

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
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World Health
Organization

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Agenda Item 4.2

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

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

Comments in reply to CL 2023/13/OCS-MAS

Comments of Australia, Brazil, Egypt, European Union, Iraq, Jamaica, Paraguay, Philippines, Singapore and AACC International

Background

1. This document compiles comments received through the Codex Online Commenting System (OCS) in response to CL 2023/13/OCS-MAS issued in March 2023. Under the OCS, comments are compiled in the following order: general comments are listed first, followed by comments on specific sections.

Explanatory notes on the Annex

2. The comments submitted through the OCS are hereby attached as **Annex I** and presented in table format.

GENERAL COMMENTS								
<p>Brazil appreciates the document prepared by Canada and thanks the opportunity to present the following comments:</p> <ul style="list-style-type: none"> In relation to “moisture”, we suggest including in the designation of the method, the temperature used in the analysis. Considering that the method AACC 44-17.01 was validated for pulses (all classes of beans except for soybeans), we suggest clarifying this exception in the designation of the commodity: “Certain pulses, except for soybeans”. As the methods proposed for gari are not validated for this matrix, we are of the opinion that it should be retained as type IV. The same applies to the method for moisture for cassava flour, whose scope does not include this matrix. In this sense, we understand that it should also be retained as type IV. The Kjeldahl method does not quote the value of the N factor. To facilitate the application by laboratories, we suggest keeping the N factor in parentheses for all Kjeldahl methods. The Procedural Manual states that preference should be given to set performance criteria according to the guidance established in the General Criteria for the Selection of Methods of Analysis using the Criteria Approach. Taking this into account, Brazil suggests establishing the criteria performance described in the table below for aflatoxins. The values proposed considered the maximum level of 15 µg/kg (CXS 193-1995) and the ratio of 70% for B1, 20% for B2, 5% for G1 and 5% for G2. CCCF can be consulted about this ratio. 								Brazil
Analyte	ML (mg/kg)	ML (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	Minimal applicable range (µg/kg)	Precision (%)	Recovery (%)	Recovery calculada por interpolação (%)
Aflatoxins, total	0,015	15	3	6	8,4 a 21,6	44	60-115	61,1 - 114,7
B1	0,0105	10,5	2,1	4,2	5,88 a 15,12	44	60-115	60,1 - 115,0
B2	0,003	3	0,6*	1,2*	1,68 a 4,32	44	40 - 120	44,4 - 118,9
G1	0,00075	0,75	0,15*	0,3*	0,42 a 1,08	44	40 - 120	40 - 120
G2	0,00075	0,75	0,15*	0,3*	0,42 a 1,08	44	40 - 120	40 - 120
* If those values could not be validated, LOD and LOQ for AFB ₂ , AFG ₁ and AFG ₂ could be up to parameters for AFB ₁ .								
Egypt appreciates the work done in the document & suggests endorsement of methods in Appendix I of CX/MAS 23/42/5								Egypt
Agree with no comments								Iraq
De acuerdo con el documento								Paraguay

The Philippines expresses appreciation for the work done by the eWG chaired by Canada to continue the review of all methods related to cereals, pulses and legumes workable package.	Philippines
SPECIFIC COMMENTS	
a) whether the methods in Appendix I of CX/MAS 23/42/5 can be endorsed	
<p>We wouldn't support the 'Block' endorsement of CX/MAS 23/42/5 Appendix I, however could support individual Appendix I line items on supply of further information and hopefully concise reasons being provided for new method amendments. As we see in the CPL eWG review process a significant number of new method proposals in Appendix I, whereas in the Dairy and F&O workable package reviews any new methods were segregated and received specific attention or referred to the relevant commodity committee for support. Noting, in this case, the CCCPL is 'Adjourned sine die', so the updating of methods by CCMAS42 where 'necessary' may be appropriate.</p> <p>In some cases, the 'new' method amendments, while appearing appropriate, could result in significant changes for the commodity standard. Using the Commodity-provision combination of Certain pulses-Moisture: The Appendix I method proposal is ISO 665 ISO 24557 / AACC 44-17.01 where the strikethrough method scope is for Oilseeds, and the proposed new method amendments scope is moisture content of pulses and ISO 24557 which states it is based on AACC approved method 44-17, Moisture — Air-oven method (pulses). However, AUSTRALIA has concerns that the impact of the proposed 'new method' changes on the Standard CXS 171-1989 (2019) acceptance specification, as ISO 665 is drying at 103 °C ± 2 °C in an oven at atmospheric pressure, while ISO 24557 / AACC 44-17.01 is drying at 130 °C ± 1 °C.</p> <p>As another example, CCCPL recently (2018) engaged with an eWG for Standard for Quinoa which led to the submission to CCMAS39. While in Appendix I, the methods endorsed at CCMAS39 (see REP18/MAS para. 15-16) for Moisture and Protein are being replaced with 'new methods' and we wonder if these updates were 'necessary'. Noting, that at CCMAS39 plenary for Moisture content in Quinoa, AACCI requested the late inclusion of method AACCI 44-15.02 as equivalent to ISO 712. Further, it was AUSTRALIA's belief that CCMAS39 agreed to endorse the general method ISO 1871 as Type IV for the determination of protein content, noting that under a project undertaken in several Andean countries, the method had been validated for protein determination in quinoa. The Committee had agreed that the method could be re-typed as Type I, pending submission of the validation data in quinoa to CCMAS40. The CCMAS40 submission does not appear to have occurred, but if ISO 1871 is validated in Quinoa, this should supersede any ISO 20483 proposal. So, it would be good to have a concise reason identified for any proposed changes.</p> <p>As will be detailed later, the reduction in method information such as Gravimetric drying temperature, and Nitrogen factors, in our opinion is not appropriate if CXS-234 is to be considered the single reference to methods of analysis and sampling for food, plus maintain format consistency with previous workable package reviews.</p> <p>For Particle size (granularity) the method 'AOAC 965.22 and ISO 3310-1' where the complementary methods proposal is based on information provided by the standard CXS 173-1989 (Type I method with sieve specifications as in ISO 3310/I – 1982 Test sieves). We believe the ISO 3310-1 is for assessing the "dimensional accuracy of the test sieve openings" i.e. equipment calibration, not specifically part of the "method required to determine/calculate the required answer". So, in the above case while 'sieve specifications as in ISO 3310/I' may be included as a footnote to reflect CXS 173, we do not believe it fits the definition of a complementary method to AOAC 965.22.</p> <p>For the Pearl millet flour and Sorghum flour – colour, according to the review that "Colour-grading equipment used in method is no longer available", it would appear this proposal or lack there-of, should mean these line items be moved from "Group 1. Methods reviewed by CPL EWG with decisions" to "Group 2. Methods requiring additional follow up action"</p>	Australia
The EUMS agree in principle with the proposed amendments listed in Appendix I, in particular the proposed shift of N-to-protein conversion factors to the relevant commodity standards.	European Union

<p>Appendix 1. Group 1. Durum wheat semolina and durum wheat flour - Moisture :</p> <p>Jamaica endorses the methods ISO 712 and ICC 110/1 as they are fit-for-purpose. The methods are equivalent.</p> <p>Appendix 1. Group 1. Peanuts (intended for further processing) - Aflatoxins, total, IAC (Holaday-Velasco minicolumn):</p> <p>There is a concern about the recommended removal of the method AOAC 979.18. Please clarify how the method does not meet the the performance criteria in the Procedural Manual.</p>	Jamaica												
<p>The Philippines supports the proposed changes and additional information in Appendix 1. Most of the methods are being used in the analysis and in participation to proficiency testing.</p> <ul style="list-style-type: none"> • Indicate Type I in the moisture method under Gari commodity on page 12 as no type of method indicated. (Appendix 1) 	Philippines												
<p>For commodity Quinoa, provision Moisture, Cereals and Grains Association (formerly AACC) would like to make the following comment:</p> <p>The commodity standard, CXS 333-2019 (2020) defers method reference to CXS 234, which currently endorses AACC method 14-15.02. The eWG has recommended shifting endorsement to ISO 712. Further search has shown AACC 14-15.02 is identical to AOAC 925.10, as these two methods were adopted simultaneously in 1925.</p> <p>There are some minor differences between the methods as shown on the table:</p> <table border="1" data-bbox="120 730 1025 858"> <thead> <tr> <th>Method</th> <th>Temp</th> <th>Time</th> <th>Test portion</th> </tr> </thead> <tbody> <tr> <td>AACC/AOAC</td> <td>130 ± 1 °C</td> <td>1 hour</td> <td>2 – 3 g</td> </tr> <tr> <td>ISO</td> <td>130 – 133 °C</td> <td>2 hr cereals 90 min flours</td> <td>5 g</td> </tr> </tbody> </table> <p>The AACC/AOAC method has been used in the global grain/cereals trade for over 90 years. It was originally developed in 1925 for use on wheat flour. It is one of these methods that while limited validation data is available, the method has proven to be reliable and robust for use in trade. The ISO method does not have validation information specific to quinoa, but does for other grain matrices.</p> <p>Cereals and Grains Association manages ongoing proficiency test (PT) programs that measure moisture in cereal commodities, pulses, and legumes. In our PT programs for moisture, this method is used by approximately 60% of users, either as primary method for testing, or a calibration method for secondary methods, such as near-infrared reflectance spectroscopy (NIR). It is the most commonly used method in the grain industry.</p> <p>We feel that the differences between the two methods is minimal, given the large amount of variation in sampling and testing normally expected with these commodities. However, if the decision is they are not identical, then the preference should go to the AACC/AOAC method as this is the method preferred by the commodity committee when the CXS 333 was originally written, and has been used since then. To change the endorsement now would be disruptive to trade and existing contracts.</p> <p>For the Commodity Whole Maize (Corn) Meal, Cereals and Grains Association would like to make the following comment:</p> <p>Commodity Standard CXS 154-1985 references method AOAC 965 as Type I method with sieve sizes as given in the CXS, with the inclusion of ISO 3310-1 as a standard for the sieve geometry. In CXS 234, the only endorsement is for AOAC 965.</p> <p>Cereals and Grains Association has recommended in place of AOAC 965, the method AACC 66-20.01. The only difference between the two methods is test portion size of 100 g for AACC vs. 50 g for AOAC. Neither method has validation data specific to whole corn meal. For a commodity such as whole corn meal, we recommend a larger test portion size, as this will give more precise readings. We anticipate that on average, the methods will</p>	Method	Temp	Time	Test portion	AACC/AOAC	130 ± 1 °C	1 hour	2 – 3 g	ISO	130 – 133 °C	2 hr cereals 90 min flours	5 g	AACC International
Method	Temp	Time	Test portion										
AACC/AOAC	130 ± 1 °C	1 hour	2 – 3 g										
ISO	130 – 133 °C	2 hr cereals 90 min flours	5 g										

<p>give the same results, but individually, the 100 g test portion will more precise. In the absence of commodity-specific validation data, better to err on the side of larger test portion.</p>	
<p>b) “items for further consideration” (para. 21 of CX/MAS 23/42/5)</p>	
<p>“whether moisture methods should be prescribed in CXS 234”, as stated in the Australian response to CL 2022/44-MAS June 2022</p> <p>We believe the moisture methods should be prescribed for proximate analysis in accordance with ‘Codex Information Document: Guidance on Process for Submission, Consideration and Endorsement of Methods’.</p> <p>Rationale:</p> <ol style="list-style-type: none"> 1. a correction for moisture content is frequently required for reporting results of the proximate methods (i.e., ash, protein, and fat). 2. ‘Complementary’ methods are ‘Two or more methods required to determine/calculate the required answer’. A ‘moisture’ is required to determine/calculate the proximate provisions in the relevant Codex standard. 3. The CXS 234 workable package reviews were in part to implement this guidance for consistency and clarity within CXS 234. Implementing exceptions, will only negate the intent of the guidance. Thus, the footnote option provided is not a suitable option. 4. The protocol followed by IDF, ISO and AOAC in the revision of the dairy group workable package would be followed for the CPL package, we suggest the correction for ‘total solids content’ in dairy product provisions is an analogous correction for ‘moisture content’ in the cereals, pulses and legume proximate provisions with an acceptance specification on a ‘dry weight basis’ including ‘ash, protein and fat’. <p>An issue arises with Codex endorsed methods such as ISO 2171 Determination of ash yield by incineration, where ISO 2171 specifies Moisture methods (ISO 171 cereals other than maize, ISO 6540 maize, ISO 24557 pulses, plus ISO 12099 using NIR can be used) which causes some conflict or duplication with respect to commodity moisture determination for Ash. Leaving a Moisture method to the discretion of the analyst may be viewed as resolving this ‘conflict’ but in a dispute we doubt this would be the case, as does the analyst do a moisture as specified by Codex CXS 234 for the commodity itself, then another moisture e.g., ISO 12099 when undertaking a moisture correction for commodity provision Ash? Thus, a specification in Codex of how the proximate’s complementary moisture correction is determined and aligned with the commodity moisture provision provides a clearer and more definitive provision.</p> <p>With respect to:</p> <ul style="list-style-type: none"> · “how best to address provisions where methods could not be found to replace methods identified for replacement (Appendix II of CX/MAS 23/42/5)”, <ul style="list-style-type: none"> o Regarding the existing CAC/RM 55 endorsement, the Codex secretariate may be able to retrieve ‘CAC abolished CAC/RM’ for publication as CXS 234 Appendix. o With regard to replacement of Kjeldahl Nitrogen methods using mercury catalysts, we would expect they could be identified. o Particle size – by sieving, replacement methods we expect could be identified. 	<p>Australia</p>
<p>The EUMS propose to consider the following points in the discussions of the Physical Working Group:</p> <p>whether moisture methods should be prescribed in CXS 234, or whether identification of the requirement for use of a commodity-specific moisture method could be achieved using a footnote with an explanation to address the requirement. A proposed footnote was introduced into Appendix I.</p>	<p>European Union</p>

<ul style="list-style-type: none"> Most of the moisture methods needed for proximate analysis of commodities have the relevant commodity in their scope, viz. ISO 24557 / AACC 44-17.01, ICC 110/1, ISO 712, ISO 6540. AOAC 925.09 (vacuum drying), which can be regarded as a 'horizontal' method, is listed for commodities which are not included in the scope of the above mentioned standards. Listing moisture methods in CXS 234-1999 has the advantage of guiding analysts to the appropriate moisture methods for proximate analysis. Therefore, the EUMS do not favour the removal of the listed moisture methods for the cereals, pulses and legumes workable package. However, for those commodities that are not covered by the scope of the currently listed moisture methods, the concerned method, mostly AOAC 925.09, could be removed and the proposed footnote added to guide users. 	
<p>the inclusion of general guidance instead of a prescribed method, to allow for classification of products (Appendix II of CX/MAS 23/42/5).</p> <ul style="list-style-type: none"> For the provision 'Ash' in all commodities AOAC 923.03 / ISO 2171 was endorsed. AOAC 923.03 requires the use of a muffle furnace at 550 °C while ISO 2171 gives the analyst the option to incinerate the sample either at 900 °C in platinum dishes or at 550 °C in quartz or silica dishes. Therefore, the comment made by the EWG, questioning the use of expensive platinum dishes on grounds of applicability, availability and cost of methods, seems to be unfounded as both methods allow the use of widely available quartz or silica dishes. Furthermore, the EUMS notice that the principle of the 'ash' methods in the cereals, pulses and legumes workable package is described as 'incineration', which is the term used by the ISO 2171 standard as well. However, the Dairy package uses the term 'ashing' together with the specified temperature. Therefore, the EUMS recommend to agree on a harmonised terminology to describe the method principle for the provision 'Ash' throughout CXS 234-1999 (e.g., 'Ash', 'incineration at xxx °C). 	
<p>Moisture methods to be used in the analysis should correspond to matrices being tested, as listed in CXS 234. The required moisture methods to be used can be easily identified using the proposed footnote.</p> <ul style="list-style-type: none"> The Standard Development Organizations should develop and validate a method intended for the commodity following the Principles for the Establishment of Codex Methods of Analysis in the Procedural Manual. We support the inclusion of general guidance instead of prescribed method to allow classification of products (Appendix II). 	Philippines
c) consider whether methods listed in Appendix II and Appendix III of CX/MAS 23/42/5 should be reviewed at a future WG on methods endorsement.	
<ul style="list-style-type: none"> As stated in b) above we believe resolutions to replace methods identified in Appendix II of CX/MAS 23/42/5 can be identified. With respect to Appendix III SDO method nominations, we note these are all type I methods, where only one method can exist or multiples when nominated/endorsed methods are 'identical'. But we support the concept that initially an assessment by the nominee (confirmed by CCMAS) that the endorsed methods no longer meet the requirements of the associated commodity standard provision. Only then will methods proposed by SDOs as updates be considered 'as necessary' and forwarded to the relevant commodity committee for consideration, or in the relevant committee absence by CCMAS42. 	Australia
<p>Furthermore, the EUMS agrees to setup a WG to continue the work for methods listed in Appendix II and III.</p>	European Union
<p>The Philippines supports the review of methods listed in Appendix II and Appendix III at a future WG on methods endorsement. There are additional methods in Appendix III that have been reviewed and considered equivalent to the methods indicated in CXS 234.</p>	Philippines
Other comments	
<p>Editorial suggestions:</p>	European Union

<ul style="list-style-type: none"> • The Dairy package mentions the drying temperature for moisture methods; for consistency this approach should be applied for the cereals, pulses and legumes workable package as well (e.g., 'Gravimetry ([oven] drying at xxx °C or vacuum [oven] drying at xxx °C). • For 'Peanuts (raw)' the EWG suggests to change the provision 'Aflatoxins, total' to 'Aflatoxins, total as sum of aflatoxins B1, B2, G1 and G2); but for 'Peanuts' the provision 'sum of aflatoxins B1, B2, G1 and G2' remains unchanged. It is recommended to align the language. • For the provision 'Fibre, crude' the method principle is sometimes described as 'Gravimetry (extraction and filtration)' for Perl millet flour, but also as 'Gravimetry (separation, incineration)', Sorghum flour, 'Gravimetry (Ceramic filter filtration)' for Soy protein products, 'ceramic fibre filtration' for Wheat protein products including wheat gluten, 'Gravimetry (separation)' for Gari, and 'Gravimetry (incineration)' for Edible Cassava flour, which is confusing. Even if the applied methods differ in detail how the non-fibre part is removed, the determination of crude fibre rests on the gravimetric determination of the insoluble residue after drying and the gravimetric determination of the ash content of the insoluble residue. Therefore, the EUMS suggest to use 'Gravimetry (oven drying and incineration)' as method principle for all crude fibre methods in the cereals, pulses and legumes workable package. 	
<p>¹A correction for moisture content is frequently required for reporting results of the proximate methods (i.e., ash, protein and fat). <u>Moisture content is the amount of water present in a sample, and it can have a significant impact on the results of the proximate methods. Therefore, it is important to correct for the moisture content of a sample when reporting the results of proximate analysis.</u> No moisture methods have been identified to correspond with the proximate methods in the current version of CXS 234. Moisture methods should correspond to those endorsed for the matrices being tested.</p>	Singapore