



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

**43rd Session**

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**REVIEW OF METHODS OF ANALYSIS IN CXS 234  
CEREALS, PULSES AND LEGUMES WORKABLE PACKAGE**

*(Prepared by the EWG led by Canada)*

Codex Members and Observers wishing to submit comments on the recommendations in this document should do so as instructed in CL 2024/14-MAS available on the Codex webpage/Circular Letters:

<https://www.fao.org/fao-who-codexalimentarius/resources/circular-letters/en/>

## **INTRODUCTION**

1. CCMAS38 (2017) agreed to continue the review and update of the *General Standard for Methods of Analysis and Sampling* (CXs 234-1999) as workable packages as described in CX/MAS 17/38/6.
2. CCMAS39 (2018) agreed to proceed with the update on workable packages for (i) cereals, pulses and legumes (CPL) and (ii) fats and oils, led by AACCI (The American Association of Cereal Chemists International)<sup>1</sup> and AOCS (American Oil Chemists' Society), respectively. All interested members and Standards Development Organizations (SDOs) were invited to assist in this work, as appropriate. The initial work to organize and establish a review of the CPL methods was led by AACCI following the protocol used by IDF (International Dairy Federation), ISO (International Organization for Standardization) and AOAC during the work on the pilot workable package (methods for milk and milk products).
3. AACCI reviewed the methods of cereals, pulses and legumes in CXs 234-1999 as follows:
  - AACCI noted the development of the Method Review sheets by New Zealand that included the review questions, information from the commodity standards, and additional relevant information. AACCI concluded that this approach would provide a basic framework for CPL method reviews across multiple analytes and matrices.
  - AACCI began by identifying all methods in CXs 234 that were applicable to CPL matrices.
4. AACCI presented the report of their work from 2018 (CX/MAS 19/40/3 Add.2) to CCMAS40 (2019) for consideration.
5. CCMAS40 (2019) agreed that AACCI, together with AOAC and ISO, would continue working on the CPL package and report back to the next session of CCMAS.
6. CCMAS41 (2021) noted the report from the observer from AACCI (CX/MAS 21/41/6) describing the progress made and that the developed workbooks<sup>2</sup> were being reviewed by relevant SDOs. It was clarified that the purpose of the review was to ensure that the methods of analysis listed in CXs 234 are fit-for-purpose and to re-type if necessary to facilitate the review process, but not to add new methods unless necessary. CCMAS noted that good progress had been made by the relevant SDOs and, in line with the review of previous workable packages, agreed with the proposal of the Chairperson that the ongoing review of the CPL workable package should continue through an EWG. In conclusion, CCMAS agreed to establish an EWG chaired by Canada (EWG-CPL), working in English only, to continue the review on the cereals, pulses, legumes and

<sup>1</sup> Now known as Cereals and Grains Association (C & G)

<sup>2</sup> Workbooks is the term used to describe the compiled spreadsheets of method information, comments (from both the SDOs and EWG experts) and citations.

derived products workable package, and to work in close coordination with the relevant SDOs (AACCI, AOAC and ISO).

7. During CCMAS42, CPL methods were endorsed and recommended for adoption / revocation (REP/23 MAS Appendix II). There were, however, a number of methods requiring further review.

8. This report is based on the EWG activity following CCMAS42 (2023), to address methods still requiring additional consideration, as identified in REP/23 MAS Appendix II Part 3 and describes the third round of the EWG effort.

#### **EWG-CPL PROCESS 2023/24**

9. As stated in the report of the 41st session of CCMAS, the terms of reference of the EWG-CPL review were to ensure that the methods of analysis listed in CXS 234 are fit-for-purpose and to re-type, if necessary, to facilitate the review process, but not to add new methods unless necessary<sup>3</sup>.

10. As acknowledged during the first two rounds of the EWG, the Chair of the EWG noted the extensive work completed by AACCI in coordination with other SDOs, namely AOAC, ICC (International Association for Cereal Science and Technology) and ISO.

11. Methods for which there was no resolution on the method or method type during CCMAS42 resulted in the need for a third round of the EWG review. The third round of the EWG-CPL involved the review of a limited number of methods to address i) comments received during CCMAS42:

<u>Commodity</u>	<u>Provision</u>
Degermed maize (corn) meal and maize (corn) grits	Ash
Sorghum flour	Ash
Sorghum grains	Ash
Wheat flour	Ash
Whole maize (corn) meal	Ash
Quinoa	Moisture
Quinoa	Protein

and ii) methods identified in the earlier rounds of the EWG as being no longer suitable, but retained in CXS 234:

<u>Commodity</u>	<u>Provision</u>	<u>Reason new method required</u>
Gari	Particle size	General guidance only
Edible cassava flour	Particle size	General guidance only
Pearl millet flour	Colour	Instrument no longer available
Sorghum flour	Colour	Instrument no longer available
Soy protein products	Crude Protein	Use of hazardous chemicals
Vegetable protein products	Crude Protein	Use of hazardous chemicals
Soy protein products	Fat	Lack of method availability
Vegetable protein products	Fat	Lack of method availability.

12. This round of the EWG began by the EWG Chair contacting SDOs with a request to identify methods that would be suitable to address the specified provisions for the commodities identified in para. 12 ii) above.

13. SDOs provided a list of suggested methods for consideration by the EWG to address each of the provisions for the corresponding commodities.

14. The EWG review was completed using workbooks based on those prepared by AACCI, using information provided by the relevant SDOs (AACCI, AOAC, ICC and ISO). As in earlier rounds of the EWG, all Codex participants were welcome to join the review of CPL methods and it was operated through the on-line Codex forum. The list of participants in the EWG is presented in Appendix II.

15. Following receipt of a list of suggested methods from SDOs, the EWG Chair prepared assignments and shared them with participants, as in previous rounds of the EWG. Each participant was assigned methods

<sup>3</sup> [REP21/MAS](#), paras 53 - 55

to review and was reminded of the guidelines for the work and general guidance on how to proceed. The Chair of the EWG expressed the desire to have each method assigned to two independent experts. Members of the EWG were then asked to review a small number of methods for all appropriate commodities and to provide feedback.

16. The Chair circulated the workbooks, collected responses, collated them, and prepared a summary of progress. This review process also identified some issues (para 18, bullet points v, vi, vii) that may require further deliberation by CCMAS either through the working group on endorsement of methods of analysis (WG) or the EWG on CPL (if re-established).

### **RESULTS OF THE EWG CONSULTATION**

17. Appendix I lists the methods for analysis of cereals, pulses and legumes and derived products, as identified in CXS 234 and/or relevant commodity standards considered by the EWG. Where methods should be considered Type I (e.g. moisture, ash), it is important to evaluate whether they are identical in cases where more than one of them is listed for the same commodity and provision. Evaluation of multiple Type I methods requires consideration of all parameters in the method including sample weights, grind size, time, temperature and other conditions (e.g. reagents, solutions, solvents).

18. From the responses of the EWG experts, the following observations were made:

- i. Methods for proximate analyses (e.g. moisture, ash) tend to be of considerable age and were developed by SDOs to meet the needs of industry in assessing the quality of traded cereals, pulses and legumes at the time of development.
- ii. Many of the currently traded commodities were yet to be commercialized at the time methods were developed, hence many of the commodities covered in the CPL standards are not mentioned within the method scope.
- iii. Owing to the timing of the original method development, validation data are scant, where available. As a result, the validation data reported do not meet with current precision data requirements. Nonetheless, most of the methods have received positive reviews from the experts. Many of the reviewed methods are in use globally and are the subject of regular proficiency testing.
- iv. Some methods have been endorsed for matrices that are not included in the method scope.
- v. In the determination of ash content, ashing is performed at either 550°C or 900°C, depending on the organization/laboratory and global region. The methods for ash determination at the different temperatures are not identical, in part due to the difference in level of detail, in addition to the difference in temperature, leading to a re-consideration of ash methods. It has been considered appropriate to identify the methods separately at the two temperatures, requiring two different provisions (e.g. ash-550 and ash-900) in each commodity standard.
- vi. Methods were recommended for removal during previous rounds of the EWG based on availability, chemicals used, or being solely guidance in nature. Replacement methods were made available by SDOs and assessed by the EWG. The PWG on endorsement is asked to consider their fitness for purpose.
- vii. Alternative [existing] methods were suggested to replace those for certain commodity/provision pairs (e.g. protein determination in soy/vegetable protein products). They were reviewed by the EWG, taking into consideration practicability, "fitness for purpose" and "Typing".

19. Some EWG reviewers recommended replacement of methods currently in commodity standards or CXS 234 with an alternate method, other than those recommended by the SDOs. Participants were reminded that according to CCMAS guidance<sup>4</sup>, changes should be proposed through the appropriate commodity committee or, if adjourned, directly to the CCMAS for consideration by the WG on methods endorsement.

20. Appendix I was prepared based on the feedback received from EWG members during the third round of the review. The appendix explains and tracks proposed changes to CXS 234. For ease of review and comparison, the appendix provides the information (Commodity, Provision, Codex Standard, Method, Principle, Type, Committee) following the new format for CXS 234. A column has been added to identify comments for consideration.

21. Unformatted text indicates that no change was required from the current listing in CXS 234. 11) Underlined **bold** text indicates an insertion into CXS 234 and represents a change from the current CXS 234.

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<sup>4</sup> Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234

22. Text that has been struck through indicates items to be deleted.

**RECOMMENDATIONS**

23. The Committee is invited to:

- Consider Appendix I and endorse the proposed changes to CXS 234, keeping in mind the original reasoning for replacing methods (para 18, bullet points v, vi, vii).

## APPENDIX I

## Methods reviewed during EWG following CCMAS42 (Round 3 of the EWG-CPL)

Cereals, Pulses and Legumes and Derived Products							
Commodity	Provision	Codex Standard	Method	Principle	Type	Committee	Comments
Degermed maize (corn) meal and maize (corn) grits	<del>Ash</del> <b>Ash-550</b>	CXS 155-1985 (2019)	AOAC 923.03 / ISO 2171 <del>ICC Method No 104/4</del> <b>and ICC 110/1</b>	<b>Calculation from moisture and Gravimetry (incineration at 550°C)</b>	I	CCCPL	Only one Type I method is allowed for each provision, to allow for the two temperatures used in the ash determination globally, a change to the provision is suggested
	<b>Ash-900</b>		<del>AOAC 923.03 / ISO 2171 / ICC Method No 104/1</del> <b>and ICC 110/1</b>	<b>Calculation from moisture and Gravimetry (incineration at 900°C)</b>	!		
Gari	Particle size	CXS 151-1989 (2019)	<del>ISO 2591-1</del> <b>ICC Recommendation 207</b>	Sieving	† !	CCCPL	To align with CXS 151: 1250 µm aperture sieve is required
Edible Cassava flour	Particle size	CXS 176-1989 (2019)	<del>ISO 2591-1</del> <b>ICC Recommendation 207</b>	Sieving	† !	CCCPL	To align with CXS 151: 600, 1200 µm aperture sieves are required
Pearl millet flour	Colour	CXS 170-1989 (2019)	<del>Modern Cereal Chemistry, 6th Ed., D.W. Kent Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.</del> <b>ISO 16624:2020</b>	Colorimetry using (specific colour grader)	IV  !	CCCPL	
Quinoa	Moisture	CXS 333-2019 (2020)	ISO 712 / <del>AACCI 44-15.02</del>	Gravimetry (oven drying)	I	CCCPL	

Quinoa	Protein ( <del>N</del> × 6.25 in dry weight basis) <sup>1</sup>	CXS 333-2019 (2020)	ISO 1871	Titrimetry (Kjeldahl digestion)	IV	CCCPL	
Sorghum flour	Ash <b>Ash-550</b>	CXS 173-1989 (2019)	AOAC 923.03 / ISO 2171 <del>ICC 104/1</del> <b>and ISO 712 / ICC 110/1</b>	<b>Calculation from moisture and Gravimetry (incineration at 550°C)</b>	I	CCCPL	Only one Type I method is allowed for each provision, to allow for the two temperatures used in the ash determination globally, a change to the provision is suggested
	<b>Ash-900</b>		<del>AOAC 923.03 / ISO 2171 / ICC 104/1</del> <b>—900°C and ISO 712 / ICC 110/1</b>	<b>Calculation from moisture and Gravimetry (incineration at 900°C)</b>	!		
Sorghum flour	Colour	CXS 173-1989 (2019)	<del>Modern Cereal Chemistry, 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.</del> <b>ISO 16624:2020</b>	Colorimetry using (specific colour grader)	IV	CCCPL	
Sorghum grains	Ash <b>Ash-550</b>	CXS 172-1989 (2019)	AOAC 923.03 / ISO 2171 <del>ICC 104/1</del> <b>and ISO 6540</b>	<b>Calculation from moisture and Gravimetry (incineration at 550°C)</b>	I	CCCPL	Only one Type I method is allowed for each provision, to allow for the two temperatures used in the ash determination globally, a change to the provision is suggested
	<b>Ash-900</b>		<del>AOAC 923.03 / ISO 2171 / ICC 104/1</del> <b>—900°C and ISO 6540</b>	<b>Calculation from moisture and Gravimetry (incineration at 900°C)</b>	!		
Soy protein products	Fat	CXS 175-1989 (2019)	<del>CAC/RM 55 Method 1</del> <b>ISO 734: 2023</b>	Gravimetry (extraction)	I	CCVP	ISO 11085 has been proposed by EWG member

Soy protein products	<u>Crude Protein; excluding added vitamins, minerals, amino acids and food additives</u>	CXS 175-1989 (2019)	AOAC 955.04D (using factor 6.25) <b>AOCS Ba 4f-00</b> <b>AACCI 46.30</b> <b>ISO 16634-1:2008</b>	Titrimetry (Kjeldahl digestion) <b>Gravimetry (Combustion)</b>	# †  <b>IV</b> <b>IV</b> <b>IV</b>	CCVP	
Vegetable protein products	Fat	CXS 174-1989 (2019)	<del>CAC/RM 55 – Method 4</del> <b>ISO 734: 2023</b>	Gravimetry (extraction)	†  <b>!</b>	CCVP	ISO 11085 has been proposed by EWG member
Vegetable protein products	<u>Crude Protein; excluding added vitamins, minerals, amino acids and food additives</u>	CXS 174-1989 (2019)	AOAC 955.04D (using factor 6.25) <b>AOCS Ba 4f-00</b> <b>AACCI 46.30</b> <b>ISO 16634-1:2008</b>	Titrimetry (Kjeldahl digestion) <b>Gravimetry (Combustion)</b>	# †  <b>IV</b> <b>IV</b> <b>IV</b>	CCVP	
Wheat flour	<del>Ash</del> <b>Ash-550</b>  <b>Ash-900</b>	CXS 152-1985 (2019)	AOAC 923.03 / ISO 2171 <del>ICC 104/1</del>  AOAC 923.03 ISO 2171 / ICC 104/1	Gravimetry ( <b>incineration at 550°C</b> )  Gravimetry ( <b>incineration at 900°C</b> )	  <b>!</b>	CCCPL	Only one Type I method is allowed for each provision, to allow for the two temperatures used in the ash determination globally, a change to the provision is suggested
Whole maize (corn) meal	<del>Ash</del> <b>Ash-550</b>  <b>Ash-900</b>	CXS 154-1985 (2019)	AOAC 923.03 / ISO 2171 <del>ICC 104/1</del> <b>and ICC 110/1</b>  AOAC 923.03 ISO 2171 / <b>ICC 104/1 and ICC 110/1</b>	<b>Calculation from moisture and Gravimetry (incineration at 550°C)</b>  <b>Calculation from moisture and Gravimetry (incineration at 900°C)</b>	  <b>!</b>	CCCPL	Only one Type I method is allowed for each provision, to allow for the two temperatures used in the ash determination globally, a change to the provision is suggested

**List of standards developed by CCCPL and CCVP (for reference purposes)**

CXS 151-1985	Standard for Gari	CCCPL
CXS 152-1985	Standard for Wheat Flour	CCCPL
CXS 153-1985	Standard for Maize (Corn)	CCCPL
CXS 154-1985	Standard for Whole Maize (Corn) Meal	CCCPL
CXS 155-1985	Standard for Degermed Maize (Corn) Meal and Maize (Corn) Grits	CCCPL
CXS 169-1989	Standard for Whole and Decorticated Pearl Millet Grains	CCCPL
CXS 170-1989	Standard for Pearl Millet Flour	CCCPL
CXS 171-1989	Standard for Certain Pulses	CCCPL
CXS 172-1989	Standard for Sorghum Grains	CCCPL
CXS 173-1989	Standard for Sorghum Flour	CCCPL
CXS 176-1989	Standard for Edible Cassava Flour	CCCPL
CXS 178-1991	Standard for Durum Wheat Semolina and Durum Wheat Flour	CCCPL
CXS 198-1995	Standard for Rice	CCCPL
CXS 199-1995	Standard for Wheat and Durum Wheat	CCCPL
CXS 200-1995	Standard for Peanuts	CCCPL
CXS 201-1995	Standard for Oats	CCCPL
CXS 202-1995	Standard for Couscous	CCCPL
CXS 249-2006	Standard for Instant Noodles	CCCPL
CXS 333-2019	Standard for Quinoa	CCCPL
CXS 163-1987	Standard for Wheat Protein Products Including Wheat Gluten	CCVP
CXS 174-1989	General Standard for Vegetable Protein Products (VPP)	CCVP
CXS 175-1989	Standard for Soy Protein Products	CCVP

## APPENDIX II

**List of Participants**

Chair

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<b>Participant</b>	<b>Country</b>
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Clémence Gaucher	France
Vincent Jauvion	France
Blanca Margarita Castellanos Valle	Honduras
Dr. Attila Nagy	Hungary
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Dr. T.G. Shrivastav	India
Angela Townson	New Zealand
Jamodu Emmanuel Babajide	Nigeria
Codex Secretariat	Republic of Korea
Hwang Kiseon	Republic of Korea
Youngjun Kim	Republic of Korea
Heather Selig	USA
Patrick Gray	USA
Timothy Norden	USA
Laura Flores	Uruguay
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