office (NFCSO) and Dr Zsuzsa Farkas, Food Chain Data Scientist, Digital Food Institute acted as the Vice-Chairperson. The Session was attended by 54 Member countries, one Member organization and 14 Observer organizations. The list of participants is contained in Appendix I.

## OPENING OF THE SESSION

2. Dr Márton Nobilis, the State Secretary of the Ministry of Agriculture of Hungary, opened the session and extended his warmest welcome to all participants. Dr Nobilis highlighted that international food safety standards underscored the dual mandate of Codex in protecting the health of consumers while facilitating fair trade in food, and it was important to ensure that these standards were accessible and that they were up-to-date. Dr Nobilis also noted that the hybrid format of this Session was an innovation that allowed delegates to contribute to technical discussions from any location in the world.
3. Dr Haris Hajrulahovic, the World Health Organization (WHO) Representative and Head of Country Office, Ms Mary Kenny, Food Safety and Consumer Protection Officer, the Food and Agriculture Organization of the United Nations (FAO) Regional Office for Europe and Central Asia and Mr Steve Wearne, Chairperson of the Codex Alimentarius Commission (CAC) (via video message) also addressed the Committee.

## Division of Competence

4. CCMAS43 noted the division of competence between the European Union (EU) and its Member States, according to paragraph 5, Rule II of the Procedure of the CAC.

## ADOPTION OF THE AGENDA (Agenda Item 1)<sup>1</sup>

5. CCMAS43 adopted the Provisional Agenda as the Agenda for the Session and agreed to discuss the following items under other business and future work (Agenda item 12), subject to the availability of time:
  - The importance of harmonization of sampling and testing methods for the determination of microplastics in foods (CRD09); and
  - Areas of work for CCMAS in the future (CRD17).

## MATTERS REFERRED TO THE COMMITTEE BY THE CODEX ALIMENTARIUS COMMISSION AND OTHER SUBSIDIARY BODIES (Agenda Item 2)<sup>2</sup>

6. The Codex Secretariat introduced the item and recalled the matters from CAC46 and the 85th Session of the Executive Committee of the Codex Alimentarius Commission (CCEXEC85) were for information only and that the matters from the seventh Session of the Codex Committee on Spices and Culinary Herbs (CCSCH7) had been considered by the virtual working group (VWG) meeting on endorsement that met on 7 May 2024 and would be considered further under Agenda item 3.
7. CCMAS43:
  - i. noted the matters for information referred by CAC and CCEXEC;
  - ii. encouraged Members and Observers to actively engage in opportunities to contribute to discussions in CCEXEC and CAC on the Codex Strategic Plan 2026-2031 through the regional coordinators; and
  - iii. noted that the reply from CCSCH7 would be addressed under Agenda item 3 (Endorsement of methods of analysis and sampling).

## ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS AND SAMPLING PLANS IN CODEX STANDARDS (Agenda item 3)<sup>3</sup>

8. CCMAS43 considered the recommendations on methods of analysis and sampling proposed for endorsement and other related matters as presented in CRD02 and CRD03.
9. CCMAS43 made the following decisions which are also presented in Appendix II of the report as follows.

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<sup>1</sup> CX/MAS 24/43/1 REV

<sup>2</sup> CX/MAS 24/43/2

<sup>3</sup> CX/MAS 24/43/3

**ENDORSEMENT OF METHODS OF ANALYSIS: CCSCH AND CCFO (Agenda item 3.1)<sup>4</sup>**Codex Committee on Spices and Culinary Herbs (CCSCH7)

## 10. CCMAS43:

- i. endorsed the methods as recommended for endorsement in CRD03, Appendix I, Tables 1 - 9 for the following spices:
  - o small cardamom
  - o allspice, juniper berry and star anise
  - o turmeric
  - o dried or dehydrated ginger
  - o cloves
  - o dried basil
  - o saffron
  - o nutmeg
  - o dried or dehydrated chilli pepper and paprika
- ii. for purposes of consistency, typed the Method V-8 for relevant provisions for all the spices and culinary herbs in question as Type IV; and aligned provision names and principles where necessary;
- iii. with regard to pending issues from the PWG:
  - o endorsed ISO 939 (moisture in cardamom and allspice, juniper berry and star anise) over the corresponding ASTA method as only one Type I is permitted;
  - o noted that the IS 1907 is an Indian Standard for cardamom specifications. However, the specification regarding light seeds is already included in the draft Standard for spices derived from dried or dehydrated fruits and berries – small cardamom. The IS 1907 refers to the IS 1790 that is a standard for methods for spices and condiments, which determines a test portion from 100 g to 200 g depending on the nature of the material. Thus, CCSCH should be consulted regarding the test portion size and if the ISO 927 is applicable for the provision light seeds;
  - o endorsed ISO 6571 over AOAC 962.17 for volatile oils on dry basis (small cardamom and allspice, juniper berry and star anise) to be consistent with decisions for other spices and culinary herbs currently listed in the *General Standard for Method of Analysis and Sampling* CXS 234-1999);
- iv. did not endorse the methods for curcuminoids content on dry basis (colouring power) as the ISO 5566 does not measure curcuminoids directly, and that the methods measures the absorption of light at 425 nm and the converts that absorption to a curcuminoid content. CCMAS43 noted that it was possible for colouring adulterants to affect this measurement value and also noted that the provision name was inconsistent with other similar provisions such as “colouring strength (expressed as crocin)” in the *Standard for Dried Floral Parts – Saffron* (CXS 351-2021). CCMAS43 requested CCSCH to either:
  - o recommend a method that directly measures the curcuminoids and then to change the provision accordingly to “curciminoids”; or
  - o change the provision name to make it clear that the provision is “colouring power expressed as curcuminoids”;
- v. endorsed the ISO 3513 (pungency, Scoville Heat Units), but did not endorse the ASTA 21.3 (pungency, Scoville Heat Units) in dried chilli and paprika. Similar to the ISO 5566 method for colouring power, the ASTA 21.3 method quantifies capsaicin and oleoresins and then converts those concentrations into “pungency, Scoville Heat Units”. The conversion to Scoville Heat Units makes the method Type I (i.e. the provision, “pungency” is defined by the conversion factor of the method), but there can only be a single Type I method for a provision. CCMAS43 requests that CCSCH either:
  - o redefines the provision to “capsaicinoids” in which case the AOAC 995.03 could be recommended for endorsement as a Type II or Type III method; or
  - o responds to CCMAS that ASTA 21.3 is preferred over ISO 3513 in which case CCMAS

may revoke the ISO 3513 and replace the method with ASTA 21.3 as a Type I method.

- vi. endorsed the Method V-8 (mould visible – cloves) as Type IV but did not endorse the ISO 927 and requested that CCSCCH should confirm whether ISO 927 would be a better Type I method. This method had been endorsed for the same provision for certain other spices and culinary herbs (e.g. dried or dehydrated ginger, turmeric).

#### Committee on Fats and Oils (CCFO28)

#### 11. CCMAS43:

- i. endorsed or proposed revocation of methods for olive oils and olive pomace oils as recommended in CRD03, Appendix II, Table 1 and for fish oils (calanus oil) in Table.3;
- ii. agreed to revoke methods for iron and copper currently listed in CXS 234 in light of the agreed numeric performance criteria for these elements and inclusion of example methods (CRD03, Appendix II, Table 2);
- iii. considered pending issues from the PWG and took the following decisions:
  - o endorsed the ISO 660 (section 9.1) / AOACS Cd 3d-63 / COI/T.20 Doc. No. 34 (acidity, free (acid value)) as Type I, but did not endorse the AOCS Ca 5a-40 as Type IV, noting that there was no compelling reason to have a Type IV co-exist with a Type I methods;
  - o noted the intervention by International Olive Council (IOC) (as also presented in CRD22) on the role of IOC, their standards development and that IOC methods were easily accessible and free for use;
  - o requested revocation of COI/T.20/Doc.No.30 (erythrodiol + uvaol) and COI/T.20/Doc.No.8 (halogenated solvents, traces) as these methods were no longer available and revocation of IUPAC 2.101 (relative density) as a Type I method was already listed in CXS 234;
  - o did not endorse the COI/T.20/Doc.No.35 (peroxide value) as a Type I method already existed in CXS 234 and CCMAS43 generally did not endorse methods that use of hazardous reagents, such as chloroform and thus there was no compelling reason to have a Type IV co-exist with a Type I method;
  - o did not endorse the AOCS Ca 6a-40 (unsaponifiable matter) as the method is not known to be validated for olive oil and olive pomace oils. It was concluded that the ISO 3596 and AOCS Ca 6b 53 methods are validated for fish and soy oil which have a high level of unsaponifiable matter and currently listed in CXS 234 would be retained. It was suggested to consider the AOCS method should data be provided in future. CCMAS will inform CCFO accordingly;
  - o noted that the Committee was unable to endorse the methods for 1,2 diglycerides (DAGs) and pyropheophytin “a” (PPP) for inclusion in CXS 234 as there were no specifications for DAGs and PPP in the revised *Standard for Olive Oils and Olive Pomace Oil* (CXS 33-1981). However, considering the request from CCFO for these methods to enable data generation and collection to assist with further work on the development of specifications for these two compounds, CCMAS agreed to recommend the ISO 29822 and COI/T.20/Doc. No 32 (1,2 diglycerides) and ISO 29841 (pyropheophytin “a”) as methods fit for purpose to determine DAG and PPP in olive oils and olive pomace oils and to recommend the Codex Secretariat to include these methods in the CL which will be issued to request for data to support the development of the related specifications by CCFO.
  - o endorsed COI/T.20/Doc.No.28 in place of COI/T.20/Doc.No.18 (wax content) as Type II; and
  - o revised the commodity for the provision on wax content in CRD03, Appendix II, Table 3 to “fish oil (calanus oil)”.

#### **ENDORSEMENT OF SAMPLING PLAN: Codex Committee on Contaminants in Foods (CCCF17) (Agenda item 3.11)<sup>5</sup>**

##### **Endorsement of the Sampling Plan for Methylmercury in Fish**

#### 12. CCMAS43:

- i. endorsed the Sampling Plan as outlined in CX/MAS 24/43/3 Add.1 Appendix I) with a revised definition of the “Decision Rule.”
- ii. agreed to inform CCCF

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<sup>5</sup> CX/MAS 24/43/3 Add.1

- 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.*

- iii. integration of the numeric performance criteria for methyl mercury and total mercury in the sampling plan into the numeric performance criteria table developed in the fish and fishery product workable package for inclusion in CXS 234 (see Agenda item 4.2).

### **PERFORMANCE CRITERIA FOR METHODS FOR CERTAIN PROCESSED FRUITS AND VEGETABLES (Agenda Item 3.2)<sup>6</sup>**

#### 13. CCMAS43 agreed to:

- i. endorse the numeric performance criteria for:
  - benzoic acid in jams, jellies, and marmalades, pickled cucumbers, mango chutney; coconut milk and coconut cream;
  - sorbates in jams, jellies, and marmalades and pickled cucumbers;
  - tin in processed fruits and vegetables and table olives; and
- ii. retain the current methods in CXS 234 for calcium in preserved tomatoes, canned citrus fruits, certain canned vegetables, canned strawberries, pickled cucumbers and citrus marmalade.

### **MATTERS PENDING FROM CCMAS42 (Agenda Item 3.3)<sup>7</sup>**

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

- 14. CCMAS43 agreed with the suggested example methods and principles that would meet the performance criteria for lead and cadmium in foods in CRD03 Appendix III and made some additions to the example methods and corrections to the principles.

#### **Review of methods of analysis for irradiated foods in the *General Methods for the Detection of Irradiated Foods (CXS 231-2001)* and their incorporation into CXS 234**

#### 15. CCMAS43:

- i. endorsed the methods in CRD03 Appendix IV for inclusion in CXS 234; and
- ii. proposed to revoke *General Methods for the Detection of Irradiated Foods (CXS 231-2001)*.

#### **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**

#### 16. CCMAS43 agreed to:

- i. recommend to CCCF to include EN 17641 as an example method in the numeric performance criteria table contained in the sampling plan for aflatoxins in certain cereals and cereal-based products including foods for infants and young children in CXS 193 with the following footnote:

*The sampling plan specifies a test portion size of 25.0 g and EN 17641 uses a test portion size of 5.0 g. No alternative method was found that meets the performance criteria, therefore EN 17641 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.*

- ii. request CCCF to consider transferring all methods numeric performance criteria in sampling plans in CXS 193 to CXS 234 once the review of numeric performance criteria exercise in CCCF is completed.

#### **Methods of analysis for determination of moisture content in dried milk**

- 17. After some discussion taken into account relevant CRDs, CCMAS43 agreed to develop a discussion paper on the application of the determination of moisture content in whey powder by New Zealand, Uruguay, Brazil, IDF, Australia and the EU, to:

- i. gather the full data, including outliers, from studies on determination of moisture in whey powders according to the 102°C method and the data related with ISO 5537|IDF 26 validation and share within the group;

<sup>6</sup> CL 2024/18-MAS; CX/MAS 24/43/3 Add.2; CX/MAS 24/43/3 Add.3 (Comments of Brazil, Colombia, Costa Rica, Egypt, Guatemala, Guyana, Paraguay, Peru, Philippines, Saudi Arabia, Uruguay and IOC, NMKL)

<sup>7</sup> CL 2024/08-MAS; CX/MAS 24/43/3 Add.4 rev.1 (Comments of Australia, Chile, Ecuador, Egypt, Guatemala, Iraq, Peru, Philippines, United Arab Emirates and GOED, IOC, MoniQA Association, NMLK)

- ii. if it is necessary, gather additional comparison data on determination of moisture in whey powders according to both methods (102°C method contained in CXS 234 – Appendix III, ISO 5537|IDF 26), to be provided, and share within the group;
  - iii. evaluate, based on this data, if the 102°C method could be exceptionally listed as Type IV for determination of moisture in whey powders, with the note “Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method as described in Appendix III is listed as Type IV”; and
  - iv. provide a recommendation for consideration by the Physical Working Group (PWG) on Endorsement prior to CCMAS44.
18. CCMAS43 agreed not to continue the consideration of the application of the determination of moisture content in dairy permeate powder due to insufficient data.

### **Conclusion**

19. CCMAS43 noted that the methods for DAGs and PPP could be recommended for use but could not be included in CXS 234 and encouraged Members/Observers to inform their counterparts at national level who would be responsible for generation of data that these methods were the preferred methods of analysis (Appendix III, Part 2).
20. CCMAS43 agreed to:
- i. submit the methods of analysis and numeric performance criteria for sorbates and benzoic acid for selected processed fruits and vegetables for adoption/revocation by CAC47, incorporating them into CXS 234 and the sampling plan for methylmercury in fish for adoption by CAC47 for inclusion in the *General Standard for Contaminants and Toxins in Food and Feed* (CXS 193-1995) (Appendix II, Parts 1 and 2);
  - ii. request CAC47 to revoke the *General Methods for the Detection of Irradiated Foods* (CXS 231 -2001);
  - iii. update CXS 234 by including example methods and principles that meet the numeric performance criteria for lead and cadmium in foods and to forward them to CAC47 for information;
  - iv. advise CCCF of the recommendation to include an example method that will meet the numeric performance criteria table for determining MLs for aflatoxins in certain cereals and cereals-based products in the related sampling plans in CXS193-1995 (see para 16);
  - v. inform CCCF, the Codex Committee on Food Hygiene (CCFH), CCFO and CCSCCH of the respective decisions taken at the session (paras 10, 11, 12, 15, 16) and refer relevant requests to CCSCCH (para 10iii, iv, v, vi);
  - vi. develop a discussion paper on the application of the determination of moisture content in whey powder as identified in para. 17; and
  - vii. re-establish the PWG on methods endorsement chaired by Australia and co-chaired by the United States of America (USA) and Hungary, working in English, French and Spanish, to meet immediately prior to CCMAS44 to consider all methods of analysis and sampling submitted by Codex Committees for endorsement, including the proposals on the workable packages: Fruit juices and Cocoa products and chocolate (see Agenda item 4); methods deferred by this session; and any other matters referred by other Codex Committees or submitted by Members and Observers.

### **REVIEW OF METHODS OF ANALYSIS IN CXS 234 (Agenda Item 4)**

21. CCMAS43 recalled that the recommendations from the electronic working group (EWGs) on the three workable packages: fats and oils, cereals, pulses and legumes; and fruit juices had been considered by the PWG on endorsement. CCMAS43 considered the recommendations presented in CRD03.

### **CEREALS, PULSES AND LEGUMES WORKABLE PACKAGE (Agenda item 4.1)<sup>8</sup>**

22. CCMAS43:
- i. endorsed the methods for:
    - o particle size in gari and edible cassava flour (should be reviewed if the appropriate sieving sizes are not included in this method);

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<sup>8</sup> CL 2024/14-MAS; CX/MAS 24/43/4; CX/MAS 24/43/4 Add.1 (Comments of Burundi, Colombia, Ecuador, Egypt, Guatemala, Indonesia, Jamaica, Panama, Papua New Guinea, Paraguay, Peru, Philippines, Sierra Leone, ICUMSA and USP)

- colour in pearl millet flour and sorghum flour;
  - moisture in quinoa;
  - fat and crude protein in soy protein products and vegetable products.
- ii. endorsed the method for protein in quinoa as Type IV, noting that the typing could be reconsidered if more information were provided. CCMAS43 was informed that seven (7) countries had provided validation data for ISO 1871 for determination of protein in quinoa. Based on the data, the method could be retyped. It was explained that normally protein methods are endorsed as Type I or Type IV and that ISO 1871 had been validated for quinoa (a pseudocereal) and the validation data was not in question. However, to reconsider the typing, additional information would be required on the specific chemicals used for the catalysts, the different reagents and their concentrations that were used and what conditions for the method were used corresponding to the validation data provided. It was agreed that a circular letter could be issued requesting the additional information for consideration by the PWG on endorsement to determine if the method could be retyped as Type I;
- iii. did not endorse the methods for ash in sorghum flour, sorghum grains, degermed maize (corn) meal and maize (corn) grits, durum wheat semolina, durum wheat flour, pearl millet flour, sorghum flour, sorghum grains, soy protein products, vegetable protein products, wheat flour, wheat protein products including wheat gluten, whole and decorticated pearl millet grains, whole maize (corn) meal and couscous as there was no consensus on these methods; and noted that:
- the current methods for ash for the aforementioned commodities in CXS 234 would remain unchanged; and
  - further information on the intent of ashing temperatures in the relevant commodity standards, would be needed to assist CCMAS to endorse the proposed methods or approval would be needed to change the provisions for ash, e.g. ash at 550 °C and ash at 900 °C, to allow endorsement of both sets of methods as Type I.

### Conclusion

#### 23. CCMAS43 agreed to:

- i. submit the methods for adoption/revocation by CAC47 (Appendix II, Parts 1 and 2);
- ii. request advice from CAC47 on whether CCMAS could have assistance in determining the original intent for ashing temperature in cereals, pulses and legumes standards, and if not, whether two provisions for ash at 550°C and 900°C for some commodities (i.e. CXS 152, CXS 154, CXS 155, CXS 172, CXS 173, CXS 202) could be acceptable to allow CCMAS to endorse recommended methods for these provisions as Type I; and
- iii. request the Codex Secretariat to issue a circular letter requesting the information outlined in para. 22 for consideration by the PWG on endorsement.

### FISH AND FISHERY PRODUCTS WORKABLE PACKAGE (Agenda item 4.2)<sup>9</sup>

#### 24. CCMAS43:

- i. agreed to remove methods for which there were no provisions in corresponding fish and fishery product standards; or for which numeric performance criteria had been developed;
- ii. endorsed the methods as recommended in CRD03 Appendix VII, Table1 and took the following decisions:
  - the AOAC 920.04 and AOAC 920.03 methods for the determination of amino acid nitrogen in fish sauce, currently listed in CXS 234 were retained unchanged as no replacement methods could be found at this stage; and
  - noted that these AOAC methods determined ammoniacal nitrogen and not amino acid nitrogen and agreed to request clarification from CCFFP for the intended use of the provision in order to determine more appropriate methods for this provision.

#### Numeric Performance Criteria

- i. agreed to retain the numeric performance criteria for histamine as contained in CXS 234 unchanged and included additional example methods and principles to the existing list that meet the numeric

<sup>9</sup> CL 2024/15-MAS; CX/MAS 24/43/5; CX/MAS 24/43/5 Add.1 (Comments of Australia, Brazil, Ecuador, Egypt, Guatemala, Indonesia, Iraq, Jamaica, Japan, Norway, Panama, Paraguay, Peru, Philippines, Sierra Leone, United Arab Emirates and NMKL, USP)

performance criteria (see CRD03 Appendix VII, Table.2) as a consequence, the typed methods listed in CXS 234 were recommended for revocation;

- ii. corrected the recovery (%) in the numeric performance criteria for determination of toxin analogues by chemical methods (biotoxins in live and raw bivalve mollusc) and included additional example methods to the list of methods that meet the numeric performance criteria (see CRD03 Appendix VII, Table 3); and
- iii. endorsed the numeric performance criteria for sodium chloride and for salt determined as chloride and expressed as sodium chloride and agreed with the example methods that meet the performance criteria (see CRD03 Appendix VII, Table 4). As a consequence, the typed methods listed in CXS 234 were recommended for revocation.

25. CCMAS43 noted that with these decisions work on the fish and fishery products had been concluded and thanked Norway, chair of the EWG and members of the EWG for their contribution.

#### **Conclusion**

26. CCMAS43:

- i. agreed to submit the methods of analysis and new and/or amended performance criteria for adoption / revocation/information by CAC47 (Appendix II, Parts 1 and 2); and
- ii. to request CCFPP to clarify the intended use of the provision for amino acid nitrogen in the *Standard for Fish Sauce* (CXS 302-2011).

#### **FRUIT JUICES WORKABLE PACKAGE (Agenda item 4.3)<sup>10</sup>**

27. Germany presented this item, highlighting the inactive status of CEN/TC174 responsible for developing methods of analysis for fruit and vegetable juices, including the 30 EN methods listed in CXS 234 and currently only IFU methods remained available. Germany suggested re-establishing the EWG to review the methods in the fruit juices workable package, stressing the need to: (i) decide whether to start with the remaining methods or adopt a method performance criteria approach; (ii) remove all EN methods from the testing methods in fruits juices; and (iii) ensure access to IFU methods for all EWG members to verify their accuracy and description.
28. IFU clarified that old CEN methods listed in CXS 234 and the *General Standard for Fruit Juices and Nectars* (CXS 247-2005) were no longer available, but conventional methods were identical (*/*) to IFU ones. While some isotopic methods were withdrawn, IFU agreed with CEN to publish three as IFU methods. IFU expressed their willingness to assist the EWG in evaluating methods for fruit juice quality and authenticity.

#### **Conclusion**

29. CCMAS43 agreed to re-establish the EWG, chaired by the Germany, working in English to review the workable package for fruit juices for consideration by CCMAS44.
30. The report of the EWG should be made available to the Codex Secretariat at least three months prior to CCMAS44.

#### Other matters

31. In view of the completion of the review of the cereals, pulses and legumes and fish and fishery products workable packages, CCMAS43 agreed to:
- i. start the review of methods in the cocoa products and chocolate workable package; and
  - ii. establish an EWG chaired by Serbia and co-chaired by the United States of America, working in English to review the cocoa products and chocolate workable package.
32. The report of the EWG should be made available to the Codex Secretariat at least three months prior to CCMAS44.

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<sup>10</sup> CX/MAS 24/43/6

**INFORMATION DOCUMENT: GENERAL GUIDELINES ON SAMPLING (CXG 50-2004) – E-BOOK WITH SAMPLING PLANS APPLICATIONS (Agenda item 5)<sup>11</sup>**

33. New Zealand, chair of the EWG, speaking also on behalf of the co-chair, Germany, introduced the item and recalled that the information document would support the implementation of the revised *General Guidelines on Sampling* (CXG 50 -2004). The EWG explained the work done in the EWG, its recommendations and a summary of the comments submitted to CL2024/16-MAS. She emphasized that further work was needed on the document for consideration and finalisation by CCMAS44, and that further guidance was needed on what should be covered by the document in order to meet the needs of members (i.e. have more structured approach to the document, type of examples to be included, what other information is need, or not need). The EWG chair also informed the Committee that a workshop had been held on the application of the sampling plan applications to help inform on further work on the information document to explain some of the statistical concepts behind the applications and a recording of the workshop would be available on the Codex website for further consultation.
34. The EWG chair also informed the Committee that a proposal had been made in the EWG to review sampling plans in CXS 234. While this matter was not related to the work on the information document, it would be useful for CCMAS to consider whether such a review should be done.

**Discussion****Information document**

35. CCMAS43 noted the general support to continue the development of the information document and noted the additional comments as follows:
- i. inclusion of real-life examples for basic products should be considered for inclusion in the document;
  - ii. the information document is a valuable source of information to design sampling plans but contains concepts which go beyond CXG50. The main users of the information document would be Codex committees who are tasked with designing acceptance samples plans for certain provision/commodity combinations. Therefore a more user-friendly, focussed approach on how to develop appropriate sampling plans using shiny apps for a number of such combination would better service these users.
  - iii. consideration should be given to how to integrate heterogeneous food lots (e.g. treenuts or grains) into the apps and whether there is a general approach to develop sampling plans based on heterogeneous food lots.
  - iv. for heterogeneous lots, there are different options possible, e.g. the more classical ISO approach or the Bayesian approach.
36. The EWG chair explained that heterogeneous lots were within the domain of sampling plans for bulk materials and there were statistical ways to describe heterogeneity within a lot, however, the intention of the document was to keep it as general as possible, but that this matter could be considered further in the future. On the issue of the different approaches (i.e. classical / Bayesian approaches), more investigation was needed before it could be decided whether to go with the classical approach or the Bayesian approach or even both approaches.
37. A Member noted that for the development of maximum levels (MLs) by the Codex Committee on Contaminants in Foods (CCCF), occurrence data were used to assess distribution of a contaminant in heterogeneous lots. Lots of occurrence data were available and could be used to determine the distribution in a lot for development of sampling plans without doing more specific work. This member proposed that CCMAS work closely with CCCF to understand how to use these data to design better sampling plans.

**Review of sampling plans contained in CXS 234**

38. CCMAS43 noted support to review the sampling plans contained in CXS 234 and that a discussion paper could be prepared in this regard.

**Conclusion**

39. CCMAS43 agreed to establish an EWG chaired by New Zealand and co-chaired by Germany, working in English, to:
- i. continue developing the information document taking into account the discussion at CCMAS43 and all written comments submitted to the meeting for comments and consideration by CCMAS44; and

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<sup>11</sup> CL 2024/16-MAS; CX/MAS 24/43/7; CX/MAS 24/43/7 Add.1 (Comments of Australia, Canada, Ecuador, Egypt, European Union, Indonesia, Iraq, Japan, Peru, Philippines, Sierra Leone)



- ii. develop a discussion paper on the review of all sampling plan in CXS 234, to:
  - a) determine what information should be included in CXS 234, and in the format of this information.
- iii. The discussion paper:
  - a) will provide review current procedures for the inclusion of sampling plans in CXS 234; and
  - b) consider sampling plan information that may be included in CXS 234 for sampling plans that will be developed under CXG 50-2004 and sampling plans from other sources noting that sampling plans are subject to endorsement by CCMAS prior to inclusion in CXS 234.

**NUMERIC PERFORMANCE CRITERIA FOR THE DETERMINATION OF NITRATE AND NITRITE IONS IN CERTAIN FOOD MATRICES (Agenda item 6)<sup>12</sup>**

- 40. The United States of America (USA), speaking also on behalf of Australia, introduced the item, highlighting the document was prepared in response to some questions from the Codex Committee on Food Additives (CCFA). The USA suggested considering the recommendations in the document and continuing work on the matter due to late availability of the document and incomplete response to CCFA's questions.
- 41. A Member proposed re-evaluating the precision value (RSD<sub>R</sub>) (%) for nitrite in Food Category 01.6.4, suggesting it might be 20.4 instead of 18.4 and requested further verification.
- 42. The Codex Secretariat explained that after multiple sessions' discussions, CCFA51 decided to establish in-going and residual levels for nitrates and nitrites in the *General Standard for Food Additives* (GSFA, CXS 192-1995). Subsequently, CCFA52 requested CCMAS to address specific questions related to testing methods, including development methods for determining proposed residual levels for representative provisions in dairy (cheese), meat, and seafood. It was emphasized that the recommendations from CCMAS were crucial for CCFA's ongoing work and that the discussion paper (CX/MAS 24/43/8) did not comprehensively address CCFA's questions (e.g. the numeric performance criteria for methods to determine the proposed residual levels for nitrate and nitrite).
- 43. CCMAS43 noted that further work was needed to respond to CCFA's questions and agreed to continue its efforts on this matter.

**Conclusion**

- 44. CCMAS43 agreed:
  - i. to re-establish the EWG chaired by the USA, and co-chaired by Australia, working in English only, with the following terms of reference:
    - a) to establish numeric performance criteria for the determination of nitrate and nitrite ions in the food matrices listed in CX/FA 21/52/7 Appendix 5, Annex 2 including adopted MLs in the GSFA and the lowest proposed residual levels;
    - b) to review the methods in CX/FA 21/52/7 Appendix 5, Annex 1 and determine if these methods meet the numeric performance criteria established for the matrices in CX/FA 21/52/7 Appendix 5, Annex 2 for both adopted MLs in the GSFA and lowest proposed residual levels;
    - c) to discuss if the methods determine both nitrate and nitrite ions and if so, whether the methods detect each ion separately or only in combination; and
    - d) to discuss if the different determination schemes (i.e. separate or combined) could have an impact on the precision and accuracy of the methods.
  - ii. the report of the EWG should be made available to the Codex Secretariat at least three months before CCMAS44; and
  - iii. to inform CCFA that CCMAS was continuing its work on this matter.

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<sup>12</sup> CX/MAS 24/43/8

**METHODS OF ANALYSIS FOR PRECAUTIONARY ALLERGEN LABELLING (Agenda item 7)<sup>13</sup>**

45. The USA, chair of the EWG, speaking also on behalf of the co-Chair, the United Kingdom, introduced the item, and recalled the decision of CCMAS42 to establish an EWG to develop a discussion paper to respond to the request from CCFL47 to support their work on precautionary allergen labelling. The EWG Chair explained the work process in the EWG and that the EWG has prepared a discussion paper which included a list of methods used worldwide. He explained that there was no consensus on harmonized methods used and that the EWG had not considered whether the methods were fit for purpose, i.e. they were not evaluated against performance characteristics or validation guidelines. The paper further identified terminologies and definitions for allergen testing and that consideration should be given to whether secondary confirmatory methods would fit within the Codex typing system should they eventually require endorsement and inclusion in CXS 234.
46. The EWG Chair noted that the discussion paper was a first step in responding to the request of CCFL, but that further work would be required to provide a full reply to CCFL.

**Discussion**

47. CCMAS43 noted the general support to continue work in the EWG, and that the methods in the Appendix I to CX/MAS 24/43/9 (which also included confirmatory methods) was a good starting point for evaluation against the CEN performance characteristics and the AOAC validation guidelines.
48. An Observer supported further work, agreed that the methods listed in the discussion paper should be validated and noted that work on methods for gluten containing cereals and wheat should be treated together and not separately.
49. On an observation that should CCFL request endorsement of immunological methods from the list, difficulties with typing might arise, the EWG Chair explained that the discussion paper had tried to address this issue, but that there were problems to fit these methods within the current Codex typing system. He proposed that CCMAS first focus on responding to the current request from CCFL and if the question of endorsement and typing of methods came up in the future, the matter could be considered then.

**Conclusion**

50. CCMAS43:
- i. noted that the discussion paper did not fully answer the questions from CCFL and agreed to re-establish the EWG, chaired by the United States of America, and co-chaired by the United Kingdom, working in English to:
    - a) request Members to submit validation data of the methods listed in Appendix I of CX/MAS 24/43/9;
    - b) evaluate the submitted validation studies through published method validation guidelines from AOAC<sup>14</sup>,<sup>15</sup> and CEN performance requirements<sup>16</sup>; and
    - c) submit a list of methods that meet either one or both of the AOAC validation guidelines and CEN performance requirements.
  - ii. recalled that the priority allergens for those listed as follows:
    - Cereals containing gluten
    - wheat and other *Triticum* species
    - rye and other *Secale* species
    - barley and other *Hordeum* species and products thereof;
    - Crustacea
    - Fish

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<sup>13</sup> CX/MAS 24/43/9

<sup>14</sup> Abbott M, Hayward S, Ross W, Godefroy SB, Ulberth F, Van Hengel AJ, Roberts J, Akiyama H, Popping B, Yeung JM, Wehling P, Taylor SL, Poms RE, Delahaut P. Validation procedures for quantitative food allergen ELISA methods: community guidance and best practices. J AOAC Int. 2010 Mar-Apr;93(2):442-50. PMID: 20480889.

<sup>15</sup> Dr. Latimer, George W, Jr. (ed.), 'Validation Procedures for Quantitative Food Allergen ELISA Methods: Community Guidance and Best Practices', in Dr. George W Latimer, Jr. (ed.), Official Methods of Analysis of AOAC INTERNATIONAL, 22nd Edition (New York, 2023; online edn, AOAC Publications, 4 Jan. 2023).

<sup>16</sup> EN 17855:2024(Main) Foodstuffs - Minimum performance requirements for quantitative measurement of the food allergens milk, egg, peanut, hazelnut, almond, walnut, cashew, pecan nut, brazil nut, pistachio nut, macadamia nut, wheat, lupine, sesame, mustard, soy, celery, fish, molluscs and crustaceans.

- Hazelnut
  - Sesame
  - Milk
  - Egg
  - Peanut
  - Cashew
  - Walnut
- iii. agreed that the report of the EWG should be made available to the Codex Secretariat at least three months prior to CCMAS44;
- iv. confirmed the decision of CCMAS42 that the EWG would not address sampling plans<sup>17</sup>;- and
- v. agreed to inform CCFL48 of the status of work and the decisions of the Committee.

**HARMONIZATION OF NAMES AND FORMAT FOR PRINCIPLES IDENTIFIED IN CXS 234 (Agenda item 8)<sup>18</sup>**

51. Brazil introduced the item and recalled the lack of consistency in determining what information should be captured in the principles outlined in CXS 234. Consequently, CCMAS42 decided that Brazil would prepare a discussion paper to consider harmonization of the name and format for the principles and provision names identified in the CXS 234.
52. To guide the work, Brazil suggested that the name of the principle reflects only descriptions of techniques directly relevant to determining the test result. The techniques used for sample preparation, extraction and separation, for example, would not be included as they are already specified within the method and thus not considered part of the principle-name. In preparing the discussion paper it was realised that some definitions were needed regarding descriptions of the analytical methods (e.g. colorimetric, sensory, etc.) to help in future work on this topic.
53. Brazil explained the criteria used to develop harmonized principles and based on these criteria; proposals were made for harmonized principles (Appendix I of CX/MAS 24/43/8).
54. Noting that further work was needed to build on the recommendations in the discussion paper, Brazil proposed that CCMAS43 should establish an EWG to continue developing the discussion paper and to provide definitions for descriptions of analytical methods; harmonized names and format for the principle and provision names and to present these revised harmonized principles and provision names in CXS 234 for consideration by CCMAS44.

**Conclusion**

55. CCMAS43 agreed to establish an EWG chaired by Brazil and co-chaired by Chile, working in English, to further develop:
- i. definitions for descriptions of analytical methods;
  - ii. harmonized names and format for principles and provision names in CXS 234-1999; and
  - iii. prepare a revised CXS 234-1999 presenting the proposed harmonized principles and provision names.
56. The report of the EWG should be made available three months prior to CCMAS44.

**APPROACH FOR THE PLACEMENT OF NITROGEN CONVERSION FACTORS (Agenda item 9)<sup>19</sup>**

57. Chile, speaking also on behalf of Brazil, introduced the item, and explained that the determination of the protein content in foods is based on the quantification of the nitrogen contained in the foods which is calculated by applying a nitrogen conversion factor (Nx). It was therefore necessary to be clear about the Nx that should be used. Therefore, considering practicality and accessibility, it was recommended to include in CXS 234-1999 an annex in which information on Nx that have been determined by Codex committees could be accessible.
58. Chile further highlighted other recommendations in CX/MAS 24/43/11, including the need to confirm Nx for certain commodities.

<sup>17</sup> REP23/MAS, para. 17

<sup>18</sup> CX/MAS 24/43/10

<sup>19</sup> CX/MAS 24/43/11

**Discussion**

59. CCMAS43 considered the recommendations outlined in CX/MAS 24/43/11 and noted the following views.
- Positioning of nitrogen conversion factors in Codex texts
60. CCMAS43 noted there was general support to include Nx as an Annex to CXS 234, as this would provide clarity and accessibility, especially to food laboratories that would need to determine the protein content of samples using methods that require calculation.
61. An Observer, while supporting an annex to CXS 234, noted that the proposed Annex would need to be modified whenever a Nx was updated in the relevant standard, and expressed their preference for the Nx values to be presented as a list as reflected in CX/MAS 24/43/11 Appendix II. The Observer expressed that it would not support the harmonization of the provision name if CCMAS decided to present the Nx values in a table, as the necessary consequential change to the provision names in the commodity standards could cause confusion.
62. CCMAS43 agreed to amend "Vegetable Protein Source" to "Plant Protein Source" and to include the Nx value for fishery products - crackers from marine and freshwater fish, crustacean and molluscan shellfish as 6.25.
- Nitrogen conversion factors for commodities
- Milk products*
63. CCMAS43 agreed to the proposed Nx value of 6.38 for milk and milk products. A view was expressed in support of having this standardized nitrogen conversion factor as this could help regulatory bodies ensure compliance amongst producers of the commodities.
- Dried meat (CXS 350R-2022) and cooked cured pork shoulder (CXS 97-1981)*
64. CCMAS43 agreed to the proposed Nx value of 6.25 for these commodities. It was also noted that for dried meat, the Nx value of 6.25 had already been proposed by the FAO/WHO Regional Coordinating Committee for Africa (CCAFRICA) when the committee submitted the method for endorsement by CCMAS (REP22/AFRICA, Appendix III).
- Infant formula*
65. An Observer highlighted that the wording related to Nx was different from that of the recently revised *Standard for Follow-up formula for Older Infants and Product for Young Children* (CXS 156-1987). It was however clarified that the wording was maintained as reflected in the *Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants* (CXS 72-1981) and in the current related footnote in CXS 234.
- Tempe (CXS 313R-2013)*
66. CCMAS43 agreed to the proposed Nx value of 5.71 and noted that this value had already been proposed by the FAO/WHO Regional Coordinating Committee for Asia (CCASIA) when the committee submitted the method for endorsement by CCMAS (REP13/ASIA, Appendix II).
- Tehena (CXS 259R-2017)*
67. Noting the absence of an Nx value in both CXS 259R-2017 and the pertinent report of the FAO/WHO Regional Coordinating Committee for the Near East (CCNE), CCMAS43 agreed to request CCNE to consider the proposed Nx value of 5.71.
- Other matters*
68. CCMAS43 exchanged views on possible ways to avoid having commodity standards that lacked Nx values moving forward. One Member highlighted that it was not the mandate of CCMAS, but that of commodity committees to set Nx values. As Nx values would depend on the characteristics of the commodity and it was possible that a different value could be used as long as it was agreed upon among traders, the Member suggested that CCMAS should simply request commodity committees to consider establishing Nx values for relevant commodities that lacked such a value rather than propose any Nx values.
69. CCMAS43 agreed with the Member's proposal. It was decided to inform the commodity committees about this matter and update the Information Document "Comprehensive Guidance for the Process of Submission, Consideration, and Endorsement of Methods for Inclusion in CXS 234" accordingly.
70. CCMAS43 also agreed to recommend that the commodity committees consider revoking the Nx values in their standards to ensure they were consolidated in one location (i.e. CXS 234) and to prevent potential inconsistencies.
71. A Member, while supporting the inclusion of nitrogen conversion factors as an Annex to CXS 234, did not support the consequential revocation of the factors from commodity standards.

Harmonization of provision names (e.g. "Protein" vs "Protein content")

72. CCMAS43 did not consider this matter under this agenda item considering the decision in para 55 to further develop harmonized provision names in CXS 234.

Expression of Nx values

73. There was general support for nitrogen conversion factors to be expressed in two decimal places.
74. However, some Members suggested that it should remain the prerogative of commodity committees to agree on the number of decimal places as Nx values in the commodity standard were determined by them. It was noted, nevertheless, that it was appropriate for CCMAS to recommend two decimal places for Nx as this has been the practice in standard literature. Recalling that the Nx value was used to multiply the analytical result to obtain the protein content, expressing it in two decimal places would also minimise rounding errors in the determination of protein content.
75. Members also supported the use of the "dot" convention in indicating decimal points. While one Member noted that it was a legal requirement in their country to use the "comma" convention instead, the Chairperson explained that the dot convention was used in international standards and Members would be free to make formatting changes in their national standards as appropriate.

**Conclusion**

76. CCMAS43 agreed to:
- i. forward the document titled "Nitrogen to protein conversion factors" for adoption as an Annex to CXS 234-1999 by CAC47 (Appendix II, Part 3);
  - ii. inform the relevant commodity committees about the "Nitrogen to Protein Conversion Factor" document and remind them that it was their responsibility to identify and report the proposed nitrogen conversion factors to CCMAS to facilitate the endorsement process;
  - iii. recommend that relevant commodity committees consider revoking Nx values in their commodity standards;
  - iv. request that CCNE consider whether it was appropriate to set the Nx value of 5.71 for tehená;
  - v. request the agreement of CAC47 to task the Codex Secretariat with reviewing the current nitrogen conversion factors in commodity standards from the relevant Codex commodity committees, which have been adjourned *sine die*, and propose the revocation of the corresponding Nx values; and
  - vi. amend the information document titled "Comprehensive Guidance for the Process of Submission, Consideration, and Endorsement of Methods for Inclusion in CXS 234" by incorporating the following text under Section 3.2 Acceptance of Methods of Analysis (Appendix IV):

*When methods for protein determination based on total nitrogen followed by calculation are submitted for endorsement, a nitrogen conversion factor should be provided. If the method is endorsed and included in CXS 234, the nitrogen conversion factor will be made available in an annex to CXS 234.*

**LISTING OF TYPE IV METHODS IN CXS 234 WHEN A TYPE I METHOD IS LISTED FOR THE SAME COMMODITY AND PROVISION (Agenda item 10)<sup>20</sup>**

77. Uruguay, Chair of the EWG, speaking also on behalf of the co-Chair, Brazil, introduced the item, providing background and history of discussions on this subject. The EWG Chair highlighted that: (i) there was no strict rule against endorsing a Type IV method when a Type I method existed, provided the performance data supports suitability; ii) as regulated in the Information Document titled "Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234," (hereafter, referred as Information Document), only one Type I method was typically listed per commodity and provision, unless there were complementary or identical methods. The Chair explained how the EWG had conducted its work, provided discussions on the necessity of coexistence between Type I and Type IV methods for the same commodity and provision. The Chair further proposed to i) continue with the selection of Type IV methods on a case-by-case basis when a "justifiable and motivating reason" was provided until appropriate selection criteria were developed; and ii) re-establish the EWG to develop co-existence or equivalence criteria for Type I and Type IV methods.

**Discussion**

78. CCMAS43 considered the recommendations and noted the following views.

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<sup>20</sup> CX/MAS 24/43/12

Endorsement of Type I and Type IV method for the same commodity provision

79. An Observer, speaking on behalf of Standards Development Organizations (SDOs) and referring to CRD08, indicated that combining Type IV methods with Type I methods for specific commodities and provisions was inappropriate as Type I methods were generally fully validated and determined a value that could only be arrived by the method, and is used for resolving trade disputes, while Type IV methods, lacking validation, should only be used when essential and validated alternatives were unavailable. It was emphasized that CXS 234 did not aim to comprehensively cover all globally available methods but was a collection of methods endorsed by the CAC. Outside of dispute situations laboratories might use any analytical method they preferred. Therefore, these Observers were of the opinion that adopting Type IV methods when there was already a Type I method in place for a certain commodity and provision should be considered an exception rather than on a case-by-case basis.
80. Another Observer underscored the importance of relying on established procedures and validated methods in Codex and cautioned against prioritizing personal preferences or regional convenience over validated methods, as this could lead to unresolved disputes. This Observer also drew the attention of CCMAS to the report of CCMAS37 which stated that *work in determining equivalence falls on the Standards development Organizations (SDO)* and encouraged CCMAS participants to consult with member of SDOs when the need arises to validate new methods, or to determine the bias between any two methods.
81. A Member proposed that the coexistence could be allowed only to ensure inclusiveness due the following considerations:
- i. there was no alternative method for Type I method according to its definition and earlier discussion in CCMAS; therefore, CCMAS should not endorse a Type IV method that has the same provision/analyte combination as a Type I method unless the method was really necessary;
  - ii. Since Type IV method might give different analytical value from the Type I method, endorsement of both methods should be exceptional and strictly limited to avoid unnecessary disputes;
  - iii. Codex standards should be ready for all Members to utilize in their regulations. If some Members who wanted to use Codex standards have faced serious difficulties to implement them, Codex needed to consider how to solve the situation; and
  - iv. the coexistence of Type I and Type IV methods would represent a compromise aimed at assisting Members facing significant implementation challenges.
82. Other members pointed out that according to the Procedural Manual (PM), a Type I method should typically be the sole method for establishing the accepted value. However, exceptions might arise, such as non-availability of reagents or legal restrictions on chemical use, justifying coexistence with a Type IV method.
83. The view that coexistence on a case-by-case basis might be necessary was also noted.
84. CCMAS43 agreed that endorsing a Type IV method when a Type I method exists should be approached as an exception and should not lead to a proliferation of Type IV methods when a Type I method already existed.

Development of co-existence or equivalence criteria for Type I and Type IV methods

85. The following views were expressed:
- i. Clear criteria are essential to address concerns regarding case-by-case scenarios. Defining when to apply this approach would enhance clarity and consistency;
  - ii. Implementing a criteria-based approach for when a Type IV method could complement a Type I method may lead to confusion and complications;
  - iii. Considering the adoption of a Type IV method alongside a Type I method for a specific commodity and provision as an exception, rather than addressing it on a case-by-case basis, is suggested. Therefore, selection criteria for these exceptional situations are deemed unnecessary and should not be pursued further; and
  - iv. the PWG on endorsement could be tasked to review existing methods and potentially eliminate outdated ones was proposed to reduce the prevalence of Type I and Type IV methods.
86. In response to the proposal for tasking the PWG on endorsement to review existing methods (see para 85(iv)), the Codex Secretariat clarified that CCMAS was currently reviewing all testing methods package by package which included the aforementioned task. Proposals emanating from this work were being considered by the WG on endorsement, therefore it was not necessary to specifically task the WG on endorsement.
87. CCMAS43 agreed not to develop the criteria for the co-existence of Type I and Type IV methods.

### Revision of the PM or the Information Document

88. In response to the suggestion of revising the PM to address the co-existence of Type I and Type IV methods for the same commodity and provision, the Codex Secretariat proposed that making changes to the PM should be avoided due to the process involved. Alternatively, revision to the Information Document was a simpler internal process, particularly for exceptional cases.
89. CCMAS43 agreed to amend the Information Document: *Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234* to address the endorsement of Type IV methods when there were existing Type I methods for the same commodity and provision.

### **Conclusion**

90. CCMAS43 agreed to
- i. Continue with the endorsement of Type IV methods when there were Type I methods for the same commodity and provision on exceptional basis when there were justifiable and motivating reasons; and
  - ii. insert the following bullet point in the Information Document: *Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234* under section 3.9 Type IV methods and their transitioning to other method types (Appendix IV):
    - v. *Under exceptional circumstances, a Type IV method can be endorsed when there is an existing Type I method for the same commodity and provision provided there is a justifiable reason.*

### **REPORT OF AN INTER-AGENCY MEETING ON METHODS OF ANALYSIS (Agenda item 11)<sup>21</sup>**

91. AOAC, as Vice-Chair of the Inter-Agency Meeting (IAM), introduced the IAM report on behalf of the Chair of the IAM. AOAC highlighted the various issues discussed in the IAM with respect to the work of CCMAS and other related matters, i.e. the review of methods of analysis for cereals, pulses and legumes in CXS 234; the numeric performance criteria for the determination of nitrate and nitrite ions in certain food matrices; methods of analysis for precautionary allergen labelling; placement of nitrogen conversion factors; listing of Type IV methods in CXS 234 when a Type I method was listed for the same commodity and provision; and methods of analysis for microplastics in food, as described in CRD04. AOAC added that some conferences, symposia, and workshops organised by IAM Members could be relevant to Members and it would be useful to share this information among Members. IAM was looking into how it could compile this information into a more usable document to share with CCMAS.
92. CCMAS43 noted that several of the issues raised in CRD04 had been considered under relevant agenda items.

### **Conclusion**

93. CCMAS43 thanked the members of IAM for their valuable contribution to the work of the Committee.

### **OTHER BUSINESS AND FUTURE WORK (Agenda item 12)**

#### The importance of harmonization of sampling and testing methods for the determination of microplastics in foods (CRD09)

94. The representative of FAO introduced the item, highlighting FAO's ongoing work in microplastics, the related discussions at the 19th FAO Subcommittee on Fish Trade, and the need for appropriate testing methods to better understand the dietary risk associated with microplastics. The FAO representative also shared that ISO 24187, while not recommending an analytical method, outlined several key principles in microplastic sampling and sample preparation. CCMAS was requested to consider recommending methods of analysis and sampling for microplastics.
95. A Member highlighted that besides fish and fish products, microplastics could be found in mineral water packaged in plastic bottles. The Member noted that it would be challenging to recommend methods of analysis at this time without an associated provision in relevant standards and suggested the possibility of inviting international metrological institutions to establish microplastic reference materials and collaborate with national competent authorities.
96. An Observer added that it would be working to establish methods of analysis for microplastics in foods in the future but recognized the complexities of this issue.

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<sup>21</sup> CRD04

97. In line with the CCMAS43's decision on methods of analysis for 1,2-DAG and PPP where there was no associated provision in the relevant commodity standard, the Chairperson suggested that in the future, CCMAS could consider recommending methods of analysis for microplastics to assist in data collection without including them in CXS 234.

**Conclusion**

98. CCMAS43 noted the information provided by the FAO representative, requested SDOs to keep the Committee updated on initiatives on microplastics and that the Committee would consider how to make efforts in this regard once more information on possible methods became available.

Areas of work for CCMAS in the future (CRD17)

99. The Chairperson highlighted possible areas of future work contained in CRD17, amongst others:
- i. revision of existing guidelines to ensure that they were up to date including the possible transfer of some provisions in the Procedural Manual (PM) to CCMAS information documents;
  - ii. conformity assessment on measurement uncertainty;
  - iii. use of biological methods to detect chemical substances; and
  - iv. organisation of virtual trainings and international proficiency testing (PT) for interested Members.
100. The Chairperson welcomed Members and Observers to share their insights or proposals for new areas of work, whether officially through normal Codex procedures or unofficially.

**Conclusion**

101. CCMAS43 noted the proposals of the Chairperson.

**DATE AND PLACE OF NEXT SESSION (Agenda item 13)**

102. CCMAS43 was informed that its 44th Session was tentative scheduled to take place from 5-9 May 2025 in Budapest, Hungary, with the final arrangements subject to confirmation by the Host Country in consultation with the Codex Secretariat.



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**APPENDIX II****Part 1. METHODS OF ANALYSIS AND SAMPLING PLANS FOR ADOPTION BY CAC47**

- 1.1. PROCESSED FRUITS AND VEGETABLES
- 1.2. CEREALS, PULSES AND LEGUMES
- 1.3. FISH AND FISHERY PRODUCTS
- 1.4. METHODS FOR DETECTION OF IRRADIATED FOODS
- 1.5. SPICES AND CULINARY HERBS
- 1.6. FATS AND OILS
- 1.7. SAMPLING PLANS

**Part 2. METHODS OF ANALYSIS FOR REVOCATION BY CAC47**

- 2.1. FATS AND OILS
- 2.2. CEREALS, PULSES AND LEGUMES
- 2.3. FISH AND FISHERY PRODUCTS

**Part 3. NITROGEN TO PROTEIN CONVERSION FACTORS FOR COMMODITIES APPROVED BY COMMODITIES COMMITTEES**

**METHODS OF ANALYSIS AND SAMPLING FOR ADOPTION BY CAC47**

(Methods and performance criteria are for inclusion in CXS 234-1999: changes indicated in ~~strikethrough~~, **bold** and/or underlined font).

**1.1 PROCESSED FRUITS AND VEGETABLES****Numeric performance criteria for benzoic acid, sorbates and tin in processed fruits and vegetables**

<u>Commodity</u>	<u>Provision</u>	<u>ML</u> <u>(mg/kg)</u>	<u>Minimum</u> <u>applicable range</u> <u>(mg/kg)</u>	<u>Limit of</u> <u>Detection</u> <u>(LOD)</u> <u>(mg/kg)</u>	<u>Limit of</u> <u>Quantification (LOQ)</u> <u>(mg/kg)</u>	<u>Precision</u> <u>(RSDR)</u> <u>(%) No</u> <u>more than</u>	<u>Recovery</u> <u>(%)</u>	<u>Example of applicable</u> <u>methods that meet the</u> <u>criteria</u>
<u>Jams, jellies, and</u> <u>marmalades</u>	<u>Benzoic</u> <u>Acid</u>	<u>1000</u>	<u>830 – 1170</u>	<u>100</u>	<u>200</u>	<u>11.3</u>	<u>95 – 105</u>	<u>ISO 5518, NMKL 124, AOAC</u> <u>983.16</u>
<u>Pickled cucumbers</u>	<u>Benzoic</u> <u>Acid</u>	<u>1000</u>	<u>830 – 1170</u>	<u>100</u>	<u>200</u>	<u>11.3</u>	<u>95 – 105</u>	<u>NMKL 124, AOAC 983.16</u>
<u>Mango chutney</u>	<u>Benzoic</u> <u>Acid</u>	<u>250</u>	<u>197 – 302</u>	<u>25</u>	<u>50</u>	<u>13.9</u>	<u>90 – 107</u>	<u>ISO 5518, NMKL 124, AOAC</u> <u>983.16</u>
<u>Coconut milk and</u> <u>coconut cream</u>	<u>Benzoic</u> <u>Acid</u>	<u>1000</u>	<u>830 – 1170</u>	<u>100</u>	<u>200</u>	<u>11.3</u>	<u>95 – 105</u>	<u>ISO 5518, NMKL 124, AOAC</u> <u>983.16</u>
<u>Jams, Jellies, and</u> <u>Marmalades</u>	<u>Sorbates</u>	<u>1000</u>	<u>830 – 1170</u>	<u>100</u>	<u>200</u>	<u>11.3</u>	<u>95 – 105</u>	<u>NMKL 124, AOAC 983.16</u>
<u>Pickled Cucumbers</u>	<u>Sorbates</u>	<u>1000</u>	<u>830 – 1170</u>	<u>100</u>	<u>200</u>	<u>11.3</u>	<u>95 – 105</u>	<u>NMKL 124, AOAC 983.16</u>
<u>Processed Fruits</u> <u>and Vegetables</u>	<u>Tin</u>	<u>250</u>	<u>197 – 302</u>	<u>25</u>	<u>50</u>	<u>13.9</u>	<u>90 – 107</u>	<u>AOAC 980.19, NMKL 126,</u> <u>NMKL 191</u>
<u>Table Olives</u>	<u>Tin</u>	<u>250</u>	<u>197 – 302</u>	<u>25</u>	<u>50</u>	<u>13.9</u>	<u>90 – 107</u>	<u>NMKL 190, EN 15764, NMKL</u> <u>126, NMKL 191</u>

## 1.2 CEREALS, PULSES AND LEGUMES

Cereals, pulses and legumes and derived products				
Commodity	Provision	Method	Principle	Type
<u>Pearl millet flour</u>	<u>Colour</u>	<u>ISO 16624</u>	<u>Diffuse reflectance Colorimetry (specific colour grader)</u>	<u>I</u>
Quinoa	Moisture <del>content</del>	ISO 712	Gravimetry (oven drying)	I
Quinoa	Protein <del>content (N x 6.25 in dry weight basis)</del>	ISO 1871	Titrimetry (Kjeldahl digestion)	IV
<u>Sorghum flour</u>	<u>Colour</u>	<u>ISO 16624</u>	<u>Diffuse reflectance Colorimetry (specific colour grader)</u>	<u>I</u>
<u>Soy protein products</u>	<u>Fat</u>	<u>ISO 734</u>	<u>Gravimetry (extraction)</u>	<u>I</u>
<u>Soy protein products</u>	<u>Crude Protein; excluding added vitamins, minerals, amino acids and food additives</u>	<u>AOCS Ba 4f-00</u> <u>AACCI 46.30</u> <u>ISO 16634-1</u>	<u>Gravimetry (Combustion)</u>	<u>IV</u> <u>IV</u> <u>IV</u>
<u>Vegetable protein products</u>	<u>Fat</u>	<u>ISO 734</u>	<u>Gravimetry (extraction)</u>	<u>I</u>
<u>Vegetable protein products</u>	<u>Crude Protein; excluding added vitamins, minerals, amino acids and food additives</u>	<u>AOCS Ba 4f-00</u> <u>AACCI 46.30</u> <u>ISO 16634-1</u>	<u>Gravimetry (Combustion)</u>	<u>IV</u> <u>IV</u> <u>IV</u>

<b>Miscellaneous products</b>				
<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>
<u>Gari</u>	<u>Particle size</u>	<u>ICC Recommendation 207</u>	<u>Sieving and Gravimetry</u>	<u>I</u>
<u>Edible Cassava flour</u>	<u>Particle size</u>	<u>ICC Recommendation 207</u>	<u>Sieving and Gravimetry</u>	<u>I</u>

**1.3 FISH AND FISHERY PRODUCTS**

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>
<u>Crackers from marine and freshwater fish, crustacean and molluscan shellfish</u>	<u>Crude Protein</u>	<u>AOAC 2001.11</u>	<u>Titrimetry (Kjeldahl Digestion)</u>	<u>IV</u>
<u>Fish and fishery products</u>	<u>Sensory and Physical Determinations</u>	<u>Described in the standard</u>	<u>Sensory analysis, Visual inspections, Counting</u>	<u>I</u>
Fish and fishery products: canned products	Drained weight	Described in the standard	<u>Weighing Gravimetry</u>	<u>I</u>
Fish and fishery products: canned products	Net weight	Described in the standard	<u>Weighing Gravimetry</u>	<u>I</u>
<u>Fish sauce</u>	<u>Total nitrogen</u>	<u>AOAC 978.02</u>	<u>Titrimetry (Kjeldahl digestion)</u>	<u>I</u>
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 <del>metre</del> meter. The dilution of fish sauce is necessary because of the	<u>Electrometry</u> <u>Potentiometry</u>	<u>III</u> <u>IV</u>

Commodity	Provision	Method	Principle	Type
		high ionic strength in the undiluted sauce.		
<b><u>Fish sauce</u></b>	<b><u>pH</u></b>	<b><u>NMKL 179</u></b>	<b><u>Potentiometry</u></b>	<b><u>II</u></b>
<b><u>Quick-frozen fish fillets</u></b>	<b><u>Gelatinous Condition (Determined as Moisture)</u></b>	<b><u>AOAC 983.18 and AOAC 950.46A</u></b>	<b><u>Gravimetry</u></b>	<b><u>I</u></b>
Quick-frozen fish sticks (fish fingers) and fish portions - breaded or in batter	Net weight	Described in the standard	<del>Weighing</del> <u>Gravimetry</u>	I
Quick-frozen fish sticks (fish fingers) and fish portions-breaded and in batter ( <del>except for certain fish species with soft flesh</del> )	Proportion of fish fillet and minced fish	WEFTA Method (Described in the standard)	Gravimetry	I
<b><u>Quick-frozen fish sticks (fish fingers), Fish Portions and Fish Fillets - Breaded or in Batter</u></b>	<b><u>Gelatinous Condition (Determined as Moisture)</u></b>	<b><u>AOAC 983.18 and AOAC 950.46A</u></b>	<b><u>Gravimetry</u></b>	<b><u>I</u></b>
<b><u>Quick-frozen Raw Scallop Products</u></b>	<b><u>Net weight</u></b>	<b><u>AOAC 963.18</u></b>	<b><u>Gravimetry</u></b>	<b><u>I</u></b>
<b><u>Quick-frozen Raw Scallop Products – Block Frozen Products</u></b>	<b><u>Drained weight</u></b>	<b><u>AOAC 967.13 and Described in the Standard</u></b>	<b><u>Gravimetry</u></b>	<b><u>I</u></b>
Salted Atlantic herring and salted sprat	Water content (Determined as Moisture)	AOAC 950.46B a)	Air-drying <u>Gravimetry</u>	I

Commodity	Provision	Method	Principle	Type
<u>Salted fish and dried salted fish of the Gadidae family of fishes</u>	<u>Moisture</u>	<u>AOAC 937.07 and AOAC 950.46B (airdrying a)</u>	<u>Gravimetry</u>	I
<u>Salted fish and dried salted fish of the Gadidae family of fishes</u>	<u>Salt saturation</u>	<u>See equation below</u>	<u>Calculation</u>	I

The % salt saturation is calculated as follows:

1.  $\text{___ \% salt in water} = (\% \text{ salt content} / (\% \text{ salt content} + \% \text{ moisture})) \times 100\%$
2.  $\text{___ \% salt saturation} = (\% \text{ salt in water} / 26.4\%^*) \times 100\%$

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

$$36 \text{ g sodium chloride} / (100 \text{ g water} + 36 \text{ g sodium chloride}) \times 100\% = 26.4\%$$

<u>Salted fish and dried salted fish of the Gadidae family of fishes</u>	<u>Water content in the whole fish</u>	<u>Described in the standard</u>	<u>Gravimetry</u>	I
<u>Smoked fish, smoke-flavoured fish and smoke-dried fish</u>	<u>Water phase salt (salt determined as chloride expressed as sodium chloride)</u>	<u>AOAC 952.08 and Sodium Chloride see method criteria</u> <u>Water phase salt = (% salt × 100) / (%water + %salt)</u>	<u>Gravimetry and Titrimetry and Calculation</u>	I
<u>Smoked fish, smoke-flavoured fish and smoke-dried fish</u>	<u>Water activity</u>	<u>ISO 18787</u>	<u>Electrometry</u>	II



**Table 1. Method performance criteria for histamine for fish and fishery products (Note: the methods performance criteria are not for adoption as they are already adopted and in CXS 234. The only amendments are the changes to example methods / principles)**

Provision	ML (mg/100 g)	Minimum applicable range (mg/100 g)	LOD (mg/100 g)	LOQ (mg/100 g)	Precision (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Histamine	10 (average)	8 – 12	1	2	16.0 <u>16</u>	90 – 107	AOAC 977.13 † / NMKL 99, NMKL 196, <u>ISO 19343</u>	Fluorimetric HPLC Fluorometry, HPLC-UV, HPLC-UV, HPLC-FLD
Histamine	20 (each unit)	16 – 24	2	4	14.4 14	90 - 107	AOAC 977.13 † / NMKL 99, NMKL 196, <u>ISO 19343</u>	Fluorimetric HPLC Fluorometry, 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 2 and may either meet the minimum applicable range, or LOD and LOQ criteria listed.

**Table 2. Criteria for determination of toxin analogues by chemical methods (Note: only the amendments in underlined font and example methods are for adoption)**

Toxin group	Toxin	Minimum applicable range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD <sub>R</sub> ) (%) No more than	Recovery percent (%)	Examples of 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	
	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 %	<del>60–115</del> <u>70-130</u>	<u>EU-harmonised SOP using HPLC-MSMS (See reference below*)</u>
	DTX1	0.03 – 0.2	0.01	0.02	44 %	<del>60–115</del> <u>70-130</u>	<u>For other methods see references **</u>
	DTX2	0.1 – 0.5	0.03	0.06	38 %	<del>60–115</del> <u>70-130</u>	
Domoic acid	DA	14 – 26	2	4	20 %	80 – 110 <u>70-130</u>	<u>EN 14176 (HPLC - UV)</u> <u>AOAC 991.26 (HPLC-UV)</u>

Toxin group	Toxin	Minimum applicable range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD <sub>R</sub> ) (%) No more than	Recovery percent (%)	Examples of applicable methods that meet the criteria
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	

\* [https://www.aesan.gob.es/en/CRLMB/docs/docs/metodos\\_analiticos\\_de\\_desarrollo/EU-Harmonised-SOP-LIPO-LCMSMS\\_Version5.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 analogues 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 2.

Where toxin analogues that are not listed in Table 2 are determined the competent authority must assess the contribution of these analogues to total toxicity whilst conducting further investigations.

**Table 3. Method Numeric Performance Criteria for screening and for determination of methylmercury\*** (Note: for adoption are the method performance criteria for pink cusk eel and orange roughy and the amendments in underlined font / examples methods / principles)

Commodity	Provision	ML (mg/kg)	Min. Appl. Range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
All <del>tuna</del> Tuna (all species)	methylmercury*	1.2	0.64 – 1.8	0.12	0.24	31	80 - 110	EN 16801 / <u>NMKL 202</u> AOAC 977.15** <u>NMKL 186** /</u> AOAC 2013.06**/ <u>EN 15763/**</u>	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 / <u>NMKL 202</u> AOAC 977.15** <u>NMKL 186** /</u> AOAC 2013.06**/ <u>EN 15763/**</u>	GC-electron capture GC-ICP/MS  AAS-flame ICP-MS ICP-MS ICP-MS
All <del>marlin</del> Marlin (all species)	methylmercury*	1.7	0.95 – 2.5	0.17	0.34	30	80 - 110	AOAC 988.11 EN 16801/ <u>NMKL 202</u> AOAC 977.15** <u>NMKL 186** /</u> AOAC 2013.06**/ <u>EN 15763/**</u>	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 / <u>NMKL 202</u> AOAC 977.15** <u>NMKL 186** /</u> AOAC 2013.06**/ <u>EN 15763/**</u>	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 (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
<u>Orange roughy</u>	<u>methylmercury*</u>	<u>0.8</u>	<u>0.40 – 1.2</u>	<u>0.08</u>	<u>0.16</u>	<u>33</u>	<u>80 - 110</u>	<u>AOAC 988.11</u> <u>EN 16801 / NMKL 202</u> <u>AOAC 977.15**</u> <u>NMKL 186** /</u> <u>AOAC 2013.06**/</u> <u>EN 15763/**</u>	<u>GC-electron capture GC-ICP/MS</u>  <u>AAS-flame ICP-MS</u> <u>ICP-MS</u> <u>ICP-MS</u>
<u>Pink cusk-eel</u>	<u>methylmercury*</u>	<u>1.0</u>	<u>0.52 – 1.5</u>	<u>0.10</u>	<u>0.20</u>	<u>32</u>	<u>80 - 110</u>	<u>AOAC 988.11</u> <u>EN 16801 / NMKL 202</u> <u>AOAC 977.15**</u> <u>NMKL 186** /</u> <u>AOAC 2013.06**/</u> <u>EN 15763/**</u>	<u>GC-electron capture GC-ICP/MS</u>  <u>AAS-flame ICP-MS</u> <u>ICP-MS</u> <u>ICP-MS</u>

\*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 \*.

**Table 4. Method Performance Criteria for Sodium Chloride and for Salt determined as Chloride expressed as Sodium Chloride**

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

## 1.4 METHODS FOR DETECTION OF IRRADIATED FOODS

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

## 1.5 SPICES AND CULINARY HERBS

Commodity	Provision	Method	Principle	Type
<u>Small cardamom</u>	<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	!
<u>Small cardamom</u>	<u>Total Ash, on dry basis</u>	<u>ISO 939 and ISO 928</u>	<u>Calculation from moisture and ash (at 550 °C), Distillation and Gravimetry</u>	!
<u>Small cardamom</u>	<u>Acid Insoluble Ash, on dry basis</u>	<u>ISO 939 and ISO 930</u>	<u>Calculation from moisture and ash (at 550 °C), Distillation and Gravimetry</u>	!
<u>Small cardamom</u>	<u>Volatile Oil on dry basis</u>	<u>ISO 939 and ISO 6571</u>	<u>Calculation from moisture and volatile oils, Distillation followed and Distillation</u>	!
<u>Small cardamom</u>	<u>Extraneous Matter</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	!
<u>Small cardamom</u>	<u>Foreign Matter</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	!
<u>Small cardamom</u>	<u>Insect defiled/infested</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	!
<u>Small cardamom</u>	<u>Immature and shrivelled capsules</u>	<u>ISO 882-1 and ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	!
<u>Small cardamom</u>	<u>Mammalian or/and other excreta (for whole)</u>	<u>Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices</u> <a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs</a>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>
<u>Small cardamom</u>	<u>Mould visible</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	!
<u>Small cardamom</u>	<u>Empty and malformed capsules</u>	<u>ISO 882-1</u>	<u>Visual Examination (counting)</u>	!
<u>Small cardamom</u>	<u>Whole insect Live/dead (For whole)</u>	<u>ISO 927</u>	<u>Visual examination (counting)</u>	!
<u>Small cardamom</u>	<u>Whole insect live/dead (For powdered/pieces)</u>	<u>AOAC 975.49</u>	<u>Flotation</u>	!



Commodity	Provision	Method	Principle	Type
<u>Allspice, juniper berry and star anise</u>	<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Total ash on dry basis</u>	<u>ISO 939 and ISO 928</u>	<u>Calculation from moisture and ash (at 550 °C), Distillation and gravimetry.</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Acid-insoluble ash on dry basis</u>	<u>ISO 939 and ISO 930</u>	<u>Calculation from moisture and ash (at 550 °C), Distillation and gravimetry.</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Volatile oils on dry basis</u>	<u>ISO 939 and ISO 6571</u>	<u>Calculation from moisture and volatile oil content, Distillation and gravimetry.</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Extraneous matter</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Foreign matter</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Mould visible</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Mammalian and other excreta (whole spice)</u>	<u>MPM V-8 Spices, Condiments, Flavors and Crude Drugs MPM: V-8. Spices, Condiments, Flavors, and Crude Drugs   FDA</u>	<u>Visual examination followed by gravimetry</u>	IV
<u>Allspice, juniper berry and star anise</u>	<u>Whole dead insects and live insects</u>	<u>ISO 927</u>	<u>Visual examination (counting)</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Insect fragments (whole spices)</u>	<u>ISO 927</u>	<u>Visual examination (counting)</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Insect fragments (For powdered / pieces)</u>	<u>AOAC 975.49</u>	<u>Flotation method</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Insect defiled</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Allspice, juniper berry and star anise</u>	<u>Rodent hair</u>	<u>AOAC 965.40</u>	<u>Flotation</u>	!

Commodity	Provision	Method	Principle	Type
<u>Turmeric</u>	<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	!
<u>Turmeric</u>	<u>Total Ash on dry basis</u>	<u>ISO 939 and ISO 928</u>	<u>Calculation from moisture and ash (at 550 °C) Distillation and gravimetry</u>	!
<u>Turmeric</u>	<u>Acid Insoluble Ash on dry basis</u>	<u>ISO 939 and ISO 930</u>	<u>Calculation from moisture and ash (at 550 °C), Distillation and gravimetry</u>	!
<u>Turmeric</u>	<u>Extraneous Matter</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Turmeric</u>	<u>Foreign Matter</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Turmeric</u>	<u>Insect defiled.</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Turmeric</u>	<u>Whole insects Live /dead (for whole)</u>	<u>ISO 927</u>	<u>Visual Examination (counting)</u>	!
<u>Turmeric</u>	<u>Whole insects Live /dead (For powdered/ pieces)</u>	<u>AOAC 975.49</u>	<u>Flotation method</u>	!
<u>Turmeric</u>	<u>Mammalian or/and Other excreta (whole)</u>	<u>Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices</u> <a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs</a>	<u>Visual examination followed by gravimetry</u>	<u>IV</u>
<u>Turmeric</u>	<u>Mould visible</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Dried or Dehydrated Ginger</u>	<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	!
<u>Dried or Dehydrated Ginger</u>	<u>Total Ash on dry basis</u>	<u>ISO 939 and ISO 928</u>	<u>Calculation from moisture and ash (at 600°C), Distillation and Gravimetry</u>	!

Commodity	Provision	Method	Principle	Type
<u>Dried or Dehydrated Ginger</u>	<u>Acid Insoluble Ash on dry basis</u>	<u>ISO 939 and ISO 930</u>	<u>Calculation from moisture and ash (at 600°C), Distillation and Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Ginger</u>	<u>Volatile Oil on dry basis</u>	<u>ISO 939 and ISO 6571</u>	<u>Calculation from moisture and ash (at 600°C), Distillation and Distillation</u>	<u>!</u>
<u>Dried or Dehydrated Ginger</u>	<u>Extraneous Matter</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Ginger</u>	<u>Foreign Matter</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Ginger</u>	<u>Insect Damage</u>	<u>Method V-8 Spices, Condiments, Flavours and Crude Drugs( Macroanalytical Procedure Manual) MPM: V-8. Spices</u>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>
<u>Dried or Dehydrated Ginger</u>	<u>Whole dead insect</u>	<u>ISO 927</u>	<u>Visual examination</u>	<u>!</u>
<u>Dried or Dehydrated Ginger</u>	<u>Mammalian/ Other Excreta (for whole)</u>	<u>Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B</u> <u><a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32</a></u>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>
<u>Dried or Dehydrated Ginger</u>	<u>Mould visible</u>	<u>ISO 927</u>	<u>Visual examination followed by Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Ginger</u>	<u>Live Insect</u>	<u>ISO 927</u>	<u>Visual Examination</u>	<u>!</u>
<u>Dried or Dehydrated Ginger</u>	<u>Calcium (as oxide) on dry basis</u>	<u>ISO 939 and ISO 928 and ISO 1003 – Annex A</u>	<u>Calculation from moisture and ash (at 600°C), and titrimetry</u>	<u>IV</u>
<u>Dried or Dehydrated Ginger</u>	<u>Sulphur dioxide</u>	<u>AOAC 990.28</u>	<u>Distillation followed by titrimetry</u>	<u>IV</u>

Commodity	Provision	Method	Principle	Type
<u>Cloves</u>	<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	<u>!</u>
<u>Cloves</u>	<u>Volatile Oil on dry basis</u>	<u>ISO 939 and ISO 6571</u>	<u>Calculation from moisture and volatile oils, Distillation and Distillation</u>	<u>!</u>
<u>Cloves</u>	<u>Total Ash on dry basis</u>	<u>ISO 939 and ISO 928</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	<u>!</u>
<u>Cloves</u>	<u>Acid Insoluble Ash on dry Basis</u>	<u>ISO 939 and ISO 930</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	<u>!</u>
<u>Cloves</u>	<u>Extraneous matter</u>	<u>ISO 927</u>	<u>Visual examination followed by Gravimetry</u>	<u>!</u>
<u>Cloves</u>	<u>Foreign matter</u>	<u>ISO 927</u>	<u>Visual examination followed by Gravimetry</u>	<u>!</u>
<u>Cloves</u>	<u>Insect damage</u>	<u>ISO 927</u>	<u>Visual examination followed by Gravimetry</u>	<u>!</u>
<u>Cloves</u>	<u>Insects / Insect fragments</u>	<u>ISO 927</u>	<u>Visual examination (counting)</u>	<u>!</u>
<u>Cloves</u>	<u>Crude fibre</u>	<u>ISO 5498</u>	<u>Gravimetry</u>	<u>!</u>
<u>Cloves</u>	<u>Mould visible (for whole)</u>	<u>Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)</u> <a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32</a>	<u>Visual examination followed by Gravimetry</u>	<u>IV</u>
<u>Cloves</u>	<u>Live insect</u>	<u>ISO 927</u>	<u>Visual examination (counting)</u>	<u>!</u>
<u>Cloves</u>	<u>Mammalian or/and Other excreta (For whole)</u>	<u>MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V 32)</u> <a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32</a>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>

Commodity	Provision	Method	Principle	Type
<u>Dried Basil</u>	<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	<u>!</u>
<u>Dried Basil</u>	<u>Total Ash on dry basis</u>	<u>ISO 939 and ISO 928</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	<u>!</u>
<u>Dried Basil</u>	<u>Acid Insoluble Ash on dry basis</u>	<u>ISO 928 and ISO 930</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	<u>!</u>
<u>Dried Basil</u>	<u>Volatile Oil on dry basis</u>	<u>ISO 939 and ISO 6571</u>	<u>Calculation from moisture and volatile oils, Distillation and Distillation</u>	<u>!</u>
<u>Dried Basil</u>	<u>Extraneous Matter</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Dried Basil</u>	<u>Foreign Matter</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Dried Basil</u>	<u>Insect Damage (whole leaves)</u>	<u>Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)</u> <u><a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32</a></u>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>
<u>Dried Basil</u>	<u>Insects / Insect Fragments</u>	<u>ISO 927</u>	<u>Visual Examination (counting)</u>	<u>!</u>
<u>Dried Basil</u>	<u>Mould damage (for whole leaves)</u>	<u>Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)</u> <u><a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32</a></u>	<u>Visual examination followed by Gravimetry</u>	<u>IV</u>
<u>Dried Basil</u>	<u>Mammalian Excreta, And Other Excreta (For whole leaves)</u>	<u>MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs</u>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>

Commodity	Provision	Method	Principle	Type
		<u>and botanicals (V 32)</u> <u><a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32</a></u>		
<u>Saffron</u>	<u>Moisture</u>	<u>ISO 3632-2</u>	<u>Gravimetry (drying at 103°C)</u>	<u>!</u>
<u>Saffron</u>	<u>Total Ash on dry basis</u>	<u>ISO 3632-2 and ISO 928</u>	<u>Calculation from moisture and ash (at 550°C), Gravimetry and Gravimetry</u>	<u>!</u>
<u>Saffron</u>	<u>Acid Insoluble Ash on dry basis</u>	<u>ISO 3632-2 and ISO 928 and ISO 930</u>	<u>Calculation from moisture and ash (at 550°C), Gravimetry and Gravimetry</u>	<u>!</u>
<u>Saffron</u>	<u>Soluble extract in cold water on dry basis</u>	<u>ISO 3632-2 and ISO 941</u>	<u>Calculation from moisture and Soluble extract, Gravimetry and Extraction</u>	<u>!</u>
<u>Saffron</u>	<u>Taste strength (expressed as picrocrocin) <math>A_{1cm}^{1\%}</math> 257 nm</u>	<u>ISO 3632-2</u>	<u>Absorbance</u>	<u>!</u>
<u>Saffron</u>	<u>Aroma strength (expressed as safranal) <math>A_{1cm}^{1\%}</math> 330 nm</u>	<u>ISO 3632-2</u>	<u>Absorbance</u>	<u>!</u>
<u>Saffron</u>	<u>Coloring strength (expressed as crocin) <math>A_{1cm}^{1\%}</math> 440 nm</u>	<u>ISO 3632-2</u>	<u>Absorbance</u>	<u>!</u>
<u>Saffron</u>	<u>Extraneous Matter</u>	<u>ISO 3632-2</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Saffron</u>	<u>Foreign Matter</u>	<u>ISO 3632-2</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Saffron</u>	<u>Insect Damage</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Saffron</u>	<u>Whole dead Insects /Insect Fragments</u>	<u>ISO 927</u>	<u>Visual examination (counting)</u>	<u>!</u>
<u>Saffron</u>	<u>Mould Visible</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Saffron</u>	<u>Mammalian Excreta (whole spice)</u>	<u>MPM V-8 Spices, Condiments, Flavors and Crude Drugs MPM: V-8. Spices, Condiments, Flavors, and</u>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>

Commodity	Provision	Method	Principle	Type
		<u>Crude Drugs   FDA</u>		
<u>Saffron</u>	<u>Rodent filth</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	!
<u>Nutmeg</u>	<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	!
<u>Nutmeg</u>	<u>Total ash on dry basis</u>	<u>ISO 939 and ISO 928</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	!
<u>Nutmeg</u>	<u>Acid-insoluble ash on dry basis</u>	<u>ISO 939 and ISO 930</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	!
<u>Nutmeg</u>	<u>Water-insoluble ash on dry basis</u>	<u>ISO 939 and ISO 929</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	!
<u>Nutmeg</u>	<u>Volatile oil content on dry basis</u>	<u>ISO 939 and ISO 6571</u>	<u>Calculation from moisture and volatile oils, Distillation and Distillation</u>	!
<u>Nutmeg</u>	<u>Extraneous matter</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Nutmeg</u>	<u>Foreign matter</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Nutmeg</u>	<u>Mould Visible</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	!
<u>Nutmeg</u>	<u>Insect defiled/infested</u>	<u>ISO 927</u>	<u>Visual Examination followed by gravimetry</u>	!
<u>Nutmeg</u>	<u>Dead insect, insect fragments, rodent contamination (hair)</u>	<u>ISO 927</u>	<u>Visual Examination (counting)</u>	!
<u>Nutmeg</u>	<u>Live insect</u>	<u>ISO 927</u>	<u>Visual Examination (counting)</u>	!
<u>Nutmeg</u>	<u>Mammalian and or other excreta (For whole and broken)</u>	<u>Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.41 <a href="https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32">https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32</a></u>	<u>Visual examination followed by gravimetry</u>	<u>IV</u>

Commodity	Provision	Method	Principle	Type
<u>Nutmeg</u>	<u>Piece of mace</u>	<u>ISO 927</u>	<u>Visual examination followed by gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Total Ash on dry basis</u>	<u>ISO 939 and ISO 928</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Acid-insoluble ash on dry basis</u>	<u>ISO 939 and ISO 930</u>	<u>Calculation from moisture and ash (at 550°C), Distillation and Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Pungency, Scoville Heat units</u>	<u>ISO 3513</u>	<u>Sensory evaluation</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Colour value</u>	<u>ISO 7541</u>	<u>Spectrophotometry</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Mammalian excreta (whole)</u>	<u>ISO 927</u>	<u>Visual examination followed by Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Mould damage (for whole chilli peppers)</u>	<u>MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V.32)</u>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Mould damage (for ground)</u>	<u>AOAC 945.94</u>	<u>Visual Examination (Howard Mould Count)</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Insect Damage (for whole chilli peppers)</u>	<u>MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs</u>	<u>Visual Examination followed by Gravimetry</u>	<u>IV</u>



Commodity	Provision	Method	Principle	Type
		<u>and botanicals (V.32)</u>		
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Extraneous matter</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Foreign matter</u>	<u>ISO 927</u>	<u>Visual Examination followed by Gravimetry</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Live insect</u>	<u>ISO 927</u>	<u>Visual Examination (counting)</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Insect fragments</u>	<u>ISO 927</u>	<u>Visual examination (counting)</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Rodent hair (Ground chilli)</u>	<u>AOAC 978.22</u>	<u>Flotation method</u>	<u>!</u>
<u>Dried or Dehydrated Chilli Pepper and Paprika</u>	<u>Rodent hair (Ground paprika)</u>	<u>AOAC 977.25 B</u>	<u>Microscopic examination</u>	<u>!</u>

## 1.6 FATS AND OILS (CCFO)

Commodity	Provision	Method	Principle	Type
Olive oils and olive pomace oils	Absorbency in ultra-violet	COI/T.20/Doc. No. 19 / <del>or</del> ISO 3656 <del>or</del> / AOCs Ch 5-91	Absorption in ultra-violet	II
Olive oils and olive pomace oils	Acidity, free (acid value)	ISO 660 (section 9.1) or / AOCs Cd 3d-63 / COI/T.20/Doc. No 34	Titrimetry	I
Olive oils and olive pomace oils	Alpha-tocopherol	ISO 9936	HPLC (UV or fluorescence)	II
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Alpha-tocopherol</u></b>	<b><u>AOCs Ce 8-89</u></b>	<b><u>HPLC (UV or fluorescence)</u></b>	<b><u>III</u></b>
Olive oils and olive pomace oils	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 and COI/T.20/Doc. No 33	Analysis of triglycerides by <del>or</del> HPLC and fatty acids by GC followed by calculation	I
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Erythrodiol + uvaol</u></b>	<b><u>COI/T.20/Doc. No 26</u></b>	<b><u>Separation and gas chromatography (FID)</u></b>	<b><u>II</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Fatty acid composition</u></b>	<b><u>COI/T.20/Doc. No 33</u></b>	<b><u>Gas chromatography (FID) of methyl esters</u></b>	<b><u>II</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Fatty acid composition</u></b>	<b><u>AOCs Ce 2-66 and AOCs Ch 2-91 / Ce 1h-05</u></b>	<b><u>Gas chromatography (FID) of methyl esters</u></b>	<b><u>III</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Fatty acid composition</u></b>	<b><u>ISO 12966-2 and ISO 12966-4</u></b>	<b><u>Gas chromatography (FID) of methyl esters</u></b>	<b><u>III</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>2-glyceryl monopalmitate percentage</u></b>	<b><u>COI/T.20/Doc. No 23</u></b>	<b><u>Hydrolysis and derivatization Gas chromatography (FID)</u></b>	<b><u>II</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Fatty acid ethyl ester content</u></b>	<b><u>COI/T.20/Doc. No 28</u></b>	<b><u>Gas chromatography (FID)</u></b>	<b><u>II</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Halogenated solvents, traces</u></b>	<b><u>ISO 16035</u></b>	<b><u>Headspace Gas chromatography (ECD)</u></b>	<b><u>II</u></b>

Commodity	Provision	Method	Principle	Type
Olive oils and olive pomace oils	Iodine value	ISO 3961 <del>et</del> / AOAC 993.20 <del>et</del> / AOCS Cd 1d-92 <del>et</del> / NMKL 39	Wijs-titrimetry	I
Olive oils and olive pomace oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	<u>Sensory Panel test</u>	I
Olive oils and olive pomace oils	Peroxide value	ISO 3960 <del>et</del> / AOCS Cd 8b-90 / <u>NMKL 158</u>	Titrimetry	I
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Relative density</u></b>	<b><u>ISO 6883 / AOCS Cc 10c-95</u></b>	<b><u>Pycnometry</u></b>	<b><u>I</u></b>
Olive oils and olive pomace oils	Refractive index	ISO 6320 <del>et</del> / AOCS Cc 7-25	Refractometry	II
Olive oils and olive pomace oils	Saponification value	ISO 3657 <del>et</del> / AOCS Cd 3-25	Titrimetry	I
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>4<math>\alpha</math>-desmethylsterol and total sterol content</u></b>	<b><u>COI/T.20/Doc. No 26</u></b>	<b><u>Separation and Gas chromatography (FID)</u></b>	<b><u>II</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>4<math>\alpha</math>-desmethylsterol and total sterol content</u></b>	<b><u>ISO 12228-2 (part 2)</u></b>	<b><u>Separation and Gas chromatography (FID)</u></b>	<b><u>III</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>4<math>\alpha</math>-desmethylsterol and total sterol content</u></b>	<b><u>AOCS Ch 6-91</u></b>	<b><u>Separation and Gas chromatography (FID)</u></b>	<b><u>III</u></b>
Olive oils and olive pomace oils	Stigmastadienes	COI/T.20/Doc. No. 11	Gas chromatography (FID)	II
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Stigmastadienes</u></b>	<b><u>ISO 15788-1</u></b>	<b><u>Gas chromatography (FID)</u></b>	<b><u>III</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Stigmastadienes</u></b>	<b><u>AOCS Cd 26-96</u></b>	<b><u>Gas chromatography (FID)</u></b>	<b><u>III</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>trans fatty acids content</u></b>	<b><u>COI/T.20/Doc No. 33</u></b>	<b><u>Gas chromatography (FID) of methyl esters</u></b>	<b><u>II</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>trans fatty acids content</u></b>	<b><u>ISO 12966-2 and ISO 12966-4</u></b>	<b><u>Gas chromatography (FID) of methyl esters</u></b>	<b><u>III</u></b>
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>trans fatty acids content</u></b>	<b><u>AOCS Ce 2-66 and AOCS Ce 1h-05</u></b>	<b><u>Gas chromatography (FID) of methyl esters</u></b>	<b><u>III</u></b>

Commodity	Provision	Method	Principle	Type
Olive oils and olive pomace oils	Unsaponifiable matter	ISO 3596 et / AOCS Ca 6b-53	Gravimetry	I
<b><u>Olive oils and olive pomace oils</u></b>	<b><u>Wax content</u></b>	<b><u>COI/T.20/Doc. No. 28</u></b>	<b><u>Gas chromatography (FID)</u></b>	<b><u>II</u></b>
Olive oils and olive pomace oils	Wax content	AOCS Ch 8-02	Gas chromatography (FID)	II III

Numeric Performance Criteria for iron and copper in Olive oils and olive pomace oils

Commodity	Provision	ML (mg/kg)	Method Performance Criteria						Principle
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Example of Methods that meet the criteria	
<u>Olive oils and olive pomace oils</u>	<u>Iron</u>	<u>3.0</u>	<u>1.8 – 4.2</u>	<u>0.3</u>	<u>0.6</u>	<u>27</u>	<u>80 – 110</u>	<u>ISO 8294</u> <u>AOAC 990.05</u> <u>AOCS Ca 17a-18</u> <u>ISO 21033</u>	<u>GF-AAS</u> <u>GF-AAS</u> <u>ICP-OES</u> <u>ICP-OES</u>
<u>Olive oils and olive pomace oils</u>	<u>Copper</u>	<u>0.1</u>	<u>0.03 – 0.17</u>	<u>0.01</u>	<u>0.02</u>	<u>44</u>	<u>80 – 110</u>	<u>ISO 8294</u> <u>AOAC 990.05</u> <u>AOCS Ca 17a-18</u>	<u>GF-AAS</u> <u>GF-AAS</u> <u>ICP-OES</u>

Commodity	Provision	Method	Principle	Type
<u>Fish oil (calanus oil)</u>	<u>Wax content</u>	<u>AOCS Ch 8-02</u>	<u>Gas Chromatography (FID)</u>	<u>IV</u>

**NUMERIC PERFORMANCE CRITERIA FOR LEAD AND CADMIUM IN FOODS – EXAMPLE METHODS** (Note: the numeric performance criteria are not for adoption. The only changes in underlined font are amendments / addition of example methods / principles)

**Table 9: Numeric performance criteria for lead and cadmium in foods**

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 <sub>R</sub> ) (%) 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%	<u>EPA 200.8,</u> <u>ISO 17294-2,</u> <u>EN 17851,</u> <u>EN 14083</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>GF-AAS</u>
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%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013.06,</u> <u>EN 15763,</u> <u>EN 17851</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u>
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013.06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>EN 14083,</u> <u>NMKL 186</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>GF-AAS</u> <u>ICP-MS.</u>
Secondary milk products (including <u>Butter, edible casein products and whey powders</u> )	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013:06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083,</u> <u>AOAC 999.11 (edible casein)</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>AAS or GF-AAS</u>

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 <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
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%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013:06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>EN 14083,</u> <u>NMKL 186,</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>GF-AAS,</u> <u>ICP-MS,</u> <u>GF-AAS</u>
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%	<u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u>
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%	<u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083,</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS.</u>
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%	<u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186,</u> <u>EN 14083,</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS.</u>

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 <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028 - 0.072	0.01 <u>0</u>	0.02 <u>0</u>	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186, EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS GF-AAS
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.01 <u>0</u>	0.02 <u>0</u>	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS GF-AAS
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.01 <u>0</u>	0.02 <u>0</u>	44	60-115%	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 (RSD <sub>R</sub> ) (%) 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 17a-18, ISO 12193, EN 17851, NMKL 186 ISO 21033	GF-AAS, ICP-OES GF-AAS, ICP-MS, ICP-MS ICP-OES.
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 186 EN 14083. NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS. 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 (RSD <sub>R</sub> ) (%) 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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS, 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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS, 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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 (RSD <sub>R</sub> ) (%) 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%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013.06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>FDA Method 4.7 Ver.1.2</u> <u>AOAC 2013.06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083.</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013.06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083.</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>

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 <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013.06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013:06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>EN 15763,</u> <u>OIV-MA-AS323-07.</u>	<u>ICP-MS,</u> <u>ICP-MS</u>
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%	<u>EN 15763,</u> <u>OIV-MA-AS323-07.</u>	<u>ICP-MS,</u> <u>ICP-MS.</u>
Pig, edible offal of	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013.06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>

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 <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS GF-AAS
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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS, GF-AAS
Wine (wine and fortified / liqueur wine) made from grapes harvested before July 2019	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	EN 15763, OIV-MA-AS323-07.	ICP-MS, ICP-MS.
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 186 EN 14083 NMKL 161	GF-AAS, ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS GF-AAS
Fresh farmed mushrooms (common mushrooms <i>Agaricus bisporus</i> }, shiitake mushrooms ( <i>Lentinula edodes</i> ), and oyster mushrooms <i>Pleurotus ostreatus</i> })	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS, ICP-MS, ICP-MS GF-AAS, 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 (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013:06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013:06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	<u>FDA Method 4.7 Ver.1.2,</u> <u>AOAC 2013.06,</u> <u>EN 15763,</u> <u>EN 17851,</u> <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS,</u> <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>

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 <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
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 186 EN 14083, AOAC 999.11 NMKL 139 NMKL 161	ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS, GF-AAS, AAS (Flame Absorption) GF-AAS
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%	EUsalt/AS 015, EN 17851, EN 14083.	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 186 EN 14083 NMKL 161	ICP-MS ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 (RSD <sub>R</sub> ) (%) 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 186 EN 14083, NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 186 EN 14083 NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 186 EN 14083 NMKL 161	GF-AAS ICP-MS, ICP-MS GF-AAS 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 186 EN 14083 NMKL 161	ICP-MS, ICP-MS, ICP-MS, ICP-MS, ICP-MS GF-AAS 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 (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>FDA Method 4.7 Ver.1.2</u> , <u>AOAC 2013:06</u> , <u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%	<u>FDA Method 4.7 Ver.1.2</u> , <u>AOAC 2013:06</u> , <u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	<u>FDA Method 4.7 Ver.1.2</u> , <u>AOAC 2013:06</u> , <u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>

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 <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%	<u>ISO 23637</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>GF-AAS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> <u>GF-AAS</u> <u>GF-AAS</u>
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%	<u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>GF-AAS</u> <u>GF-AAS</u>
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%	<u>ISO 23637</u> <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>GF-AAS</u> <u>ICP-MS</u> , <u>ICP-MS</u> , <u>GF-AAS</u> , <u>GF-AAS</u>
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%	<u>EUsalt/AS 015</u> , <u>EN 17851</u> , <u>EN 14083</u>	<u>ICP-OES</u> , <u>ICP-MS</u> , <u>GF-AAS</u>
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%	<u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>GF-AAS</u>

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 <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
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%	<u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>GF-AAS</u>
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%	<u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>GF-AAS</u>
Cephalopods	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%	<u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>GF-AAS</u> <u>GF-AAS</u>

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 <sub>R</sub> ) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Marine bivalve molluscs (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%	<u>EN 15763</u> , <u>EN 17851</u> , <u>NMKL 186</u> <u>EN 14083</u> <u>NMKL 161</u>	<u>ICP-MS</u> , <u>ICP-MS</u> , <u>ICP-MS</u> , <u>GF-AAS</u> <u>GF-AAS</u>

**SAMPLING PLAN FOR METHYLMERCURY CONTAMINATION IN FISH  
(FOR ADOPTION BY CAC47 AND FOR INCLUSION IN CXS 193)**

**GENERAL CONSIDERATIONS**

**DEFINITION**

<b>Lot</b>	<p>An identifiable quantity of a food commodity delivered at one time and determined by the official to have common characteristics, such as origin, variety, type of packing, packer, consignor, or markings.</p> <p>A lot of whole fish should consist of one species and the length and/or weight should be comparable. In case the length and/or weight of the fish is not comparable, the consignment may still be considered as a lot, but a specific sampling procedure has to be applied (as described in paragraph 8).</p>
<b>Sublot</b>	Designated part of a larger lot in order to apply the sampling method on that designated part. Each sub-lot must be physically separate and identifiable.
<b>Sampling plan</b>	A procedure for sampling of food from a certain lot with a view of a specific chemical analysis of that lot, in order to ensure that the sample that is taken, is representative for the concentration of the concerned chemical within the lot.
<b>Methylmercury test procedure</b>	A methylmercury test procedure consists of three steps: sample selection, sample preparation and methylmercury quantification. It contains an accept/reject level.
<b>Decision rule</b>	The lot is accepted if the test result is less than or equal to the Codex maximum level (ML); otherwise, the lot is rejected.
<b>Incremental sample</b>	The quantity of material taken from a single random place in the lot or sub-lot.
<b>Aggregate sample</b>	The combined total of all the incremental samples that is taken from the lot or sub-lot. The aggregate sample has to be at least as large as the laboratory sample or samples combined. The entire aggregate sample should be comminuted in a mill.
<b>Laboratory sample</b>	A sample intended for the laboratory, which consists out of a comminuted quantity of fish muscle, or whole fish. The laboratory sample may be a portion of or the entire aggregate sample. If the aggregate sample is larger than the laboratory sample(s), the laboratory sample(s) should be removed in a random manner from the homogenised aggregate sample.
<b>Test portion</b>	A randomly removed portion of the comminuted laboratory sample for the extraction of the methylmercury for chemical analysis.

**MATERIAL TO BE SAMPLED**

1. Each lot or sub-lot which is to be examined must be sampled separately.
2. Fresh or frozen whole (in general after removing digestive tract) or dressed fish (eviscerated fish with head and tail removed) and other non-bulk fishery products of lots greater than or equal to 15 metric tons (MT) should be subdivided into sub-lots of 15-30 MT in accordance with Table 2.
3. Lots of fishery products traded as bulk commodities of greater than 100 MT should be subdivided into sub-lots in accordance with Table 1 to be sampled separately.

**Table 1. Subdivision of sub-lots according to bulk consignment lot weight**

Commodity	Lot weight (MT <sup>a</sup> )	Weight or number of sub-lots (MT)
Fishery products (traded as bulk consignments)	≥ 1500	500
	> 300 and <1500	3 sub-lots (minimum 100 MT)
	≥ 100 and ≤300	100
	< 100	-

<sup>a</sup>1 metric tonne (MT) = 1000 kilograms

**Table 2. Subdivision of sublots according to other products lot weight**

Commodity	Lot weight (MT <sup>a</sup> )	Weight or number of sub-lots (MT)
Fish (traded as non-bulk consignments)	≥ 15	15-30
	< 15	-

<sup>a</sup>1 metric tonne (MT) = 1000 kilograms

4. Taking into account that the weight of the lot is not always an exact multiple of the weight of the sub-lots, the weight of the sub-lot may exceed the mentioned weight by a maximum of 20 %.

**INCREMENTAL SAMPLE**

5. The recommended minimum number of incremental samples taken from the lot or sub-lot is dependent on the size of the lot or sub-lot as specified in Table 3.
6. The aggregate sample should contain a quantity of sample of at least 1 kilogram. The minimum weight of the incremental sample should be determined by dividing 1 kilogram by the required number of incremental samples as listed in Table 3. Incremental samples taken from a lot or sub-lot should be of comparable weight.

**Table 3. Number of incremental samples to be taken depending on the weight of the lot or subplot**

Lot weight (MT <sup>a</sup> )	Number of incremental samples	Minimum laboratory sample weight (kg)
≤ 0.05	3	1
> 0.05 - ≤ 0.5	5	1
> 0.5	10	1

<sup>a</sup>1 metric tonne (MT) = 1000 kilograms

7. Whole fish are considered to be of comparable length and weight class where the differences in size and/or weight do not exceed about 50%.
8. For lots where fish are not of comparable length and/or weight the following approaches are to be applied to taking the incremental samples:
  - a. Where a length or weight class/category is predominant (80% or more of the fish lot or sub-lot are within the same length and/or weight class), the aggregate sample is combined only from incremental samples of fish within the predominant category and outliers are excluded. This aggregate sample is to be considered as being representative for the whole lot/sub-lot.
  - b. Where there is no predominant weight or size class and where the overall length and/or weight of the fish present in the lot or sub-lot varies by more than 50% but less than 100%, the lot or sub-

lot is separated into two length or weight classes and separate aggregate samples are composited from incremental samples taken independently from each length and/or weight class.

- c. Where there is no predominant weight or size class and where the overall length and/or weight of the fishes present in the lot differ more than 100%, the lot or sub-lot is separated into three length or weight classes and separate aggregate samples are composited from incremental samples taken independently from each length or weight class.
9. For lots or sub-lots of whole fish the part of the fish where the incremental sample is taken is informed by the weight of the whole fish as specified in Table 3. Some examples on sampling of batches of fishes of different size and/or weight can be found in Annex II.

**Table 4. Tissue area the incremental sample is taken from for whole fish based on weight classes**

<b>Weight class of an individual whole fish</b>	<b>Sampled part</b>
< 1 kg	Whole fish (after removing the digestive tract) For lots of 0.05MT or greater where the aggregate sample would exceed 3 kg the midline (halfway between the gill opening and the anus) strip from backbone to belly can be sampled
1-6 kg	Midline (halfway between the gill opening and the anus) strip from backbone to belly
> 6 kg	Midline (halfway between the gill opening and the anus) strip from backbone to belly Alternatively, equal composite parts of muscle from behind the head and close to the tail can be sampled For tuna, incremental samples can instead be taken from the muscle from close to the tail.

#### **PACKAGING AND TRANSPORTATION OF SAMPLES**

10. Each laboratory sample should be placed in a clean, inert container offering adequate protection from contamination, loss of analytes by adsorption to the internal wall of the container and against damage in transit. All necessary precautions, for example temperature control and storage in airtight containers, should be taken to avoid any change in composition of the sample which might arise during transportation or storage (for example avoiding excess heat or the sample drying out).

#### **SEALING AND LABELLING OF SAMPLES**

11. Each laboratory sample taken for official use shall be sealed at the place of sampling and identified. A record must be kept of each sample, permitting each lot, or sub-lot, to be clearly identified and giving the date and place the sampling occurred, together with any additional information likely to be of assistance to the analyst.

#### **SAMPLE PREPARATION PRECAUTIONS**

12. In the course of sampling, precautions, such as correct-sampling technique and limitation of cross contamination, should be taken to avoid any changes which would affect the levels of methylmercury, adversely affect the analytical determination, or make the aggregate samples unrepresentative.
13. Wherever possible, apparatus and equipment coming into contact with the sample should not contain mercury and should be made of inert materials, e.g. plastics such as polypropylene, polytetrafluoroethylene (PTFE) etc. These should be acid cleaned to minimise the risk of contamination. High quality stainless steel may be used for cutting edges to take increment samples and make comminuted samples.

#### **HOMOGENIZATION – GRINDING**

14. The complete aggregate sample should be finely comminuted and thoroughly mixed using a process that has been demonstrated to achieve complete homogenization. Depending on the equipment available frozen samples may need to be thawed prior to homogenisation.

**TEST PORTION**

15. Procedures for selecting the test portion from the comminuted laboratory sample should be a random process. Following homogenization and thorough mixing, the test portion can be selected from any location throughout the comminuted laboratory sample.
16. It is suggested that three test portions be selected from each comminuted laboratory sample. The three test portions will be used for enforcement, appeal, and confirmation if needed.

**ANALYTICAL METHODS**

17. A criteria-based approach, whereby a set of performance criteria is established with which the analytical method used should comply, is appropriate. The performance criteria-based approach has the advantage that, by avoiding setting down specific details of the method used, developments in methodology can be exploited without having to reconsider or modify the specific method. Utilizing this approach, laboratories would be free to use the analytical method most appropriate for their facilities.
18. Refer to The Procedural Manual of the Codex Alimentarius Commission for principles for the establishment of methods of analysis.
19. Method performance criteria for methylmercury and total mercury are detailed for the species of fish for which there are Codex MLs in CXS 234-1999.
20. 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.

**RECONDITIONING LOTS/SUB-LOTS**

21. A lot or sub-lot where fish are not of comparable length and/or weight that is separated into 2 to 3 length and/or weight classes should be analysed sequentially from the largest class first.
22. A lot or sub-lot where fish are not of comparable length and/or weight can be considered in compliance with the ML if the methylmercury concentration of the aggregate sample taken from the highest length and/or weight class is below the ML. However, export or trade requirements (e.g. certificates of analysis) may require testing lots or sub-lots of smaller length and/or weight classes.
23. Where the methylmercury concentration in the aggregate sample taken from a length and/or weight class is above the ML then the next largest length/weight class should also be analysed. If the methylmercury concentration in this sample is below the ML the lot or sub-lot can be reconditioned to remove length and/or weight classes that exceed the ML to ensure the remaining fish are in compliance with the ML.
24. For a lot or sub-lot separated into three length or weight classes paragraph 23 should be repeated for the smallest length/weight classes if the methylmercury concentration in the aggregate sample taken from the middle length/weight class is also above the ML.



**ANNEX****Examples on how to apply provisions in the Sampling Plan****EXAMPLE 1**

**In case the size and/or weight of the fishes present in the lot differs more than 50 % but less than 100 %: two separate representative samples are taken from each size or weight class/category within a lot.**

*Example: 5 MT lot of fishes with weights from 2 kg to 3.5 kg.*

A first aggregate sample is taken of the smaller sized (lot relative) fishes, which weigh about 2-2.75 kg: 10 incremental samples (fishes) are taken. Each incremental sample is constituted from the muscle meat of the middle part of the fish (slice backbone to belly, symmetrically taken around line B in Figure 1) and weighs about 100 grams. This results in one aggregate sample of about 1 kg to be homogenised and analysed separately.

A second aggregate sample is taken of the larger sized (lot relative) fishes, which weigh about 2.75 -3.5 kg: 10 incremental samples (fishes) are taken. Each incremental sample is constituted from the muscle meat of the middle part of the fish (slice backbone to belly, symmetrically taken around line B in Figure 1) and weighs about 100 grams. This results in one aggregate sample of about 1 kg to be homogenised and analysed separately.

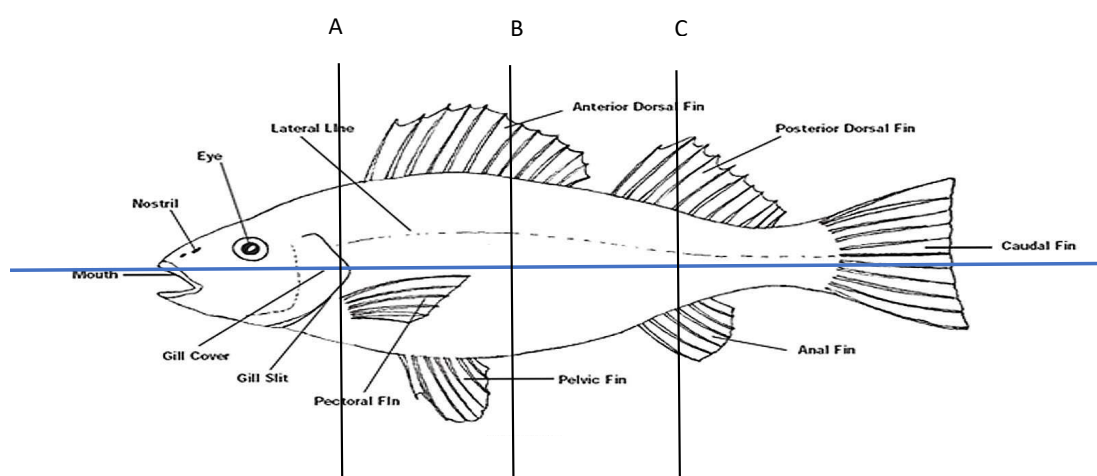


Figure 1: The different sections of a fish.

A) Laboratory performs a sequential analysis:

First the sample of the larger sized fishes is homogenised and analysed separately.

- In case the analytical result is compliant, the whole lot is compliant.
- In case the analytical result is non-compliant, as a second step the sample of the smaller sized fishes is homogenised and analysed separately.
- In case the analytical result of the sample of the smaller sized fishes is non-compliant, the whole lot is non-compliant.
- In case the analytical result of the sample of smaller sized fishes is compliant, then the smaller sized fishes (2-2.75 kg) have to be sorted out and these fishes are compliant. The remaining larger sized fishes (2.75-3.5 kg) are non-compliant.

B) Laboratory analyses both samples at the same time:

- In case both analytical results are compliant, the whole lot is compliant.
- In case both analytical results are non-compliant, the whole lot is non-compliant.
- In case the sample of the smaller sized fishes (2-2.75 kg) is compliant and the sample of the larger sized fishes (2.75-3.5 kg) not, then the smaller sized fishes (2-2.75 kg) have to be sorted out and these small sized fishes are compliant. The remaining larger sized fishes (2.75-3.5 kg) are non-compliant.

**EXAMPLE 2**

**In case the size and/or weight of the fishes present in the lot differs more than 100%: three separate representative samples are taken from each size or weight class/category within a lot**

*Example: 10 MT lot of fishes with weights from 2 kg to 8 kg.*

A first aggregate sample is taken of the smaller sized (lot relative) fishes, which weigh about 2-4 kg: 10 incremental samples (fishes) are taken, each incremental sample is constituted from the muscle meat of the middle part of the fish (slice backbone to belly, symmetrically taken around line B in Figure 1) and weighs about 100 grams. This results in one aggregate sample of about 1 kg, to be homogenised and analysed separately.

A second aggregate sample is taken of the fishes of medium size (lot relative) of about 4-6 kg: 10 incremental samples (fishes) are taken, each incremental sample is constituted from the muscle meat of the middle part of the fish (slice backbone to belly) and weighs about 100 grams. This results in one aggregate sample of about 1 kg, to be homogenised and analysed separately.

A third aggregate sample is taken of the larger sized (lot relative) fishes of about 6-8 kg: 10 incremental samples (fishes) are taken, each incremental sample is

- constituted of the right side dorso-lateral muscle meat in the middle part of the fish (symmetrically around line B in Figure 1 and above the horizontal line in Figure 1) and weighs about 100 grams. This results in one aggregate sample of about 1 kg to be homogenised and analysed separately.

OR

- constituted of equal parts of 50 grams of the muscled meat close to the tail part (the region around line C in Figure 1) and the muscle meat close to the head part of one fish (the region of line A in Figure 1) which are combined to form an incremental sample of about 100 grams per fish. This results in one aggregate sample of about 1 kg to be homogenised and analysed separately.

**METHODS OF ANALYSIS FOR REVOCATION BY CAC47****2.1 FATS AND OILS**

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>
Olive oils and olive pomace oils	Difference between the actual and theoretical ECN 42 triglyceride content	AOCS Ce 5b-89	Analysis of triglycerides of HPLC and calculation	I
Olive oils and olive pomace oils	Erythrodiol + uvaol	COI/T.20/Doc.no. 30	Gas chromatography	II
Olive oils and olive pomace oils	Halogenated solvents, traces	COI/T.20/Doc. no. 8	Gas chromatography	II
Olive oils and olive pomace oils	Iron and copper	ISO 8294 or AOAC 990.05	AAS	II
Olive oils and olive pomace oils	Relative density	IUPAC 2.101, with the appropriate conversion factor.	Pycnometry	I
Olive oils and olive pomace oils	Sterol composition and total sterols	COI/T.20/Doc. no. 30   ISO 12228-2 or AOAC Ch 6-91	Gas chromatography	II
Olive oils and olive pomace oils	Trans fatty acids content	COI/T.20/Doc no. 17 or ISO 15304 or AOCS Ch 2a-94	Gas chromatography of methyl esters	II
Olive oils and olive pomace oils	Unsaponifiable matter	ISO 18609	Gravimetry	I
Olive oils and olive pomace oils	Wax content	COI/T.20/Doc. no. 18	Gas chromatography	II

**2.2 CEREALS, PULSES AND LEGUMES**

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>
Pearl millet flour	Colour	<i>Modern Cereal Chemistry</i> , 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry using specific colour grader	IV
Quinoa	Moisture content	AACCI 44-15.02	Gravimetry	I
Sorghum flour	Colour	<i>Modern Cereal Chemistry</i> , 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry using specific colour grader	IV
Soy protein products	Fat	CAC/RM 55 - Method 1	Gravimetry (extraction)	I
Soy protein products	Protein	AOAC 955.04D (using factor 6.25)	Titrimetry, Kjeldahl digestion	II
Vegetable protein products	Fat	CAC/RM 55 - Method 1	Gravimetry (extraction)	I
Vegetable protein products	Protein	AOAC 955.04D (using factor 6.25)	Titrimetry, Kjeldahl digestion	II
Gari	Granularity	ISO 2591-1	Sieving	I
Edible cassava flour	Granularity	ISO 2591-1	Sieving	I

**2.3 FISH AND FISHERY PRODUCTS**

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>
Fish and fishery products	Histamine	AOAC 977.13	Fluorimetry	II
Fish and fishery products	Mercury	AOAC 977.15	Flameless atomic absorption spectrophotometry	III
Boiled dried salted anchovies	Sodium Chloride (chloride expressed as sodium chloride)	AOAC 937.09	Titrimetry	II
Canned shrimps or prawns	Size, determination of	Described in the Standard	Number per 100 g	I
Fish sauce	Total nitrogen	AOAC 940.25	Digestion	I
Fish sauce	Sodium chloride	AOAC 976.18	Potentiometry	II
Fish sauce	Sodium chloride	AOAC 937.09	Titrimetry	IV
Fish sauce	Histamine	AOAC 977.13	Fluorimetry	II
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
Quick frozen fish sticks (fish fingers) and fish portions - breaded or in batter	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Salted fish and dried salted fish of the Gadidae family of fishes	Salt	Described in CXS 167-1989	Titrimetry (Mohr) (Salt determined as chloride expressed as sodium chloride)	I

Commodity	Provision	Method	Principle	Type
Salted fish of the Gadidae family	Salt Content Water content	Sampling and method described in the standard	Gravimetry	I
Smoked fish, smoke-flavoured fish and smoke-dried fish	Water activity	ISO 21807	Electrometry	III
Smoked fish, smoke-flavoured fish and smoke-dried fish	Water phase salt	AOAC 937.09 Described in standard <sup>xiii</sup>	Calculation	I
Sturgeon caviar	Salt content	Described in CXS 167-1989	Titrimetry (Mohr) Salt determined as chloride expressed as sodium chloride	I

<sup>xiii</sup> % salt × 100/(% water + % salt).

**PART 3****NITROGEN TO PROTEIN CONVERSION FACTORS FOR COMMODITIES APPROVED BY COMMODITY COMMITTEES****Animal Protein Source**

Milk and milk products - 6.38

Meat - 6.25

Cook cured ham - 6.25

Infant formula - The calculation of the protein content of infant formulas prepared ready for consumption may be based on N x 6.25, unless a scientific justification is provided for the use of a different conversion factor for a particular product. The value of 6.38 is generally established as a specific factor appropriate for conversion of nitrogen to protein in other milk products, and the value of 5.71 as a specific factor for conversion of nitrogen to protein in other soy products.

**Fish and fishery products**

Crackers from marine and freshwater fish, crustaceans and molluscan shellfish - 6.25

**Plant Protein Source**

Wheat, wheat protein products - 5.71

Soya and non-ferment soybean products - 5.71

Maize - 6.25

Quinoa - 6.25

Sorghum - 6.25

Tempe - 5.71

Gochujang - 6.25

Products produced by separation from wheat and soya grains and flours of certain non-protein constituents (starch, other carbohydrates) - 6.25

**APPENDIX III****PART 1. METHODS OF ANALYSIS AND SAMPLING WHICH REMAINS UNCHANGED IN CXS 234 AS A RESULT OF DECISIONS BY CCMAS43**

1. CEREALS, PULSES AND LEGUMES
2. FISH AND FISHERY PRODUCTS
3. FATS AND OILS

**PART 2. METHODS RECOMMENDED FOR 1,2 DIGLYCERIDES AND PYROPHEOPHYTIN "a"**



## PART 1

## METHODS OF ANALYSIS AND SAMPLING WHICH REMAINS UNCHANGED IN CXS 234 AS A RESULT OF DECISIONS BY CCMAS43

## 1. CEREALS, PULSES AND LEGUMES

Commodity	Provision	Method	Principle	Type
Degermed maize (corn) meal and maize (corn) grits	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Durum wheat semolina and durum wheat flour	Ash	AOAC 923.03 / ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Pearl millet flour	Ash	AOAC 923.03 / ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Sorghum flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Sorghum grains	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Soy protein products	Ash	AOAC 923.03 ISO 2171: (Method B)	Gravimetry	I
Vegetable protein products	Ash	AOAC 923.03 ISO 2171 and AOAC 925.09	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Wheat flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Wheat protein products including wheat gluten	Ash	AOAC 923.03 ISO 2171 and AOAC 925.09	Calculation from moisture and gravimetry (incineration at 550 °C)	I

Commodity	Provision	Method	Principle	Type
Whole and decorticated pearl millet grains	Ash	AOAC 923.03 / ISO 2171 and ISO 712/ ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Whole maize (corn) meal	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I

## 2. FISH AND FISHERY PRODUCTS

Commodity	Provision	Method	Principle	Type
Fish sauce	Amino acid nitrogen	AOAC 920.04 and AOAC 920.03	Determining formaldehyde titration method subtracting by ammoniacal nitrogen (magnesium oxide method)	I
Frozen abalone (covered by glaze)	Net weight	AOAC 963.18	Gravimetry	I
Frozen fish and fishery products	Thawing and cooking procedures	Described in the standards	Thawing and heating	I
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
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
Quick-frozen fish fillets	Net weight of products covered by glaze	Described in the standard	Water spraying and sieving	I
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

Commodity	Provision	Method	Principle	Type
Smoked fish, smoke-flavoured fish and smoke-dried fish	Water activity	NMKL 168	Electrometry	III
Live and raw bivalve molluscs	Paralytic toxicity shellfish	AOAC 959.08	Mouse bioassay	IV
Live and raw bivalve molluscs	Paralytic toxicity shellfish	AOAC 2011.27	Receptor binding assay	IV

### 3. FATS AND OILS

Commodity	Provision	Method	Principle	Type
Olive oils and olive pomace oils	Insoluble impurities in light petroleum	ISO 663	Gravimetry	I
Olive oils and olive pomace oils	Moisture and volatile matter	ISO 662	Gravimetry	I
Olive oils and olive pomace oils	<u>Stigmastadienes</u>	ISO 15788-2	HPLC	III

### METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

Commodity categories	Method of sampling	Notes
<b>Fats and oils</b>		
Olive oils and olive pomace oils	ISO 661 and ISO 5555	
Fish oils	ISO 5555	

## PART 2

## METHODS RECOMMENDED FOR 1,2 DIGLYCERIDES AND PYROPHEOPHYTIN "a"

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>
Olive oils and olive pomace oils	1,2 Diglycerides	COI /T.20/Doc. No 32	Gas chromatography (FID)
Olive oils and olive pomace oils	1,2 Diglycerides	ISO 29822	Gas chromatography (FID)
Olive oils and olive pomace oils	Pyropheophytin "a"	ISO 29841	HPLC UV or FLD

## Appendix IV

**PROPOSED REVISION TO THE INFORMATION DOCUMENT: COMPREHENSIVE GUIDANCE FOR THE  
PROCESS OF SUBMISSION, CONSIDERATION AND ENDORSEMENT OF METHODS  
FOR INCLUSION IN CXS 234**

The additions are highlighted in **bold/underlined**.

Section 3.2 Acceptance of Methods of Analysis

**iv. d. When methods for protein determination based on total nitrogen followed by calculation are submitted for endorsement, a nitrogen conversion factor should be provided. If the method is endorsed and included in CXS234, the nitrogen conversion factor will be made available in an annex to CXS234.**

Section 3.9 Type IV methods and their transitioning to other method types

**v. Under exceptional circumstances, a Type IV method can be endorsed when there is an existing Type I method for the same commodity and provision provided there is a justifiable reason.**