

# CODEx ALIMENTARIUS COMMISSION



Food and Agriculture  
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World Health  
Organization

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**Agenda Item 3**

**MAS44/CRD02 Rev.1**

**May 2025**

**ORIGINAL LANGUAGE ONLY**

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

**44th Session**

**Virtual**

**5 – 8 May and 14 May 2025**

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

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

#### **Agenda Item 2 – Matters referred to the Committee by the Codex Alimentarius Commission and/or its subsidiary bodies (CX/MAS 25/44/2 and CX/MAS 25/44/2-Add.1)**

##### **Matters arising from other subsidiary bodies**

##### **CCFFP: method for determination of amino acid nitrogen in fish sauce**

1. The virtual working group (VWG) reviewed the response from the 36<sup>th</sup> session of the Codex Committee on Fish and Fishery Products (CCFFP36, 2024) regarding the determination of amino acid nitrogen in fish sauce. CCFFP36 informed CCMAS that the methods as presented were still necessary. The VWG had no additional comments or questions to the CCFFP response, and thus the VWG recommends retaining the methods as they currently appear in the *Recommended methods of analysis and sampling* (CXS 234-1999). The method, principle, and typing are found in Appendix I, Table 1.

##### **CCAFRICA: methods for the determination of chloride in dried meat**

2. The VWG reviewed the response from the 25<sup>th</sup> session of the FAO/WHO Coordinating Committee for Africa (CCAFRICA25, 2025) on methods for the determination of chloride in dried meat which requested these methods be included in CXS 234-1999. The VWG had no additional comments or questions, and recommends including the methods as written in Appendix I, Table 2.

##### **CCFA: method for the determination of sodium chloride in food grade salt**

3. The VWG also reviewed the revision to the *Standard for food grade salt* (CXS 150-1985) referred by the 55<sup>th</sup> session of the Codex Committee on Food Additives (CCFA55) who requested that CCMAS consider inclusion of the method for sodium chloride, as currently described in CXS 150-1985, in CXS 234-1999. The VWG reviewed the method and the sampling plan including editorial revisions and recommended that moving both into CXS 234-1999 as written in Appendix I, Table 3.
4. The VWG recommends retention and/or endorsement of the methods and sampling plan at the type indicated in Appendix I, Tables 1-3.

#### **Agenda Item 3 – Endorsement of methods of analysis and sampling plans for provisions in Codex standards (CX/MAS 25/44/3 and CX/MAS 25/44/3-Add.1)**

##### **Methods of analysis referred by the Codex Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU)**

5. The VWG reviewed the methods of analysis referred by CCNFSDU and recommendations are recorded in this document. These include methods of analysis for dietary fibre in all foods, as well as numerous provisions for follow up and infant formula.
6. An observer organization explained the need to update the method for dietary fibre and included background information in MAS44/CRD04. The request from CCNFSDU included a footnote that accompanied the method.

The footnote states: *\*Isolated, purified, and/or synthetic fibres captured by AOAC 2022.01/ICC Standard 191/AACC 32-61.01 that do not meet the Codex definition of dietary fibre in the Guidelines on nutrition labelling (CXG 2-1985) should be subtracted from the final measurement, where deemed appropriate by competent authorities.* Some members requested that the footnote be removed since it was restating the definition of dietary fibre in the *Guidelines on nutrition labelling* (CXG 2-1985), and other members supported the inclusion of the footnote at the request of CCNFSDU.

7. The VWG recommends:

- endorsement of the new method for dietary fibre inclusive of the footnote as shown in Appendix II, Table 1; and
- returning the following question back to CCNFSDU:
  - Would this footnote also apply to all or a subset of dietary fiber methods in Table 6 of CXS 234-1999, and if so, should CCMAS apply the footnote to the other methods? Reference MAS44/CRD04 for further information on whether the footnote is appropriate when applied only to AOAC 2022.01/ICC Standard 191/AACCI 32.61.01.

8. The VWG also reviewed the methods of analysis referred by CCNFSDU for follow-up formula and infant formula. The VWG recommended changes to the provision for Vitamin A noting that AOAC 2012.10 / ISO 20633 determines the supplemented forms of vitamin A (retinyl palmitate and retinyl acetate). One member of the VWG suggested inclusion of additional methods that were previously endorsed for infant formula but not referred to CCMAS by CCNFSDU for follow up formula. Those methods are captured in Appendix II, Table 2.

9. Some members requested establishment of numeric performance criteria for the provisions outlined in the *Standards for follow up formula for older infants and product for young children* (CXS 156-1987) and *infant formula and formulas for special medical purposes intended for infants* (CXS 72-1981). These suggested numeric performance criteria are listed in MAS44/CRD10. The VWG discussed changes to the table and method typing to performance criteria but referenced a decision at CCMAS40 (2019) when the committee requested CCNFSDU to consider establishing numerical method performance criteria for calcium, copper, iron, magnesium, manganese, phosphorous, potassium, sodium and zinc for infant formula. In response, CCNFSDU stated their preference to continue listing type II methods as specific methods were preferred for dispute settlement purposes. The VWG agreed to recommend CCMAS again query CCNFSDU on the establishment of numeric performance criteria for some provisions on follow up formula.

10. The VWG recommends:

- endorsement of method for follow up and infant formulas as shown in Appendix II, Table 2;
- revocation and retyping of the methods for dietary fibre in all foods and for certain provisions in follow up formula in Appendix II, Table 3; and
- returning the following question back to CCNFSDU regarding numeric performance criteria:
  - Would CCNFSDU prefer that CCMAS set numeric performance criteria rather than method typing for provisions for infant and follow-up formula?

### **Other relevant matters arising from the amendment of CXS 234-1999**

#### Methods of analysis where the principle and typing were not indicated in the respective commodity standards

11. The VWG did not review the tables in CX/MAS 25/44/3-Add.1 or the tables in MAS44/CRD07. Both documents contain tables with methods that appear in commodity standards but where methods do not appear in CXS 234-1999, however it was decided that there was not sufficient time during the VWG to complete these tables, and that it may be a better option to form an electronic working group to complete the tables prior to CCMAS45. The tables are in Appendix III, Tables 1, 6, 7, and 8.

12. The VWG recommends:

- retaining the methods in their respective commodity standards for the present time; and
- CCMAS to consider whether to form an electronic working group (EWG) to take up the work to review the methods of analysis in Appendix III, Tables 1, 6, 7 and 8. The EWG recommendations would be considered at CCMAS45.

#### Method of analysis by description, where specific references to published methods have been provided

13. The VWG discussed the two options presented for Determination of Fish Content in Quick frozen fish sticks (fish fingers), fish portions, and fish fillets – breaded or in batter and consensus was reached to present the methods of analysis directly in the commodity table in CXS 234-1999 with no appendix.

14. The VWG recommends endorsing Appendix III, Table 2 as shown for inclusion in CXS 234-1999.
- Submission of revised methods for the determination of chloride expressed as sodium chloride in butter and cheese, and numeric performance criteria for copper in milk fat products (MAS44/CRD05)**
15. An Observer organization proposed new methods for the determination of chloride expressed as sodium chloride in butter and cheese. The VWG reviewed the proposed methods and the proposed changes to CXS 234-1999 in MAS44/CRD05. There was a discussion on the applicability and range of the new methods. The VWG agreed to retain the current typing of methods in CXS 234-1999 and endorse the new methods as type III until such time when additional validation data was submitted that would show the proposed methods were fit for purpose. When the new methods are shown to be fit for purpose CCMAS would reconsider the typing.
16. The VWG also reviewed the submitted table for numeric performance criteria for copper in milk fat products and agreed to remove ISO 5738 | IDF 76 and AOAC 960.40 as example methods because these methods do not meet the criteria.
17. There was no change to the numeric performance criteria for iron.
18. The VWG recommends:
- retaining the current methods and endorsing the new methods for the determination of chloride expressed as sodium chloride in butter and cheese as shown in Appendix III, Table 3; and
  - removal of ISO 5738 | IDF 76 and AOAC 960.40 as example methods that meet the numeric performance criteria for copper in milk fat products as shown in Appendix III, Table 4.

**Amendments to numeric performance criteria in fish sauce (MAS44/CRD06)**

19. The VWG reviewed the numeric performance criteria prepared by Norway in consultation with the Codex Secretariat presented in MAS44/CRD06. There was consensus that the tables as presented were appropriate including the examples of applicable methods that meet the criteria.
20. The VWG recommends endorsing the numeric performance criteria as presented in Appendix III, Table 5.

**Agenda Item 4 – Matters pending from CCMAS43**

**Methods of analysis for protein in quinoa**

21. The VWG reviewed the responses to the circular letter (CL 2024/91-MAS) regarding the determination of protein in quinoa by ISO 1871. There was mixed support for retention of ISO 1871 as a type I or type IV method. Some members expressed the opinion that ISO 1871 was sufficiently prescriptive that it could be considered a type I method, while other members indicated that ISO 1871 was only a guideline for protein determination, and was not sufficiently specific to be considered a type I method. ISO 20483 was offered as an alternative type I method for the determination of protein in quinoa but currently lacks validation data for pseudo-cereals such as quinoa. The Observer from ISO mentioned that quinoa could be added as a validated matrix in the future, and that ISO may consider taking up that work if needed.
22. The VWG discussed that ISO 1871 was endorsed as a type I method for other commodities, namely teheana. There was consensus that the method endorsements of teheana and quinoa should be consistent with respect to protein determination by ISO 1871. The VWG discussed whether to revoke the type I endorsement of ISO 1871 for teheana, and ask for step-by-step details including the reagents and catalysts that was used in the validation study. Harmonized and documented details of ISO 1871 could be added as an annex in CXS 234-1999 as a temporary patch until such time as either ISO 1871 or ISO 20483 was endorsed as a type I method.
23. The VWG recommends:
- CCMAS consider whether the method specifics submitted in response to CL 2024/91-MAS are sufficient to endorse ISO 1871 as a type I method, or whether it should remain as a type IV; and
  - CCMAS consider the typing of ISO 1871 as a type I method for the determination of protein in teheana, and whether it should be changed to a type IV method to be consistent with quinoa.

**Determination of moisture content in whey powder (CX/MAS 25/44/5)**

24. The VWG had a lively discussion on the two methods to determine moisture content in whey powder. Two proposals for a way forward were presented in the discussion paper CX/MAS 25/44/5 and the VWG debated the merits of each. The 102°C oven drying method was previously endorsed as a type IV method for other dairy powders, but there was a concern that the method would not yield sufficiently precise data for high natural lactose dairy products such as whey powder. A consensus was reached in that the 102°C method would be recommended for endorsement in CXS 234-1999 as a type IV method with a footnote which explains the reason for coexisting type I and type IV methods. The table as agreed is shown in Appendix IV, Table 1.

25. The VWG recommends:

- endorsing the 102°C oven drying method (Described in CXS 234-1999 Appendix XI) as a type IV method with an accompanying footnote. The table is found in Appendix IV, Table 1; and
- if endorsed by CCMAS, the scope of the method in Appendix XI in CXS 234-1999 will need to be expanded to include whey powders.

#### **Agenda Item 5 – Review of methods of analysis in CXS 234**

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##### **Fruit juices workable package (CX/MAS 25/44/6)**

26. There was a brief introduction and discussion on the fruit juices workable package. However, due to a lack of corresponding numeric criteria against which to evaluate the methods, the VWG was unable to come to a consensus on endorsement of the methods. A suggestion was made to form a task group of experts in the area who would be able to assist CCMAS in determining whether the fruit juice methods were still fit for purpose. Another suggestion was that there were at least some methods with corresponding numeric criteria that CCMAS could finish reviewing this year, with the remainder of the methods for future work.

##### **Cocoa products and chocolate workable package (CX/MAS 25/44/7)**

27. There was again a brief introduction and discussion on the cocoa products and chocolate workable package. However, discussions were stopped during the VWG session on 30 April 2025, due to the end of the allotted time for the VWG session.

28. On 5 May 2025, the VWG resumed the work from the stoppage point during the previous session and began the review of the cocoa products and chocolate workable package (CX/MAS 25/44/7).

29. The VWG recommends:

- a further review on the provision for “Cocoa Butter (determined as fat)” and whether this provision requires a moisture (determined as water) calculation.
- endorsement of the methods and typing for the provisions discussed as seen in Appendix V, Table 1.
- revocation of the provisions and method for “Fat, total” as this provision does not appear in CXS 87-1981 Table 1.
- further discussion and endorsement decisions on the following provisions, which the VWG did not have time to complete:
  - Cocoa (cacao) mass or cocoa / chocolate liquor, and cocoa cake, Fat
  - Cocoa butter, free fatty acids
  - Cocoa butter, Unsaponifiable matter
  - Cocoa powders (cocoa) and dry cocoa-sugar mixtures, Moisture

#### **Agenda Item 7 – Numeric performance criteria for the determination of nitrate and nitrite ions in food matrices**

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30. The Chair of the EWG gave a brief background on the questions posed to CCMAS from CCFA regarding the determination of nitrate and nitrite ions in selected food matrices. CCFA requested CCMAS to establish numeric criteria in specified food matrices, to provide information on the available methods to meet both the adopted ML and the lowest proposed ML, and some specifics on the methods such as whether the methods measure nitrate and nitrite ions separately or in combination. The VWG reviewed Appendices I and II in CX/MAS 25/44/10, and after minor editorial changes, the VWG agreed to recommend CCMAS to send the completed tables back to CCFA in response to their request.

31. The VWG recommends:

- endorsement of the numeric performance criteria including the example methods that meet the criteria in Appendix VI, Table 1; and
- informing CCFA that for some of the lowest proposed residual MLs, no method could be identified that would meet the criteria. The subcategories where no multi-laboratory method could be identified are:
  - Processed Cheese (01.6.4),
  - Cured (including salted) non-heat treated processed meat, poultry, and game products in whole pieces or cuts (08.2.1.1),
  - Salmon substitutes, caviar, and other fish roe products (09.3.3)

**Agenda Item 8 – Methods of analysis for precautionary allergen labelling**

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32. The Chair of the EWG gave a brief introduction to the request from the Codex Committee on Food Labelling (CCFL) to recommend suitable methods for the determination of allergens in foods. The EWG process was described, and the database of methods including validation data was shown (CX/MAS 25/44/11 Appendix II). It was shown that the methods and their validation data in Appendix II could be used by CCFL to select suitable methods. However, there was general agreement in the VWG that due to the late arriving EWG report, and the size of the methods and scope in CX/MAS 25/44/11 Appendix II, more time would be useful to examine and review the methods and their associated validation data. There was also a consensus that the form of the appendix containing methods should be simplified to a more user-friendly format for CCFL. Finally, it was discussed that many of the methods rely upon proprietary intellectual property, and CCMAS should include in its response that inclusion of the methods is a 'recommendation', and not an 'endorsement'.
33. Some members suggested that the EWG could be re-established for one more year before a response was sent to CCFL. It was noted that CCMAS was tentatively scheduled to meet again in 2026 prior to the next CCFL meeting.
34. The VWG recommends:
- informing CCFL of the recent publication of standard development organization guidelines including:
    - AOAC Appendix M
    - EN 17855 (ELISA)
    - EN 17644 (LC-MS)
    - EN 17254 (ELISA Gluten)
    - EN 15634 (PCR)
  - considering if the EWG should be re-established to provide CCFL a simplified response.

**Appendix I: Matters arising from other subsidiary bodies**

Table 1: Codex Committee on Fish and Fishery Products (CCFFP36, 2024)

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	<b>Comment</b>
Fish sauce	Amino acid nitrogen	AOAC 920.04 and AOAC 920.03	Determining formaldehyde titration method Subtracting by ammoniacal nitrogen (magnesium oxide method)	I	VWG recommends retaining methods as in CXS 234

Table 2: FAO/WHO Coordinating Committee for Africa (CCAFRICA25, 2025)

<b>Commodity</b>	<b>Provisions</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	<b>Comment</b>
Dried meat	Chloride as sodium chloride ( $\geq 1.0\%$ )	ISO 1841-1	Titrimetry (Volhard method)	III	VWG recommends accepting as presented
Dried meat	Chloride as sodium chloride ( $\geq 0.25\%$ )	ISO 1841-2	Titrimetry (potentiometry)	II	
<u>Dried meat</u>	<u>Chloride as sodium chloride</u>	<u>AOAC 935.47 and AOAC 937.09B</u>	<u>Titrimetry (Volhard method)</u>	<u>III</u>	

Table 3: Codex Committee on Food Additives (CCFA55, 2025)

**CXS 234: PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES**

<b>Miscellaneous products</b>					<b>Comments</b>
<b>Commodity</b>	<b>Provisions</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	
Food-grade salt	Iodine	WHO/UNICEF/ICCIDD method <sup>1</sup> Only applicable to a product which has been fortified with iodate	Titrimetry using sodium thiosulphate	IV	VWG recommends moving table and method into CXS 234  <b>Inclusion in CXS 234 requires</b> a. Fix/amend the WHO/UNICEF/ICCIDD method reference link for the Iodine method.
Food-grade salt	Sodium chloride	<del>Described in the standard</del> <u>see Appendix {XXV} Food-grade salt – Part A, Determination of Sodium Chloride content</u>	Calculation	I	

<sup>1</sup> Assessment of iodine deficiency disorders and monitoring their elimination. A guide for programme managers. Third edition, Annex 1: Titration method for determining salt iodate and salt iodine content. World Health Organization, Geneva, 2007. The report is available from <https://www.who.int/publications/i/item/9789241595827>

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|  | b. State the CXS 234 Appendix location for the Determination of Sodium Chloride.<br>c. Create an CXS 234 Appendix for 'Food Grade Salt' with a<br>-'Part A, Determination of Sodium Chloride Content'<br>-'Part B, Sampling.'<br>d. The method performance criteria for Lead and Cadmium is already included in CXS 234.<br>e. Include the Food grade salt sampling plan as a line item in CXS 234 'Part B – Methods of Sampling by Commodity Categories and Names'. |
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## **CXS 234: APPENDIX {XXV}**

### **FOOD GRADE SALT**

#### **Part A. DETERMINATION OF SODIUM CHLORIDE CONTENT**

This method allows the calculation of sodium chloride content, as provided for in CXS 150 Section 3.1, on the basis of the results of the determinations of sulphate (~~Method 9.4~~), calcium and magnesium (~~Method 9.5~~), potassium (~~Method 9.6~~) and loss on drying (~~Method 9.7~~). Convert sulphate to  $\text{CaSO}_4$  and unused calcium to  $\text{CaCl}_2$ , unless sulphate in sample exceeds the amount necessary to combine with calcium, in which case convert calcium to  $\text{CaSO}_4$  and unused sulphate first to  $\text{MgSO}_4$  and any remaining sulphate to  $\text{Na}_2\text{SO}_4$ . Convert unused magnesium to  $\text{MgCl}_2$ . Convert potassium to  $\text{KCl}$ . Convert unused halogens to  $\text{NaCl}$ . Report the  $\text{NaCl}$  content on a dry matter basis, multiplying the percentage  $\text{NaCl}$  by  $100/100-P$ , where  $P$  is the percentage loss on drying.

#### **Part B. SAMPLING: METHOD FOR THE SAMPLING OF FOOD GRADE SALT FOR THE DETERMINATION OF SODIUM CHLORIDE**

##### **1. SCOPE**

This method specifies the sampling procedure to be applied when determining the main component in order to assess the food grade quality of sodium chloride (salt) as provided for in the Codex Standard for Food Grade Salt, Section 3: "Essential Composition and Quality Factors".

The criterion to be used for acceptance or rejection of a lot or consignment on the basis of this sample is also provided.

##### **2. FIELD OF APPLICATION**

This method is applicable to the sampling of any type of salt intended for use as food, either prepacked or in bulk.

##### **3. PRINCIPLE**

This method represents a variables sampling procedure for mean quality: blended bulk sample analysis.

A blended bulk sample is produced in such a way that it is representative of the lot or consignment. It is composed of a proportion of items drawn from the lot or consignment to be analyzed.

Acceptance criterion is on the basis that the mean value obtained from analyses of those blended bulk samples must comply with the provision in the Standard.

#### **4. DEFINITIONS**

The terms used in this sampling method refer to those in the "*General Guidelines on Sampling*" (CAC/GL 50-2004) unless stated otherwise.

#### **5. EQUIPMENT**

The sampling equipment used should be adapted to the nature of the tests to be carried out (for example: sampling by borer, sampling equipment made of chemically inert material, etc.). The containers used for collecting the samples should be made of a chemically inert material and should be air-tight.

#### **6. PROCEDURE**

##### **6.1 PREPACKED SALT**

Sampling may be carried out by "random sampling" or by "systematic sampling". The choice of the method to be used depends on the nature of the lot (for example: if the packages are marked with successive numbers, systematic sampling may be suitable).

##### **6.1.1 Random sampling**

Draw the  $n$  items from the lot in such a way that each item in the lot has the same chance of being selected.

##### **6.1.2 Systematic sampling**

If the  $N$  units in the lot have been classified and can be numbered from 1 to  $N$ , the 1-in- $k$  systematic sampling of  $n$  items can be obtained as follows:

- a) Determine the  $k$  value as  $k = N/n$ . (If  $k$  is not an integer, then round to the nearest integer).
- b) From the first  $k$  items in the lot take one at random and then take every  $k^{\text{th}}$  item thereafter.

##### **6.2 SALT IN BULK**

Here, the lot is fictitiously divided into items (strata); a lot with a total mass of  $m$  kg is considered to be composed of  $m/100$  items. In this case, it is necessary to draw up a "stratified sampling" plan appropriate to the lot dimension. The samples are selected from all the strata in proportion to the stratum sizes.

Note: Stratified sampling of a population which can be divided into different subpopulations (called strata) is carried out in such a way that specified proportions of the sample are drawn from the different strata.

##### **6.3 CONSTITUTION OF THE SAMPLE**

6.3.1 The size and the number of the items forming the sample depend on the type of salt and the lot magnitude. The minimum size to be taken into account should be in accordance with one of the following specifications according to the circumstances:

- 250 g of salt in bulk or prepacked in more than 1 kg packages;
- one package for prepacked salt in 500 g or 1 kg packages.

The appropriate number of samples to be drawn from the lot, shall be determined in accordance with "*General Guidelines on Sampling*" (CAC/GL 50-2004).



6.3.2 Combine and mix well the different items drawn from the lot. This blended bulk sample constitutes the laboratory sample. More than one laboratory sample may be composed in such a manner.

## 7. ACCEPTANCE CRITERION

7.1 Determine the NaCl content (%) of at least two test portions of the laboratory sample.

7.2 Calculate the average of the results obtained for the n test portions of the laboratory sample using:

$$\bar{x} = \frac{\sum x}{n} (n \geq 2)$$

7.3 In accordance with the provision for the relevant NaCl content (%), a lot or a consignment shall be considered acceptable if the following condition is verified:

$\bar{x} \geq$  minimum level specified.

## 8. SAMPLING REPORT

The sampling report should contain the following information:

- a) type and origin of the salt;
- b) alterations of state of the salt (e.g. presence of foreign matter);
- c) date of sampling;
- d) lot or consignment number;
- e) method of packing;
- f) total mass of lot or consignment
- g) number, unit mass of packages and whether the mass is given net or gross;
- h) number of items sampled;
- i) number, nature and initial position of sampled items;
- j) number, composition and mass of the bulk sample(s) and the method used to obtain and conserve it (them);
- k) names and signatures of the people who carried out the sampling.

### **CXS 234: PART B – METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES**

Commodity categories	Method of sampling	Notes
<u>Miscellaneous products</u>		
<u>Food-grade salt</u>	<u>see Appendix {XXV} Food-grade salt – Part B, Sampling:</u>	

**Appendix II: Methods of analysis and sampling plans for provisions in Codex standards**

Table 1: CCNFSDU44 Method for dietary fibre

General methods that measure both the higher (monomeric units > 9) and the lower molecular weight fraction (monomeric units ≤9)					Comments
Standard	Provisions	Method	Principle	Type	VWG recommends endorsement including the footnote
All foods	Method applicable for determining the content of insoluble and soluble dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches.	<a href="#">AOAC 2022.01/</a> <a href="#">AACC 32-61.01/</a> <a href="#">ICC Standard No. 191</a>	Enzymatic-Gravimetry and High Pressure Liquid Chromatography (HPLC)	I	

\*Isolated, purified, and/or synthetic fibres captured by AOAC 2022.01/ICC Standard 191/AACC 32-61.01 that do not meet the Codex definition of dietary fibre in the *Guidelines on nutrition labelling* (CXG 2-1985) should be subtracted from the final measurement, where deemed appropriate by competent authorities.

Table 2: Methods for provisions in follow-up formula and infant formula

Commodity	Provision	Method	Principle	Type	Comments
Follow-up formula	Vitamin A palmitate (retinyl palmitate), Vitamin A acetate (retinyl acetate)	AOAC 2012.10 / ISO 20633	HPLC-UV	II	VWG Recommends Endorsement
	Vitamin A	EN 12823-1 (all-trans-retinol and 13-cis-retinol) Vitamin A (both natural + supplemental ester forms) aggregated and quantified as individual retinol isomers (13-cis and all-trans)	HPLC-UV or FL	III	
	Vitamin E	AOAC 2012.10 / ISO 20633	HPLC-UV	II	VWG Recommends Endorsement
		EN 12822 (Measures vitamin E (both natural + supplemental ester forms) aggregated and quantified as individual tocopherol congeners (α, β, γ, δ))	HPLC-UV or FL	III	

Commodity	Provision	Method	Principle	Type	Comments
	Vitamin D	AOAC 992.03 Measures all rac-vitamin E (both natural + supplemental ester forms) aggregated and quantified as $\alpha$ -congeners	HPLC-UV	III	
		AOAC 2016.05 / ISO 20636	LC-MS	II	VWG Recommends Endorsement
		EN 12821 / NMKL 167 (D2 and/or D3 measured as single components. Hydroxylated forms not measured)	HPLC-UV	III	
		AOAC 995.05 D2 and D3 measured	HPLC-UV	III	
	Thiamine	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II	VWG Recommends Endorsement
		AOAC 986.27 <sup>2</sup>	Fluorimetry	III	
		EN 14122 (Measures all vitamin B1 forms (natural and added free, bound and phosphorylated) following extraction and conversion to thiamine)	HPLC-FL (with pre-or post-column derivatization to thiochrome)	III	
	Riboflavin	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II	VWG Recommends Endorsement
		EN 14152 (Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin)	HPLC-FL	III	

<sup>2</sup> Care should be taken in the application of the method due to spectral interference.

Commodity	Provision	Method	Principle	Type	Comments
	Niacin	AOAC 985.31 <sup>2</sup>	Fluorimetry	III	
		AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II	VWG Recommends Endorsement
		EN 15652 (Free and bound and phosphorylated forms measured either as aggregate of nicotinic acid + nicotinamide, or as individual forms)	HPLC-FL (with post-column photochemical derivatization)	III	
		AOAC 985.34 (niacin (preformed) and nicotinamide)	Microbioassay and turbidimetry	III	
	Vitamin B <sub>6</sub>	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II	VWG Recommends Endorsement
		EN 14166 (Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine)	Microbioassay	III	
		AOAC 985.32	Microbioassay	III	
		AOAC 2004.07 / EN 14164 (Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine)	HPLC-FL	III	
	Vitamin B <sub>12</sub>	AOAC 2011.10 / ISO 20634	HPLC-VIS	II	VWG Recommends Endorsement
		AOAC 986.23 (Measures total vitamin B12 as cyanocobalamin)	Turbidimetry	III	
		AOAC 2014.02	LC-UV	III	VWG Recommends Endorsement

Commodity	Provision	Method	Principle	Type	Comments
	Pantothenic acid	AOAC 2012.16 / ISO 20639	UHPLC-MS/MS	II	VWG Recommends Endorsement
	Folic Acid	EN 14131 (Total folate (free + bound), aggregated and measured as folic acid)	Microbio assay	III	
		AOAC 992.05 (Measures free folic acid + free, unbound natural folates, aggregated, and measured as folic acid)	Microbio assay	III	
		AOAC 2011.06 / ISO 20631	LC-MS/MS	II	VWG Recommends Endorsement
	Vitamin C	AOAC 2012.22 / ISO 20635	HPLC-UV	II	VWG Recommends Endorsement
	Biotin	AOAC 2016.02 / ISO 23305	HPLC-UV	II	VWG Recommends Endorsement
		EN 15607 (d-biotin) (Measures total D-biotin [free + D-biocytyl])	HPLC- FLD (post-column derivatization)	III	
	Iron	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement
		AOAC 985.35	FAAS	III	
		AOAC 999.11   NMKL 139	AAS after dry ashing	III	
	Calcium	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement

Commodity	Provision	Method	Principle	Type	Comments
		ISO 8070 IDF 119	FAAS	III	
		AOAC 985.35	FAAS	III	
	Phosphorus	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement
		AOAC 986.24	Spectrophotometry (molybdovanadate)	III	
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement
	Magnesium	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement
		ISO 8070 IDF 119	FAAS	III	
		AOAC 985.35	FAAS	III	
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement
	Sodium	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement
		ISO 8070 IDF 119	FAAS	III	
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement
	Chloride	AOAC 2016.03 / ISO 21422   IDF 242	Potentiometry	II	VWG Recommends Endorsement
		AOAC 986.26	Potentiometry	III	
	Potassium	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement
		ISO 8070 IDF 119	FAAS	III	
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement
	Manganese	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement

Commodity	Provision	Method	Principle	Type	Comments
		AOAC 985.35	FAAS	III	
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement
	Iodine	AOAC 2012.15 / ISO 20647   IDF 234	ICP-MS	II	VWG Recommends Endorsement
	Selenium	AOAC 2011.19 / ISO 20649   IDF 235	ICP-MS	II	VWG Recommends Endorsement
		AOAC 2006.03	ICP-OES	III	
		EN 14627	HGAAS	III	
		AOAC 996.16	Fluorimetry	III	
		AOAC 996.17	HGAAS	III	
	Copper	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement
		AOAC 985.35	FAAS	III	
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement
	Zinc	AOAC 2015.06 / ISO 21424   IDF 243	ICP-MS	II	VWG Recommends Endorsement
		AOAC 985.35	FAAS	III	
		AOAC 2011.14 / ISO 15151   IDF 229	ICP-OES	III	VWG Recommends Endorsement
	Total nucleotides	AOAC 2011.20 / ISO 20638	LC SPE -LC-UV	II	VWG Recommends Endorsement
	Choline	AOAC 999.14	Enzymatic colorimetric method with limitations on applicability due to choline and ascorbate concentration	III	

Commodity	Provision	Method	Principle	Type	Comments
		AOAC 2015.10 / ISO 21468	UHPLC-MS/MS	II	VWG Recommends Endorsement
	Myo-inositol	AOAC 2011.18 / ISO 20637	LC-pulsed amperometry	II	VWG Recommends Endorsement
	L-carnitine	AOAC 2015.10 / ISO 21468	UHPLC-MS/MS	II	VWG Recommends Endorsement
	Total amino acids (excluding taurine and tryptophan) for use according to section 3.1.3 (a) notes 2) and 3) of CXS 156-1987	AOAC 2018.06 / ISO 4214   IDF 254 / AACC 07-50.01	UHPLC-UV	II	VWG Recommends Endorsement
	Tryptophan	AOAC 2017.03 For use according to Section 3.1.3 (a) notes 2 and 3 of CXS 72-1981	HPLC-FLD	II	VWG Recommends Endorsement
	Total fatty acids	AOAC 2012.13 / ISO 16958   IDF 231	GC-FID	II	VWG Recommends Endorsement
		AOAC 996.06	Gas chromatography	III	
	Crude protein	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	I	VWG Recommends Endorsement
Infant formula	Folic acid	AOAC 2011.06 / <b><u>ISO 20631</u></b>	LC-MS/MS	II	VWG Recommends Endorsement of ISO 20631



Table 3: Methods of Analysis included in CXS 234-1999 for revocation or retyping

Commodity	Provision	Method	Principle	Type	Comments/ Notes
All Foods	Method applicable for determining the content of insoluble and soluble dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches.	<del>AACC Int'l 32-50.01</del> AOAC 2011.25	<del>Enzymatic Gravimetry</del> High Pressure Liquid Chromatography	I	VWG recommends revocation
Follow-up formula	Vitamin A	AOAC 992.04	HPLC-UV	III	Retype as III
		AOAC 992.06	HPLC-UV	III	unchanged
		<del>AOAC 974.29</del>	<del>Colorimetry</del>	<del>IV</del>	Revoke
	Pantothenic acid	AOAC 992.07	Microbioassay	II III	Retype to III
	Iodine	<del>AOAC 992.24</del>	<del>Ion selective potentiometry</del>	III	Revoke method due to method measuring iodide only and not iodine

**Appendix III: Other relevant matters arising from the amendment of CXS 234-1999**

Table 1: Methods of analysis where the principle and typing were not indicated in the respective commodity standards

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	<b>Comments</b>
<u>Fish and fishery products</u>					
Crackers from marine and freshwater fish, crustacean and molluscan shellfish	Crude protein	AOAC 920.87 or 960.52			Suggestion to complete these tables via EWG inclusive of the fats and oils, fishery products, CPL and other commodity categories for CCMAS45
Crackers from marine and freshwater fish, crustacean and molluscan shellfish	Moisture	AOAC 950.46B (air drying)			
Raw bivalve molluscs (shucked)	Drained weight	AOAC 953.11			
<u>Fats and oils</u>					
Edible Fats and Oils not Covered by Individual Standards	Peroxide Value	ISO 3961:1998			
Edible Fats and Oils not Covered by Individual Standards	Soap content	BS 684 Section 2.65			
Named animal fats	Fatty acid composition	ISO 5508: 1995/ 5509: 1999			
Named animal fats	Soap content	BS 684 Section 2.5			
Fat Spreads and Blended Spreads	Milk fat content (Butyric acid)	AOAC 990.27; AOCS Ca 5c-87 (97)			
Fat Spreads and Blended Spreads	Salt content	IDF 12B: 1988, ISO CD 1738 or AOAC 960.29.			
Fat Spreads and Blended Spreads	Vitamin A	AOAC 985.30; AOAC 992.04; or JAOAC 1980, <u>63</u> , 4.			
Fat Spreads and Blended Spreads	Vitamin D	AOAC 981.17			
Fat Spreads and Blended Spreads	Vitamin E	ISO 9936:1997			

<i>Commodity</i>	<i>Provision</i>	<i>Method</i>	<i>Principle</i>	<i>Type</i>	<i>Comments</i>
<u>Cereals, pulses and legumes</u>					
Maize (corn)	Broken kernels	ISO 5223-1983 <sup>3</sup>			
Sorghum grains	Fibre, crude	ICC 113		I	
Sorghum grains	Fibre, crude	ISO 6541 (1981)			
Rice	Head rice	ISO 7301 (Annex A)			
Rice	Large broken kernel	ISO 7301 (Annex A)			
Rice	Medium broken kernel	ISO 7301 (Annex A)			
Rice	Small broken kernel	ISO 7301 (Annex A)			
Rice	Chips	ISO 7301 (Annex A)			
Rice	Heat-damaged kernels	ISO 7301 (Annex A)			
Rice	Damaged kernels	ISO 7301 (Annex A)			
Rice	Immature kernels	ISO 7301 (Annex A)			
Rice	Chalky kernels	ISO 7301 (Annex A)			
Rice	Red kernels	ISO 7301 (Annex A)			

#### **APPENDIX \*: DETERMINATION OF MINIMUM TEST WEIGHT IN CEREALS, PULSES AND LEGUMES**

##### **Oats**

The test weight shall be the weight per ISO 7971-1986 or any other equipment giving equivalent results expressed as kilograms per hectolitre as determined on a test portion of the original sample

##### **Wheat and durum wheat**

The test weight shall be the weight per ISO 7971-1986 expressed in kilograms per hectolitre as determined on a test portion of the original sample.

<sup>3</sup> 4.50 mm metal sieve

Table 2: Method of analysis by description, where specific references to published methods have been provided

VWG recommended option 2

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	<b>Comments</b>
<u>Fish and fishery products</u>					
Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	Determination of fish content (declaration) – Nitrogen	ISO 937	Distillation (Kjeldahl)	II	Principle naming should be aligned with other principles in CXS 234
Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	Determination of fish content (declaration) – Moisture	ISO 1442	Gravimetry	II	
Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	Determination of fish content (declaration) – Total fat	ISO 1443	Gravimetry	II	
Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	Determination of fish content (declaration) – Ash	ISO 936	Gravimetry	II	

Table 3: Revised methods for the determination of chloride expressed as sodium chloride for butter and cheese

Commodity	Provision	Method	Principle	<del>Codex STAN</del>	Proposed Type	Comment
Butter	Salt ( <u>Determined as chloride expressed as NaCl</u> )	AOAC 2016.03 / ISO 21422   IDF 242	Potentiometry ( <del>Determination of chloride, expressed as sodium chloride</del> )	<del>279</del>	III	VWG recommends endorsing as type III
Butter	Salt ( <u>Determined as chloride, expressed as NaCl</u> )	ISO 15648   IDF 179	Potentiometry ( <del>Determination of chloride, expressed as sodium chloride</del> )	<del>279</del>	II	VWG recommends retaining as type II
Cheese	Sodium Chloride ( <u>Determined as chloride, expressed as NaCl</u> )	AOAC 2016.03 / ISO 21422   IDF 242	Potentiometry ( <del>Determination of chloride, expressed as sodium chloride</del> )	<del>283</del>	III	VWG recommends endorsing as type III
Cheese	Sodium chloride ( <u>Determined as chloride, expressed as NaCl</u> )	ISO 5943   IDF 88	Potentiometry ( <del>determination of chloride, expressed as sodium chloride</del> )	<del>283</del>	II	VWG recommends retaining as type II

Table 4: Numeric Performance Criteria for methods of analysis for copper and iron in milk fat products and edible casein products

Commodity	Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery	Minimum Applicable Range		Examples of applicable methods that meet the criteria	Principle	Comments
							Minimum	Maximum			
Milk fat products	Copper	0.05	0.010	0.020	44.0	60-115%	0.028	0.072	AOAC 2015.06 / ISO 21424   IDF 243	ICP mass spectrometry	VWG recommends endorsing numeric performance criteria
									ISO 5738   IDF 76	Photometry, (diethyldithiocarbamate)	Remove
									AOAC 960.40	Photometry, (diethyldithiocarbamate)	Remove

Table 5: Amendments to numeric performance criteria in fish sauce

Commodity	Provision	ML (%)	Min. Appl. Range (%)	LOD (%)	LOQ (%)	Precision (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle	Comment
Fish Sauce	Sodium chloride and Salt determined as Chloride expressed as Sodium chloride	<del>20 (NaCl)</del> Minimum limit <b>From 20 (NaCl)</b>  <b>From 12 (Cl<sup>-</sup>)</b>	18 - <b>22</b>  11 - <b>13</b>	2.0  1.2	4.0  2.4	5.1  5.5	98-102	NMKL 178  AOAC 971.27  AOAC 937.09  AOAC 976.18	Potentiometric titration Potentiometric titration Titration  <b>Potentiometric titration</b>	VWG recommends performance criteria and the example methods as shown
Salted Atlantic herring and salted sprat	Sodium chloride and Salt determined as Chloride expressed as Sodium chloride	From 1 to 20 (NaCl)  From 0.6 to 12 (Cl <sup>-</sup> )	0.9 – 22  0.5 - 13	0.1  0.06	0.2  0.12	8.0  8.6	97-103	NMKL 178  AOAC 971.27  AOAC 976.18	Potentiometric titration Potentiometric titration Potentiometric titration Titration	VWG recommends performance criteria and the example methods as shown
Salted Fish and dried salted fish of Gadidae family of fishes		From 12 (NaCl)  From 7.3 (Cl <sup>-</sup> )	11 – 13  6.8 – 8.1	1.2  0.8	2.4  1.5	5.5  5.9	98-102	AOAC 937.09		

Commodity	Provision	ML (%)	Min. Appl. Range (%)	LOD (%)	LOQ (%)	Precision (RSD <sub>R</sub> ) (%) No more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle	Comment
Sturgeon Caviar		From 3 to 5 (NaCl)	2.7 -5.5	0.3	0.6	6.8	97-103			
		From 1.8 to 3.0 (Cl <sup>-</sup> )	1.7 – 3.4	0.2	0.4	7.3				

\* The highest value in the Min. Appl. Range is calculated by using the highest value of the ML, while the rest i.e. the lowest value in the Min. Appl. Range, LOD, LOQ, Precision, and Recovery are calculated based on the lowest value of the ML.

Table 6: Methods of analysis that appear in cereals, pulses and legumes (CPL) standards but not in CXS 234-1999

Commodity	Provision	Method	Principle	Type	Comments
<b><u>Cereals, pulses and legumes</u></b>					
Maize (corn)	Broken kernels	ISO 5223-1983 <sup>4</sup>			Suggestion to complete these tables via EWG inclusive of the fats and oils, fishery products, CPL and other commodity categories for CCMAS45
Sorghum grains	Fibre, crude	ICC 113		I	
Sorghum grains	Fibre, crude	ISO 6541 (1981)			
Rice	Head rice	ISO 7301 (Annex A)			
Rice	Large broken kernel	ISO 7301 (Annex A)			
Rice	Medium broken kernel	ISO 7301 (Annex A)			
Rice	Small broken kernel	ISO 7301 (Annex A)			
Rice	Chips	ISO 7301 (Annex A)			
Rice	Heat-damaged kernels	ISO 7301 (Annex A)			
Rice	Damaged kernels	ISO 7301 (Annex A)			
Rice	Immature kernels	ISO 7301 (Annex A)			

<sup>4</sup> 4.50 mm metal sieve

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	<b>Comments</b>
<b><u>Cereals, pulses and legumes</u></b>					
Rice	Chalky kernels	ISO 7301 (Annex A)			
Rice	Red kernels	ISO 7301 (Annex A)			
Rice	Red-streaked kernels	ISO 7301 (Annex A)			
Rice	Pecks	ISO 7301 (Annex A)			
Rice	Maximum recommended levels of other types of rice	ISO 7301 (Annex A)			
Wheat and durum wheat	Minimum test weight	See Appendix *			
Wheat and durum wheat	Shrunken and broken kernels	ISO 5223-1983			
Wheat and durum wheat	Edible grains other than wheat and durum wheat	ISO 7970-1987: (Annex C)			
Wheat and durum wheat	Damaged kernels	ISO 7970-1987: (Annex C)			
Oats	Minimum test weight	See Appendix *			



Table 7: Methods of analysis indicated in cereals, pulses and legumes (CPL) standards to be under development or to be determined

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	<b>Comments</b>
<b><u>Cereals, pulses and legumes</u></b>					
Wheat and durum wheat	Insect bored kernels	To be developed			
Oats	Hull-less and broken kernels	To be developed			
Oats	Edible grains other than oats	To be developed			
Oats	Damaged kernels	To be developed			
Oats	Wild oats	To be developed			
Oats	Insect bored kernels	To be developed			
Oats	Blemished grains	To be developed			
Peanuts	In-pod defects: Empty pods	To be determined			
Peanuts	In-pod defects: Damaged pods	To be determined			
Peanuts	In-pod defects: Discoloured pods	To be determined			
Peanuts	Kernel defects: Damaged kernels	To be determined			
Peanuts	Kernel defects: Discoloured kernels	To be determined			
Peanuts	Kernel defects: Broken and split kernels	To be determined			
Peanuts	Peanuts other than the designated type	To be determined			

Table 8: Methods of analysis proposed for revocation consequential to the decisions of CAC46

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	<b>Comments</b>
<b><u>Cereals, pulses and legumes</u></b>					
Degermed maize (corn) meal and maize (corn) grits	Protein (N x 6.25)	ISO 1871: 1975			
Degermed maize (corn) meal and maize (corn) grits	Crude fat	ISO 5986: 1983			
<b><u>Fats and oils</u></b>					
Named vegetable oils	Fatty acid composition	ISO 5509: 2000			

**Appendix IV: Matters pending from CCMAS43**

Table 1: Determination of moisture in whey powders

<b>Commodity</b>	<b>Provision</b>	<b>Method</b>	<b>Principle</b>	<b>Type</b>	<b>Comments</b>
<b><u>Milk and milk products</u></b>					
Whey powders	Water <sup>xiii</sup> (moisture)	ISO 5537   IDF 26	Gravimetry (drying at 87°C)	I	Existing
Whey powders	Water <sup>xiii</sup> (moisture) *	Described in Appendix XI	Gravimetry (drying at 102°C)	IV	Endorsed as IV w footnote

<sup>xiii</sup> Water content excluding the crystallized water bound to lactose (generally known as moisture content).

\* 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. If trading partners agree, the Type IV method can be used in international trade; otherwise ISO 5537 | IDF 26 should be used. In a dispute situation, the Type I method shall be used. This 102°C method is less precise, and results may not be consistent with results obtained with ISO5537 | IDF 26, in particular for powders with high natural lactose such as whey powders.

**Appendix V: Review of methods of analysis in CXS 234: Cocoa products and chocolate workable package**

Table 1: CXS 234-1999 method review and recommended revisions

Commodity	Provision	Method	Principle	Type	Standard	Committee	Comments
Chocolate and chocolate products	Cocoa butter <b>(determined as fat)</b>	<u>ICA No. 26 / AOAC 977.10</u> and AOAC 963.15 / <u>ICA No.14</u> IOCCC 14	<b>Calculation from moisture (Determined as Water) and Gravimetry</b> (Soxhlet extraction)	I	<u>CXS 87-1981</u>	<u>CCCPC</u>	CCMAS plenary will make final decision;  Q: does AOAC 963.15 have a moisture correction?
Chocolate and chocolate products	Fat-free cocoa solids	<u>ICA No. 26 / AOAC 977.10</u> and AOAC 931.05	Oven evaporation and factor <b>Calculation from moisture (Determined as Water) and gravimetry</b>	I	<u>CXS 87-1981</u>	<u>CCCPC</u>	VWG recommends endorsement as type I
Chocolate and chocolate products	Fat-free milk solids <b>(Determined as Milk Protein)</b>	<u>ICA No. 26 and ICA No.17 and ICA No.13 or / AOAC 977.10 and AOAC 955.04C and IOCCC 17 or AOAC 939.02</u>	Titrimetry, Kjeldahl digestion; after extraction of milk proteins <b>Calculation from moisture content, and Titrimetry (Kjeldahl digestion) content of extracted and precipitated milk protein.</b>	I	<u>CXS 87-1981</u>	<u>CCCPC</u>	VWG recommends endorsement as type I
Chocolate and chocolate products	Fat, total <b>on dry basis</b>	<u>ICA No. 26 / AOAC 977.10 and AOAC 963.15</u>	<b>Calculation from moisture (Determined as Water) and Gravimetry</b> (Soxhlet extraction)	I	<u>CXS 87-1981</u>	<u>CCCPC</u>	No specification for total fat in CXS 87 table 1. VWG recommends removing this entry in CXS 234

Commodity	Provision	Method	Principle	Type	Standard	Committee	Comments
Chocolate and chocolate products	Milk Fat	<u>ICA No. 5</u> <del>IOCCC 5</del> AOAC 945.34; 925.41B; 920.80	Titrimetry/Distillation	† <u>IV</u>	<u>CXS 87-1981</u>	<u>CCCPC</u>	VWG recommends endorsement as type IV
Chocolate and chocolate products	Moisture	<u>ICA No. 1</u> <del>IOCCC 26 or</del> AOAC 977.10 (Karl Fischer method)	Gravimetry- <u>drying at 100-102° C</u>	† <u>IV</u>	<u>CXS 87-1981</u>	<u>CCCPC</u>	VWG recommends endorsement as type IV. <b>Further discussion may be required about coexistence of a type II rational method and type IV defining method</b>
<u>Chocolate and chocolate products</u>	<u>Moisture (Determined as Water)</u>	<u>ICA No. 26 / AOAC 977.10</u>	<u>Titrimetry - Karl Fischer</u>	<u>II</u>	<u>CXS 87-1981</u>	<u>CCCPC</u>	VWG recommends endorsement as type II
Chocolate and chocolate products	Non-cocoa butter vegetable fat	AOCS Ce 10-02	<u>Gas chromatography</u>	<u>IV</u>	<u>CXS 87-1981</u>	<u>CCCPC</u>	VWG recommends endorsement as type IV
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Cocoa shell (determined as spiral vessel count)	AOAC 968.10	<u>Microscopy - Spiral vessel count</u>	I	<u>CXS 141-1983</u>	<u>CCCPC</u>	VWG recommends endorsement as type I, but suggested to send comment back to CAC on provision name change
<u>Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake</u>	<u>Cocoa shell (determined as stone cell count)</u>	<u>AOAC 970.23</u>	<u>Microscopy - Stone cell count</u>	I	<u>CXS 141-1983</u>	<u>CCCPC</u>	VWG recommends endorsement as type I, but suggested to send comment back to CAC on provision name change

Commodity	Provision	Method	Principle	Type	Standard	Committee	Comments
Cocoa (cacao) mass or cocoa/chocolate liquor, and cocoa cake	Fat	<del>ICCGG ICA No. 14</del> <u>ICA No. 26 / AOAC 977.10 and AOAC 963.15 / ICA No. 14</u>	<del>Gravimetry</del> <u>Calculation from moisture (Determined as Water) and Gravimetry (Soxhlet extraction)</u>	I	<u>CXS 141-1983</u>	<u>CCCPC</u>	Comment: Provision name should be "Fat on dry basis"; Fat/Cocoa butter content can be determined by direct soxhlet with higher precision (ICA-37). We recommend adding ICA-37, and making the method applicable for cocoa liquor/mass/cake. ICA-37 is already common and generates less chemical waste.  For water content, Karl Fischer titration is good for cocoa mass because it has a low (~1%) water content that makes gravimetric determination less reliable. For cocoa cake, drying at 103 C is more applicable.
Cocoa butter	Free fatty acids	<del>ISO 660 or / AOCS Cd 3d-63</del>	Titrimetry	I	<u>CXS 86-1981</u>	<u>CCCPC</u>	
Cocoa butter	Unsaponifiable matter	<del>ISO 3596 or / ISO 18609 or / AOCS Ca 6b-53</del>	Titrimetry after extraction with diethyl ether	I	<u>CXS 86-1981</u>	<u>CCCPC</u>	Comment: ISO 3596 and AOCS Ca 6b-53 use diethyl ether.
Cocoa powders (cocoa) and dry cocoa-sugar mixtures	Moisture <u>(Determined as Water)</u>	<del>ICCGG ICA No. 26 / AOAC 977.10</del>	<del>Gravimetry</del> <u>Titrimetry - Karl Fischer</u>	<del>I</del> II	<u>CXS 105 - 1981</u>	<u>CCCPC</u>	

Table 2: Proposed methods for cocoa and chocolate products in CXS 234-1999

**Cocoa products and chocolate**

Commodity	Provision	Method	Principle	Type	Standard	Committee	Comments
Chocolate and chocolate products	Cocoa butter equivalents in cocoa butter and plain chocolate	ISO 23275-1 and ISO 23275-2 / AOCS Ce 11-05	GC-FID	I	CXS 87-1981	CCCPC	IRMM 801 will be available next year; limited availability. Both ISO methods are necessary. There was subsequent information provided by a member stating Cacao butter reference mixture (IRMM 801) is available...although it is not listed as a CRM'
Chocolate and chocolate products	Cocoa Butter Equivalents in Milk Chocolate	ISO 11053 / AOCS Ce 11a-07	GC-FID	I	CXS 87-1981	CCCPC	

**Appendix VI: Numeric Performance Criteria for Nitrate and Nitrite Ions in Certain Food Matrices**

Table 1: Numeric Performance Criteria for adopted MLs

Food Additive	Subcategory for which value was provided	Adopted Maximum Levels (CXS 192-1995)*	Calculated method performance criteria based on Maximum level (mg/kg)					
			Min Appl. Range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD <sub>R</sub> (%))	Recovery (%)	Examples of applicable methods that meet the criteria
01.6 (Cheese and analogues)								
Nitrate	01.6.2 (Ripened cheese)	35 mg/kg as residual NO <sub>3</sub> ion.	<u>25 – 45</u>	3.5	7	<u>19</u>	80 – 110	<b>Multi-laboratory validation</b> - ISO 14673-3   IDF 189-3: 2004 <b>Single-laboratory validation</b> - ISO 14673-2   IDF 189-2: 2004^
08.0 (Meat and meat products, including poultry and game)								
Nitrite	08.2.2 (Heat-treated processed meat, poultry, and game products in whole pieces or cuts/)	80 mg/kg as residual NO <sub>2</sub> ion.	<del>100 – 160</del> <u>60 – 110</u>	<del>13</del> <u>8</u>	<del>26</del> <u>16</u>	<del>15.4</del> <u>17</u>	<del>90 – 107</del> <u>80 – 110</u>	<b>Multi-laboratory validation</b> - AOAC Method 973.31; NMKL 165: 2000 Ed.; <b>Single-laboratory validation</b> - Afanda et al., (2025); lammarino et al. 2013; Ferreira et al. (2008) for Ham; Siu et al., 1998 for Salami and Ham
Nitrite	08.3 (Processed comminuted meat, poultry, and game products)	80 mg/kg as residual NO <sub>2</sub> ion.	<del>100 – 160</del> <u>60 – 110</u>	<del>13</del> <u>8</u>	<del>26</del> <u>16</u>	<del>15.4</del> <u>17</u>	<del>90 – 107</del> <u>80 – 110</u>	<b>Multi-laboratory validation</b> - AOAC Method 973.31; NMKL 165: 2000 Ed.; <b>Single-laboratory validation</b> - Afanda et al., (2025); lammarino et al., 2013; Ferreira et al., (2008) for Ham; Siu et al., 1998 for Salami and Ham

Notes: \* Maximum levels specification in CXS 192-1995 Revision 2024.

<sup>^</sup> In the absence of LOD or LOQ being specified in the method, the collaborative study report being unavailable at-this-time, and relying on in-house validation data, the validation designated as SLV, although the SLV status may be reviewed with additional data.

Table 2: Numeric performance criteria for the lowest proposed residual MLs for representative provisions in dairy (cheese), meat, and seafood as provided in CX/FA 21/52/7 Appendix 5 Annex 2.

Food Additive	Subcategory for which value was provided	Lowest Proposed Residual ML (mg/kg)	Notes	Calculated method performance criteria based on the Lowest Proposed Residual ML					Examples of applicable methods that meet the criteria
				Min Appl. Range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD <sub>R</sub> (%))	Recovery (%)	
01.6 (Cheese and analogues)									
Nitrate	01.6.2.1 (Ripened cheese, includes rind)	7	As NO <sub>3</sub>	4.5 – 9.5	0.7	1.4	<u>24</u>	80 – 110	Multi-laboratory validation - ISO 14673-3 I IDF 189-3: 2004 Single-laboratory validation - ISO 14673-2 I IDF 189-2: 2004^
Nitrite	01.6.4 <del>4</del> (Processed cheese) <i>*(see note 1)</i>	2	As NO <sub>2</sub>	1.1 – 2.9	0.2	0.4	<u>29</u>	80 – 110	Multi-laboratory validation – not available. Single-laboratory validation – not available.
08.0 (Meat and meat products, including poultry and game)									
Nitrate	Same residual proposed in multiple food categories including 08.2.1.1 (Cured (including salted) non-heat treated processed meat, poultry, and game products in whole pieces or cuts)	7	As NO <sub>3</sub>	4.5 – 9.5	0.7	1.4	<u>24</u>	80 – 110	Multi-laboratory validation – not available. Single-laboratory validation - Afanda, et al.,(2025); Ferreira et al., (2008) for Ham
Nitrite	08.2.1.3 (Fermented non-heat treated processed meat, poultry, and game products in whole pieces or cuts)	33	As NO <sub>2</sub>	<u>24 – 42</u>	3.3	6.6	<u>19</u>	80 – 110	Multi-laboratory validation - EN 12014-3:2005, NMKL 165: 2000 Ed.; AOAC Method 973.31; Single-laboratory validation - Afanda, et al., (2025), Ferreira et al., (2008) for Ham; Siu et al., 1998 for Salami, Ham
09.0 (Fish and fish products, including molluscs, crustaceans, and echinoderms)									
Nitrite	09.3.3 (Salmon substitutes, caviar, and other fish roe products)	4.4	As NO <sub>2</sub>	2.7 – 6.1	0.44	0.88	<u>26</u>	80 – 110	Multi-laboratory validation – not available. Single-laboratory validation – not available.

Notes:

1. The subcategory doesn't match the description in Annex 2, as Food category No. 01.6.1 is 'Unripened cheese'; while Food Category No 01.6.4 is 'Processed Cheese'.

<sup>^</sup>. In the absence of LOD or LOQ being specified in the method, the collaborative study report being unavailable at-this-time, and relying on in-house validation data, the validation designated as SLV, although the SLV status may be reviewed with additional data).