

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
United Nations



World Health
Organization

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Agenda Item 2, 3 and 4

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

42nd Session
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REPORT OF PHYSICAL WORKING GROUP ON ENDORSEMENT

Agenda Item 2 - Matters Referred to the Committee by the Codex Alimentarius Commission and Other Subsidiary Bodies (CX/MAS 21/42/2 and CX/MAS 21/42/2 Add.1)

MATTERS ARISING FROM OTHER SUBSIDIARY BODIES

MATTERS FOR ACTION OR INFORMATION

Codex Committee on Contaminants in Foods (CCCF15, 2022)

Methylmercury in fish: Sampling plans

The PWG noted the CCCF agreement to consider CCMAS recommendation on sampling plans at CCCF17 (2024).

Review of methods of analysis for contaminants

The PWG reviewed the performance criteria for lead and cadmium referred by CCCF (CCCF REP22/CF15, Appendix VIII: Part I) for inclusion in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999).

After some discussion about the appropriate number of digits after the decimal place, the PWG agreed to make minor changes to the numeric criteria referred by CCCF (Appendix I, Table I). This agreement to advance numeric criteria resulted in the need to remove the methods (Appendix I, Table II) from CXS 234.

The PWG was not able to review and determine if the methods (REP22/CF15, Appendix VIII, Part II) were appropriate as examples of available methods that meet the criteria. Additionally, other methods were not submitted as examples of available methods that meet the criteria.

The PWG recommended

Endorsement of the numeric criteria (Appendix I, Table I)

Removal of the methods from CXS 234 (Appendix I, Table III)

Further work to review the methods (Appendix I, Table II) and other methods to identify “examples of available methods that meet the criteria”.

Codex Committee on Food Hygiene (CCFH53, 2022)

Review of the methods of analysis for irradiated foods in the General Standard for Methods for the Detection of Irradiated Foods (CXS 231-2001) and their incorporation into the Recommended Methods of Analysis and Sampling (CXS 234-1999)

The PWG considered the list of methods from CXS 230 with the additional text further describing the commodity and precision (Appendix II). The PWG noted that there was little information on the methods

and how they are applied to the provisions. It was noted that while specific method steps are not required, some information about the methods would be useful in understanding the typing and the appropriateness to the specific matrices.

The PWG recommended

The methods listed in CXS 230 (Appendix II) not be endorsed at this time

Additional information on the methods be gathered and the information and methods be submitted for additional review at CCMAS43.

Codex Committee on Food Additives (CCFA53, 2023)

Testing methods related to nitrates and nitrites

The PWG considered the request by CCFA to (i) establish criteria for the detection of nitrate and nitrite ions in a variety of food matrices (CX/FA 21/52/7 Appendix 5, Annex 2), and (ii) provide information on available methods for detection (CX/FA 21/52/7 Appendix 5, Annex 1).

Based on the amount of information needed to fulfill CCFA request the PWG recommended:

The establishment of an electronic Working Groups (eWG) chaired by USA, working in English to assist in the response to the request from CCFA (CX/MA 23/42/2 para 31, to create a report for consideration at CCMAS43 which will address the following items:

- establish numeric performance criteria for the detection of nitrate and nitrite ions in the food matrices listed in CX/FA 21/52/7 Appendix 5, Annex 2.
- review the methods in CX/FA 21/52/7 Appendix 5, Annex 1 and determine if these methods meet the numeric performance criteria establish for the matrices in CX/FA 21/52/7 Appendix 5, Annex 2
- discuss if the methods detect both nitrate and nitrite ions and if so, whether the method detects each ion separately or only in combination
- discuss if the different detection schemes (i.e., separate or combined) could have an impact on the precision and accuracy of the methods

Codex Committee on Food Labelling (CCFL47, 2023)

Food allergen labelling – precautionary allergen labelling

The Committee noted the request from CCFL (CX/MAS 23/42/2 Add. 1) to recommend suitable analytical methods and guidance on their validation and applications including sampling plans for determining allergenic proteins in foods. CCMAS noted that the detection and quantification of allergenic proteins in foods is an important but challenging food safety area and that CCMAS was not in position to answer the request from CCFL during the physical working group on method endorsement (PWG). CCMAS would like to support CCFL efforts to revise the *General Standard for the Labelling of Pre-packaged Foods* (CXS 1-198) and the developing guidance on precautionary allergen labelling.

The PWG recommended

The establishment of an electronic working, chaired by the United States and Co-Chaired by the United Kingdom to develop a discussion paper which would discuss best practices for the selection of validated analytical methods, and for the validation of such methods. The discussion paper should consider the following for the allergens listed in Table 11 of FAO/WHO report "*Risk Assessment of Food Allergens Part 2: Review and Establish Threshold Levels in Foods for the Priority Allergens*":

- define standardized and harmonized terminology and definitions for allergen testing methods
- currently available test methods and validation status for the priority allergenic proteins listed in CX/FL 23/47/5 appendix I and noting the validated scope (food matrices, processed food) of these method

- required information for method evaluation and validation, including antibodies used (if ELISA), cross-reactivity, assay applicability, selectivity, stability (ruggedness), calibration procedures, sensitivity, range of quantification, LOD/LOQ, accuracy/trueness, extraction efficiency, precision, robustness, applicability, recovery and practicability, and whether it reports total protein. Validation requirements for the testing of allergenic proteins in foods including accuracy/trueness, extraction efficiency, precision, robustness, applicability, recovery and practicability.
- confirmatory methods for cases of potential analytical cross reactivity, and examples of such possibly including second ELISA confirmation, DNA based detection, and/or mass spectrometry techniques
- reference to other 'best practice' guidance documents, including SDO validation procedures and relevant Codex texts

The EWG will not address the second question from CCFL on sampling plans as sampling plans are covered by Revision of the General Guidelines on Sampling (CXG 50-2004).

Agenda Item 3 - ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PLANS FOR PROVISIONS IN CODEX STANDARDS (CX/MAS 21/42/3 and CX/MAS 21/42/3 Add.1)

CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU43, 2023)

Methods of analysis for provisions in the Standard for Follow-Up Formula (CXS 156-1987)

Methods for vitamin B12; total amino acids (excluding taurine and tryptophan), and tryptophan in the Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants (CXS 72-1981)

The PWG reviewed the methods referred by CCNFSDU (Appendix III)

The PWG recommends

endorsement of AOAC 2014.02 for vitamin B12 as a type III, and

endorsement of AOAC 2018.06 / ISO 4214 | IDF 254 /AACC 07-50.01 for total amino acids (excluding taurine and tryptophan) as a Type II, and

endorsement of AOAC 2017.03 the for Tryptophan as a Type II.

CODEX COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH¹, CCSCH⁶)

Methods of analysis for provisions in the Standard for Dried Roots, Rhizomes and Bulbs – Dried or Dehydrated ginger (CXS 343-2021), Standard for Dried Floral Parts-Cloves (CXS 344-2021), Standard for Dried Leaves - Dried Basil (CXS 345-2021), Standard for Dried Floral Parts – Saffron (CXS 351-2021), Standards for Dried Seeds – Nutmeg (CXS 352-2022), Standard for Dried or Dehydrated Chilli Pepper and Paprika (CXS 353-2022), draft Standard for Dried Small Cardamom, draft Standard for Spices Derived from Dried Fruits and Berries (Part A - Allspice, Juniper berry and Star anise)

The PWG reviewed the methods referred by CCSCH (Appendix IV) and had questions about the choice list of provisions, the choice of methods and the typing.

The PWG recommends

not endorsing the methods

returning the following questions to CCSCH

¹ REP21/SCH05 paras 36, 65, 81, 115, 149 and Appendices II, III, IV, V, VI

² REP22/SCH06 paras 39, 59, 80, 107, 121(i) and Appendices III, IV, V, VI, VII Part A

CCMAS has noted that the same method (e.g. ISO 927) will have different typing in different tables, was there a compelling reason for this change in typing?

CCMAS noted that 2 different methods will be used in conjunction, one as type I and one as type IV (e.g. ISO 927 and MPM V8) is there a reason for this mixing of Type I and Type IV methods?

Different methods are used across different commodities that appear to have similar characteristics, are there compelling reasons for these changes?

May standards appear to contain redundant provisions (e.g. insects, excreta) is this intentional and should the methods be consistent?

FAO/WHO COORDINATING COMMITTEE FOR AFRICA (CCAFRICA24)³

Methods of analysis for provisions in the Standard for Dried Meat (CXS 350-2022)

The PWG recommended

Endorsement of the methods and at the Type indicated (Appendix V).

The sampling plan not be endorsed and a new plan developed once the changes to GL 50 are complete.

Refer a question to CCAFRICA regarding the removal AOAC 935.47 and 937.09b and if this removal was deliberate or if the methods should have been retained as Type III?

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA22)⁴

Methods of analysis and sampling for provisions in the draft Regional Standard for Soybean Products Fermented with Bacillus Species,

The PWG recommended

Endorsement of the methods as shown Appendix VI Table I.

Regional Standard for Cooked Rice Wrapped in Plant Leaves

The PWG discussed the extraction procedure and the methods of analysis methods ISO 3960 or AOCS Cd 8b-90 and confirmed the methods are identical. There was no validation data establishing the performance (e.g., recovery, precision) of the rice extraction step.

SDO expressed concern in the efficiency of the extraction process given the long conditions and the application of the peroxide value determination to this matrix. Given these concerns, the PWG recommended

The method be endorsed as a Type IV (Appendix VI Table II)

That CCMAS suggest the validation of the method should be undertaken expeditiously and the data presented to CCMAS for review.

FAO/WHO COORDINATING COMMITTEE FOR NORTH AMERICA AND SOUTHWEST PACIFIC (CCNASWP16)⁵

Methods of analysis for provisions in the draft Regional Standard for Fermented Noni Fruit Juice

The PWG reviewed the method validation data for scopoletin and deacetylasperulosidic acid and determined they met the requirements for a successful validation. The PWG also reviewed the updated method. The PWG also noted that the methods for pH and ethanol has previously been endorsed. Finally the PWG noted that a validation of an IFU method for Brix value (soluble solids) was still underway

The PWG recommended

³ REP22/AFRICA24, para. 40 (i) and Appendix III V

⁴ REP23/ASIA22, para. 50 (ii) 83 (ii) and Appendix V, VII

⁵ REP23/NASWP16, para. 28 (iii) 73 (i) and Appendix III, VII Part A

Endorsement of the methods for scopoletin as a Type IV

Endorsement of the method for deacetylasperulosidic acid as a Type IV

Wait for the completion of the IFU led study before endorsing a method for Brix value (soluble solids)

Revised SOP for the Identification of Kavalactones and Flavokavains in Fresh and Dried Kava Products by HPTLC in the Regional Standard for Kava Products for Use as a Beverage when Mixed with Water (CXS 336R-2020)

The PWG reviewed the revised SOP and noted some deficiencies in the description of how the final determination should be made.

The PWG recommended

Not endorsing the SOP and asking for further edits to address the lack of instruction on the final determination steps,

CODEX COMMITTEE ON CONTAMINANTS IN FOODS (CCCF16)

Sampling Plans Provisions in the General Standard for Contaminants in Food and Feed (CXS 193-1995)

The PWG reviewed the sampling plans for aflatoxin and the numeric performance Criteria. It was noted that the sampling plan was consistent with the plans established for other mycotoxins.

The PWG recommended;

Endorsement of the sampling plan

Clarification to CCCF, that upon the completion off the revisions of the General Guidelines on Sampling, the plan should be evaluated to determine if it was still within the guidelines.

In general, the PWG agreed with the sum of components approach, but it was noted that the use of a footnote in the table referred by CCCF, led to the creation of multiple numeric criteria for each commodity. In an attempt to remedy that situation and develop a single set of criteria for each commodity some edits were made to the table provided by CCCF. A number of interested delegations have reviewed the table, but the PWG has not. The table is presented in Appendix VIII for review and potential endorsement by the committee.

Additionally, if the numeric criteria are endorsed, it has been recommended by some delegations that there be a change to the Information Document on Sum of Components, in order to reflect this new example. The PWG was generally in favor of the idea, but has not reviewed the new text.

The PWG recommends

Review of the numeric criteria table in Appendix VIII

Review by the committee of the following text and consideration to add it to the end of the Information Document on Sum of Components if the table in Appendix VIII is endorsed.

*“If the components included in the ML definition are not present in constant ratios and where the inclusion of weighting factors of the individual components results in LOD/LOQ values or minimum applicable range that cannot be validated, ML/n should be used to determine the criterion for LOD. (e.g. $1/5*ML/n$) and for LOQ (e.g. $2/5*ML/n$) or for the minimum applicable range (e.g. $ML/n \pm 2s_R$), with n being the number of components included in the ML definition.”*

URUGUAY, ARGENTINA, AND BRAZIL

The PWG reviewed the method (Annex 2) and method data presented by Uruguay, Argentina and Brazil and considered the endorsement of the method as a Type I and replacement of method ISO 5537 | IDF 26. Delegations supported the endorsement citing concerns about the cost and availability of equipment for ISO 5527 | IDF 26. Other delegations did not support endorsement as a Type I, noting that ISO 5537 | IDF

26 was developed after concerns about and poor performance of method IDF 26A, which used a identical drying temperature at Annex 2.

After extensive discussion a consensus on the endorsement of Annex 2 as Type I method could not be reached in the PWG.

Determination of lactose and fat content in the Recommended Methods of Analysis and Sampling (CXS 234-1999)

The PWG considered the new scope of ISO 22662 | IDF 198 which has been revised to broaden the scope of matrices to include dairy permeate liquid and dairy permeate powder in light of the recently published Codex standard on Dairy Permeates (CXS 331). A new entry line for CXS 234 is therefore proposed.

The PWG recommended

Endorsement of ISO 22662 | IDF 198 as Type II method for the determination of lactose in dairy permeate powders.

The PWG considered the information from ISO and IDF that standards for the determination of fat in various dairy matrices have been reorganized and revised. The revision of 10 IDF/ISO standards for various dairy matrices led to the combination into 2 standards. The first of them is focused on matrices requiring the use of the Schmid-Bondzynski-Raztlaff principle, while the second one is based on the Röse-Gottlieb principle. The merger of these standards into 2 aims at a full editorial alignment in the description for the concerned matrices and facilitating their utilization by the users of the standards.

The PWG recommended

Endorsement/approval of the change to CXS 234 to reflect the new ISO, IDF standard (Appendix IX).

CODEX OBSERVERS

Dietary Fibre Provisions in the Recommended Methods of Analysis and Sampling (CXS 234-1999)

The PWG did not consider the dietary fiber method, as the Observer decided to withdraw the submission and present to the CCNFSDU for review and potential referral to CCMAS43.

REVIEW OF METHODS OF ANALYSIS IN CXS 234 FATS AND OILS WORKABLE PACKAGE

The PWG considered the work and comments of the electronic working group on the Fats and Oils workable package (Appendix X).

The PWG recommends

Endorsement of the table (appendix X) including the footnote related to AOCS Cc 12-59 and its listing as a Type IV, even when a Type I exists for the same commodity and provision,

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

The PWG considered the work and comments of the electronic working group on the Cereal Pulses and Legumes workable package (Appendix XI, Group 1, Group 2, Group 3).

Review of the work left the PWG with 2 groups of methods. Group 1 first where final recommendations have been made. Group 2 which require additional follow-up and review. There are also a 3 group of methods which were not reviewed by the PWG. These are methods which have been recommended for consideration for endorsement. These methods maybe suitable replacements for method currently in CXS 234, but as was the procedure with the Dairy Workable Package, these will need to be submitted to the endorsement working group at a future CCMAS.

The PWG recommends

Endorsement of the methods in Group 1

Further review of the methods in Group 2, either through a re-established EWG or through PWG at CCMAS43

Communication to interested parties that the method submission process, including the method template should be used to submit a method for review and potential endorsement by the working group on endorsement.

REVIEW OF METHODS OF ANALYSIS IN CXS 234 PROCESSED FRUIT AND VEGETABLE WORKABLE PACKAGE

The PWG considered the work and comments of the electronic working group on the Processed Fruit and Vegetable workable package (Appendix XII, Group 1, Group 2).

Group I contains the results of the EWG and revisions endorsed by the PWG.

- a. There are several provisions for which numeric performance criteria can be created, and this was recommended by some member countries. These criteria can be developed over the next year to be considered at a future CCMAS. In the meantime, the revised entries should stay in CXS 234.
- b. Some CAC/RM methods and some methods included in the related commodity standards are listed in the revised table. These CAC/RM methods and methods in commodity standards are to be moved into CXS 234.

Group 2 II contains two items for further consideration:

- c. The provision for tin (Sn), which has a ML of 250 mg/kg (CXS 193) and applies to Canned foods (other than beverages). The PWG agreed to keep the entry as is for the time being and develop numeric performance criteria to be endorsed at a future CCMAS, as well as considered the commodity name since there is a different ML for canned beverages which may be confusing.

The method for mineral oil in raisins (CAC/RM 51) cannot be found in the *STANDARD FOR RAISINS CXS 67-1981**. Once that method is found, it should be moved into CXS 234

The PWG recommended

Endorsement of Group I methods and movement of CAC/RM standards to CXS 234

Consideration and development of numeric criteria for provisions in Group 1 methods

Development of numeric criteria for Tin

Other items for consideration

There were a number of overarching issues which arose during the EWG on the workable packages and in the PWG discussion. Although the PWG did not recommend any specific action, it was agreed that the topics would be presented to the Committee for consider on future work by CCMAS. These items included:

1. A discussion and decision on the names and format used for the principles identified in CXS 234. The same principle is often identified in different ways, and there is not consistency in what information should be captured in the principle. For example, if the specific detector used should be identified.
2. The incorporation and placement of nitrogen conversion factors in CXS 234. It is agreed that CCMAS should not set the conversion factor, but there are different opinions on if the conversation factor should appear in CXS 234 or remain solely in the commodity standard.
3. Equivalency of Type I methods.
4. The listing of Type IV methods in CXS 234, when a Type I method is listed for the same commodity and provision. At CCMAS42 we have taken the approach that to have both a Type I and Type IV, there should be a justifiable and motivating reason. If this approach is supported by the committee, changes should be made to the CCMAS information document to describe this

situation. If there is a decision to not have Type I and Type IV for the same commodity and provision, changes to the Procedural Manual and CCMAS information document should be made.

Appendix I

Table I: Numeric performance criteria for lead and cadmium for endorsement and inclusion in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999)

Numeric performance criteria for lead and cadmium in foods

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of Detection (LOD) (mg/kg)	Limit of Quantification (LOQ) (mg/kg)	Precision (RSD _R) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Natural mineral waters	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%		
Infant formula, formula for special medical purposes intended for infants and follow-up formula	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%		
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%		
Secondary milk products	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%		
Fruit juices, except juices exclusively from berries and other small fruits	lead	0.03	0.017 - 0.043	0.006	0.012	44	60-115%		
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		

Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Edible fats and oils	lead	0.08	0.045 - 0.115	0.016	0.032	44	60-115%		
Berries and other small fruits, except cranberry, currant, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Bulb vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Canned fruits	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Canned vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Fruits, except cranberry, currants, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Legume vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Meat and fat of poultry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		

Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Fortified / Liqueur wine from grapes harvested after 2019	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Pig, edible offal of	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Cattle, edible offal of	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cranberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Currants	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Elderberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Wine (wine and fortified / liqueur wine) made from grapes harvested before July 2019	lead	0.2	0.08 - 0.32	0.02	0.040	41	80-110%		
Fish	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Fresh farmed mushrooms (common mushrooms (<i>Agaricus bisporous</i>), shiitake mushrooms (<i>Lentinula edodes</i>), and oyster mushrooms (<i>Pleurotus ostreatus</i>))	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		

Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Table olives	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%		
Natural mineral waters	cadmium	0.003	0.0017 - 0.0043	0.0006	0.0012	44	40-120%		
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Bulb vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Legume vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		

Chocolate containing or declaring < 30% total cocoa solids on a dry matter basis	cadmium	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%		
Chocolate containing or declaring \geq 30% to <50% total cocoa solids on a dry matter basis	cadmium	0.7	0.35 - 1.05	0.07	0.14	34	80-110%		
Chocolate containing or declaring \geq 50% to < 70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table chocolate	cadmium	0.8	0.40 - 1.20	0.08	0.16	33	80-110%		
Chocolate containing or declaring \geq 70% total cocoa solids on a dry matter basis, including sweet chocolate, Gianduja chocolate, semi – bitter table chocolate, Vermicelli chocolate / chocolate flakes, and bitter table	cadmium	0.9	0.46 - 1.34	0.09	0.18	33	80-110%		
Cephalopods	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%		
Marine bivalve mollusks (clams, cockles and	cadmium	2	1.1 - 2.9	0.2	0.4	29	80-110%		

mussels), except oysters and scallops									
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Appendix I**Table II: Removal of analytical methods for lead from CXS 234 and transfer to the column of “example of applicable methods that meet the criteria”, if they meet the performance criteria**

Commodity	Provision	Method	Principle	Type
Fats and Oils and Related Products				
Fats and Oils (all)	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Named Vegetable Oils	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Olive Oils and Olive Pomace Oils	Lead	AOAC 994.02 or ISO 12193 or AOCS Ca 18c-91	AAS	II
Butter	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	NMKL 139 (Codex general method) AOAC 999.11	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	IV
Edible casein products	Lead	ISO/TS 6733 IDF/RM 133	Spectrophotometry (1,5-diphenylthiocarbazone)	IV
Processed Fruits and Vegetables				
Table olives	Lead	AOAC 999.11 NMKL 139 (Codex general method)	AAS (Flame absorption)	II
Miscellaneous Products				
Food grade salt	Lead	EuSalt/AS 015	ICP-OES	III
Food grade salt	Lead	EuSalt/AS 013	Atomic absorption spectrophotometry	IV

Appendix II

New texts added are shown in **bold/underlined** font. Texts proposed for deletion are shown in ~~strikethrough~~.

Commodity	Provision	Method	Principle	Type
Food containing fat (<u>e.g., raw meat and chicken, cheese, fruits</u>)	Detection of irradiated food - <u>Detection of radiation-induced hydrocarbons</u>	EN 1784 : 1996	Gas chromatographic analysis of hydrocarbons	Type II
Food containing fat (<u>e.g., raw meat and chicken, liquid whole egg</u>)	Detection of irradiated food - <u>Detection of radiation-induced 2-alkylcyclobutanones</u>	EN 1785 ¹ : 1996	Gas chromatographic/mass spectrometric analysis of 2-alkylcyclobutanones	Type III
Food containing bone	Detection of irradiated food - <u>Radiation induced Electron Spin Resonance (ESR) signal attributed to hydroxyapatite (principal component of bones)</u>	EN 1786: 1996	ESR spectroscopy	Type II
Food containing cellulose (<u>e.g., nuts and spices</u>)	Detection of irradiated food - <u>Radiation induced Electron Spin Resonance (ESR) signal attributed to crystalline cellulose</u>	EN 1787: 2000	ESR spectroscopy	Type II
Food containing silicate minerals (<u>e.g., herbs, spices, their mixtures and shrimps</u>)	Detection of irradiated food - <u>Thermoluminescence glow ratio used to indicate the irradiation treatment of the food</u>	EN 1788: 2004	Thermoluminescence	Type II
Food containing silicate minerals (<u>e.g., shellfish, herbs, spices, seasonings</u>)	Detection of irradiated food - <u>Measurement of photostimulated luminescence intensity</u>	EN 13751 ² : 2002	Photostimulated luminescence	Type III
Food containing crystalline sugar (<u>e.g., dried fruits and raisins</u>)	Detection of irradiated food - <u>Radiation induced Electron Spin Resonance (ESR) signal attributed to crystalline sugar</u>	EN 13708: 2004	ESR spectroscopy	Type II

Commodity	Provision	Method	Principle	Type
Herbs <u>and</u> spices and raw minced meat ³	Detection of irradiated food - <u>Difference between total microorganism count and viable microorganism count</u>	EN 13783:2004 NMKL 231 (2002)	Direct Epifluorescent Filter Technique/Aerobic Plate Count (DEFT/APC) (screening method)	Type III
Food containing DNA (e.g., food products, both of animal and plant origin such as various meats, seeds, dried fruits and spices)	Detection of irradiated food - <u>Detection of DNA fragmentation presumptive to irradiation treatment.</u>	EN 13784:2004	DNA comet assay (screening method)	Type III

Notes

¹ One Member noted that 2-alkylcyclobutanone was also present in some non-irradiated foods and hence EN1785 may need further consideration as a method for detection of irradiated foods.

² Consideration should be given to whether EN13751 should be specified as a screening method.

³ No information was found on validation of the method for this commodity.

Appendix III**CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU43)**

Methods of analysis for provisions in the Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants (CXS 72-1981)

Commodity	Provision	Method	Principle	Type
Infant Formula	<u>Vitamin B12</u>	<u>AOAC 2014.02</u>	<u>LC-UV</u>	<u>III</u>
	<u>Total amino acids (excluding taurine and tryptophan)</u> <u>For use according to Section 3.1.3 (a) footnotes 3 and 4 of CXS 72-1981</u>	<u>AOAC 2018.06 / ISO 4214 </u> <u>IDF 254 /AACC 07-50.01</u>	<u>UHPLC-UV</u>	<u>II</u>
	<u>Tryptophan</u> <u>For use according to Section 3.1.3 (a) footnotes 3 and 4 of CXS 72-1981</u>	<u>AOAC 2017.03</u>	<u>HPLC</u>	<u>II</u>

Appendix IV

CODEX COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH5)**Methods of analysis for provisions in the Standard for Dried Roots, Rhizomes and Bulbs – Dried or Dehydrated Ginger (CXS 343-2021)**

Parameter	Method	Principle	Type ¹
Moisture	ISO 939	Distillation	I
Total Ash on (dry basis)	ISO 939 and ISO 928	Calculation from moisture and ash Distillation and Gravimetry	I
Acid Insoluble Ash on (dry basis)	ISO 939 and ISO 930	Calculation from moisture and ash Distillation and Gravimetry	I
Volatile Oil on (dry basis)	ISO 939 and ISO 6571	Calculation from moisture and volatile oil Distillation and distillation	I
Extraneous Matter	ISO 927	Visual Examination followed by and Gravimetry	I
Foreign Matter	ISO 927	Visual Examination followed by and Gravimetry	I
Insect Damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices ISO 927	Visual Examination	IV
Whole dead insect	ISO 927	Visual examination and Gravimetry	I
Mammalian/ Other Excreta	MPM V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices (For whole) ISO 927	Visual Examination followed by and Gravimetry	IV
Mould visible	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices ISO 927	Visual examination	IV
Live Insect	ISO 927 AOAC 960.51	Visual Examination Visual Examination	IV IV
Calcium (as oxide) on (dry basis)	ISO 1003, Annex A	Chemical reaction followed by gravimetry	IV
SO ₂	AOAC 963.20	Colorimeter	II

¹ According to the definition of “types of method of analysis” as per Codex Procedural Manual Section II

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

Parameter	Method	Principle	Type ¹
Moisture	ASTA 2.0	Distillation	I
Volatile oil (dry basis)	<u>ASTA 2.0 and</u> ISO 6571	<u>Calculation from moisture and ash</u> Distillation and Distillation Volumetry	I
Total ash (dry basis)	<u>ASTA 2.0 and</u> ISO 928	<u>Calculation from moisture and ash</u> Distillation and Gravimetry	I
Acid Insoluble Ash (dry basis)	<u>ASTA 2.0 and</u> ISO 930	<u>Calculation from moisture and ash</u> Distillation and Gravimetry	I
Extraneous matter	ISO 927	Visual examination and Gravimetry	I
Foreign matter	ISO 927	Visual examination and Gravimetry	I
Insect damage	ISO 927 <u>Method V-8 Spices, Condiments, Flavours and Crude Drugs</u>	Visual Examination Visual Examination	IV IV
Insects/Excreta/Insect fragments	ISO 927	Visual Examination and Gravimetry	IV
Crude fibre	ISO 5498	Gravimetry	I
Mould visible	<u>Method V-8 Spices, Condiments, Flavours and Crude Drugs</u> ISO 927	Visual Examination	IV
<u>Mould visible</u>	<u>Method V-8 Spices, Condiments, Flavours and Crude Drugs</u>	<u>Visual Examination</u>	<u>IV</u>
Live insect	ISO 927	Visual Examination	IV
Mammalian or/and Other excreta	<u>Method V-8 Spices, Condiments, Flavours and Crude Drugs</u> ISO 927	Visual Examination	IV

¹ According to the definition of “types of method of analysis” as per Codex Procedural Manual Section II

*Latest edition or version of the approved method should be used

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

Parameter	Method	Principle	Type
Moisture	ISO 939	Distillation	I
Total Ash <u>(dry basis)</u>	<u>ISO 939 and</u> ISO 928	<u>Calculation from moisture and ash</u> <u>Distillation and Gravimetry</u>	I
Acid Insoluble Ash <u>(dry basis)</u>	ISO <u>928-939</u> and ISO 930	<u>Calculation from moisture and ash</u> <u>Distillation and Gravimetry</u>	I
Volatile Oil <u>(dry basis)</u>	<u>ISO 939 and</u> ISO 6571	<u>Calculation from moisture and volatile oil</u> Distillation <u>and Volumetry</u> <u>Distillation</u>	I
Extraneous Matter	ISO 927	Visual Examination <u>followed by and</u> <u>Volumetry</u> <u>Gravimetry</u>	I
Foreign Matter	ISO 927	Visual Examination <u>followed by and</u> <u>Volumetry</u> <u>Gravimetry</u>	I
Insect Damage	<u>Method V-8 Spices, Condiments, Flavours and Crude Drugs</u> <u>(Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)</u> <u>ISO 927</u>	Visual Examination	IV
Insects/Excreta/ Insect Fragments	<u>Method appropriate for particular spice from AOAC Chapter 16, subchapter 14</u> <u>ISO 927</u>	Visual Examination <u>and</u> <u>Gravimetry</u>	<u>IV¹</u>
Mould damage	<u>Method V-8 Spices, Condiments, Flavours and Crude Drugs</u> <u>(Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)</u> <u>ISO 927</u>	Visual examination (for whole)	<u>IV²</u>
Mammalian Excreta, And Other Excreta	<u>ISO Method V-8 Spices, Condiments, Flavours and Crude Drugs</u> <u>(Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39-B)</u> <u>927</u> (For whole)	Visual Examination <u>and</u> <u>Gravimetry</u>	I

* Latest edition or version of the approved method should be used.

² According to the definition of “types of method of analysis” as per Codex Procedural Manual Section II.

CODEX COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH6)**Methods of analysis for provisions in the Standard for Dried Floral Parts – Saffron (CXS 351-2021)**

Provision	Method	Principle	Type
Moisture	ISO 3632-2	Gravimetry	I
Total Ash <u>(dry basis)</u>	ISO 3632-2 and ISO 928	<u>Calculation from moisture and ash</u> Gravimetry <u>and Gravimetry</u>	I
Acid Insoluble Ash <u>(dry basis)</u>	ISO 3632-2 and ISO 930	<u>Calculation from moisture and ash</u> Gravimetry <u>and Gravimetry</u>	I
<u>Water soluble solids Soluble extract in cold water? (dry basis)</u>	ISO 3632-2 and ISO 941	<u>Calculation from moisture and ash</u> <u>Gravimetry and Gravimetry Extraction</u>	<u>IV?</u>
Taste strength (expressed as picrocrocin) $A_{1cm}^{1\%}$ 257 nm	ISO 3632-2	Absorbance	<u>IV?</u>
Aroma strength (expressed as safranal) $A_{1cm}^{1\%}$ 330 nm	ISO 3632-2	Absorbance	<u>IV?</u>
Coloring strength (expressed as crocin) $A_{1cm}^{1\%}$ 440 nm	ISO 3632-2	Absorbance	<u>IV?</u>
Extraneous Matter	ISO 3632-2	Visual Examination followed by Gravimetry	I
Foreign Matter	ISO 3632-2	Visual Examination followed by Gravimetry	I
Insect Damage	ISO 927	Visual Examination followed by Gravimetry	I
Whole dead Insects /Insect Fragments	ISO 927	Visual Examination <u>followed by Gravimetry</u>	I
Visible mould	<u>ISO 927 Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macro-analytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32</u>	Visual Examination <u>followed by and</u> Gravimetry	I
Mammalian Excreta	<u>Macro-analytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole) ISO 927</u>	Visual Examination <u>followed by and</u> Gravimetry	I
Other Excreta	AOAC 993.27 (For Ground)	Enzymatic Detection	IV

		Method	
Rodent filth	ISO 927	Visual Examination	I

¹ Latest edition or version of the approved method should be used

² The methods of analysis will be included in CXS 234-1999 after endorsement by CCMAS and the following text replace the Table

“For checking the compliance with this standard, the methods of analysis and sampling contained in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) relevant to the provisions in this standard, shall be used.”

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

For checking the compliance with this standard, the methods of analysis and sampling contained in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) relevant to the provisions in this standard, shall be used.

Provision	Method ¹	Principle	Type
Moisture content	ISO 939	Distillation	I
Total ash <u>(dry basis)</u>	ISO 939 and ISO 928	<u>Calculation from moisture and ash</u> Distillation <u>and</u> Gravimetry	I
Acid-insoluble ash <u>(dry basis)</u>	ISO 939 and ISO 930	<u>Calculation from moisture and ash</u> Distillation <u>and</u> Gravimetry	I
Water-insoluble ash <u>(dry basis)</u>	ISO 939 and ISO 929	<u>Calculation from moisture and ash</u> Distillation <u>and</u> Gravimetry	I
Volatile oil content <u>(dry basis)</u>	ISO 939 and ISO 6571	<u>Calculation from moisture and volatile oil</u> Distillation <u>and</u> Distillation	I
Extraneous matter	ISO 927	Visual examination followed by <u>and</u> <u>g</u> Gravimetry	I
Foreign matter	ISO 927	Visual examination followed by <u>and</u> <u>g</u> Gravimetry	I
Visible mould?	ISO 927	Visual examination and <u>followed by</u> <u>g</u> Gravimetry	I
Insect defiled/infested	MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V-32)ISO 927	Visual Examination followed by <u>and</u> <u>g</u> Gravimetry	I
Dead insect, insect fragments, rodent contamination	ISO 927	Visual examination	I
Live insect	ISO 927	Visual examination	I
Mammalian and or other excreta	ISO 927 Macroanalytical Procedure Manual (MPM) USFDA technical bulletin V.41	Visual examination followed by <u>and</u> <u>g</u> Gravimetry	I

Piece of mace	ISO 927	Visual examination followed by and gGravimetry	I
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The methods of analysis will be included in CXS 234-1999 after endorsement by CCMAS

For checking the compliance with this standard, the methods of analysis and sampling contained in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) relevant to the provisions in this standard, shall be used.

¹ Latest edition or version of the approved methods should be used.

Methods of analysis for the provisions off size, when sized and broken/damaged among the whole to be developed.

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

Provision	Method ¹	Principles	Type ²
Moisture	ISO 939	Distillation	I
Total Ash (<u>dry basis</u>)	ISO 939 and ISO 928	<u>Calculation from moisture and ash</u> Distillation <u>and</u> Gravimetry	I
Acid-insoluble ash (<u>dry basis</u>)	ISO 939 and ISO 930	<u>Calculation from moisture and ash</u> Distillation <u>and</u> Gravimetry	I
Pungency Scoville Heat units	ASTA 21.3	Chromatography	IV
<u>Pungency Scoville Heat units</u>	ISO 3513	Sensory evaluation	I
Colour value	ISO 7541	Spectrophotometry	<u>IV?</u>
Mammalian excreta	ISO 927	Visual examination <u>followed by</u> <u>and</u> Gravimetry (whole)	I
Mould damage	<u>MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V-32) ISO 927 (for whole)</u>	Visual Examination (<u>for whole</u>)	I
	AOAC 945.94 (<u>for Ground</u>)	Visual Examination (<u>for Ground</u>)	I
Insect Damage	<u>MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices herbs and botanicals (V-32) ISO 927</u>	Visual Examination <u>followed by</u> <u>and</u> Gravimetry	I
Extraneous matter ³	ISO 927	Visual Examination <u>followed by</u> <u>and</u> Gravimetry	I
Foreign matter ⁴	ISO 927	Visual Examination <u>followed by</u> <u>and</u> Gravimetry	I
Live insect	ISO 927 <u>/?</u> AOAC 960.51	Visual Examination	I
Insect filth	ISO 927	Visual Examination	I
Insect fragments	ISO 927	Visual examination counting	I
Rodent hair	AOAC 978.22 (Ground chilli) <u>AOAC 977.25 B (Ground paprika)</u>	Microscopic examination <u>Microscopic examination</u>	I †
<u>Rodent hair</u>	<u>AOAC 977.25 B (Ground paprika)</u>	<u>Microscopic examination</u>	‡

¹Latest edition or version of the approved method should be used.

²According to the definition of "types of method of analysis" as per Codex Procedural Manual Section II

³ Vegetative matter associated with the plant from which the product originates but not accepted as part of the final product.

⁴ Any visible/detectable objectionable foreign matter or material not usually associated with the natural components of the spice plant, such as sticks, stones, burlap bagging, metal, etc.

The methods of analysis will be included in CXS 234-1999 after endorsement by CCMAS and the following text shall replace the Table

"For checking the compliance with this standard, the methods of analysis and sampling contained in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) relevant to the provisions in this standard, shall be used

Methods of analysis for provisions in the draft Standard for Dried Small Cardamom

Provision	Method ¹	Principle	Type ²
Moisture	ISO 939	Distillation	I
Total Ash (<u>dry basis</u>)	ISO 939 and ISO 928	<u>Calculation from moisture and ash</u> Distillation and Gravimetry	I
Acid Insoluble Ash (<u>dry basis</u>)	ISO 939 and ISO 930	<u>Calculation from moisture and ash</u> Distillation and Gravimetry	I
Volatile Oil (<u>dry basis</u>)	ISO 939 and ISO 6571	<u>Calculation from moisture and volatile oil</u> Distillation followed by <u>Distillation Volumetry</u>	I
Extraneous Matter	ISO 927	Visual Examination <u>followed by and</u> Gravimetry	I
Foreign Matter	ISO 927	Visual Examination <u>followed by and</u> Gravimetry	I
Insect defiled/infested	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices <u>ISO 927</u>	Visual Examination followed by Gravimetry	I
Immature and shrivelled capsules	ISO 927	Visual Examination followed by Gravimetry	I
Mammalian or/and other excreta	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices <u>ISO 927</u>	Visual Examination followed by Gravimetry	I
Mould visible	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8. Spices <u>ISO 927</u>	Visual Examination followed by Gravimetry	I
Empty and malformed capsules	<u>IS_19074907:1984</u>	Visual Examination followed by Gravimetry	I
Whole insect Live/dead	ISO 927	Visual examination followed by Gravimetry	I

Light seeds	ISO 927	Visual examination followed by Gravimetry	I
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¹ Latest edition or version of the approved method should be used

² According to the definition of “types of method of analysis” as per Codex Procedural Manual Section II

* The methods of analysis will be included in CXS 234-1999 after endorsement by CCMAS and the following text replace the Table

“For checking the compliance with this standard, the methods of analysis and sampling contained in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) relevant to the provisions in this standard, shall be used”

Methods of analysis for provisions in the draft Standard for Spices Derived From Dried Fruits and Berries (Part A - Allspice, Juniper berry And Star anise)

Sl. No	Spices	Provision	Method ^{1,2}	Principles	Type
1	Dried Allspice Dried Juniper Berries Dried Star Anise	Moisture	ISO 939	Distillation	I
		Total ash (dry basis)	ISO 939 and ISO 928	<u>Calculation from moisture and ash</u>	I
		Acid- insoluble(dry basis)	ISO 939 and ISO 930	<u>Calculation from moisture and ash</u>	I
		Volatile oils(dry basis)	ISO 939 and ISO 6571	<u>Calculation from moisture and volatile oil</u>	I
		Extraneous matter	ISO 927	Visual examination followed by <u>and</u> gravimetry	I
		Foreign matter	ISO 927	Visual examination followed by <u>and</u> gravimetry	I
		Mould visible	ISO 927	Visual examination followed by <u>and</u> gravimetry	I
		Mammalian excreta	MPM V-8 Spices, Condiments, Flavors and Crude Drugs A. General methods for spices herbs and botanicals (V-32) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs ISO 927 (Applicable to whole form of the spices)	Visual examination followed by <u>and</u> gravimetry	I
		Whole dead insect	ISO 927	Visual examination	I?
			AOAC 969.44	Flotation method	IV?
		Insect fragments	ISO 927	Visual examination counting	I?
AOAC 975.49	Flotation method		IV?		

SI. No	Spices	Provision	Method ^{1,2}	Principles	Type
		Insect damage	<p>MPM V-8 Spices, Condiments, Flavours and Crude Drugs General methods for spices herbs and botanicals (V-32)ISO 927 (Applicable to whole form of the spices)</p>	Visual examination followed by and g Gravimetry or counting	I
		Mould damage	<p>MPM V-8 Spices, Condiments, Flavours and Crude Drugs General methods for spices herbs and botanicals (V-32)ISO 927 (Applicable to whole form of the spices)</p>	Visual examination followed by and g Gravimetry or counting	I
2	Allspice (whole, cracked/ pieces)	Filth (list all the filth here-for example - mammalian excreta?)	AOAC 965.40	Flotation	I
	Allspice (Ground/ powdered)	Light filth (list all the filth here-for example- mammalian excreta?)	AOAC 981.21	Flotation	I
3	Juniper Berries, Star Anise, (cut/broken, ground/ powdered)	Light filth (list all the filth here-for example- mammalian excreta)	AOAC 975.49?	Flotation	I

¹ Latest edition or version of the approved method should be used

² The methods of analysis will be included in CXS 234-1999 after endorsement by CCMAS and the following text replace the Table

“For checking the compliance with this standard, the methods of analysis and sampling contained in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) relevant to the provisions in this standard, shall be used.”

FAO/WHO COORDINATING COMMITTEE FOR AFRICA (CCAFRICA24)**Methods of analysis and sampling for provisions in the Regional Standard for Dried Meat (CXS 350-2022)****1. METHODS OF ANALYSIS AND SAMPLING****8.1 Methods of Analysis⁶**

Provision	Method	Principles	Type
Moisture Content	AOAC 950.46B	Gravimetry	I
Total Fat	ISO 1443	Gravimetry	I
Nitrogen Protein* *nitrogen-to-protein conversion factor = 6.25	ISO 937*	Calculation and Titrimetry	I
Chloride as Sodium Chloride (≥ 1.0%)	ISO 1841-1	Titrimetry (Volhard method)	III
Chloride as Sodium Chloride (≥ 0.25%)	ISO 1841-2	Titrimetry (Potentiometry)	II
Ash	ISO 936	Gravimetry	I
Water Activity	ISO 18787	Electrometry	II
*nitrogen-to-protein conversion factor = 6.25			

⁶ After adoption, the table containing the Methods of Analysis will be removed and replaced with the following Text, as per the requirements of the Procedural Manual:

“For checking the compliance with this standard, the methods of analysis and sampling contained in the Recommended Methods of Analysis and Sampling (CXS 234-1999) relevant to the provisions in this standard, shall be used.”

APPENDIX VI

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA22)**Methods of analysis and sampling for provisions in the draft Regional Standard for Soybean Products Fermented with *Bacillus Species***

Table I

Commodity	Provision	Method	Principles	Type
<u>Natto</u>	<u>Moisture</u>	<u>AOAC 925.09</u>	<u>Gravimetry</u>	!
<u>Natto</u>	<u>Protein</u>	<u>AOAC 988.05</u>	<u>Titrimetry, (Kjeldahl)</u>	!
<u>Natto</u>	<u>Lipid Content</u>	<u>AOAC 963.15</u>	<u>Gravimetry (Soxhlet)</u>	!
<u>Cheonggukjang</u>	<u>Moisture</u>	<u>AOAC 934.01</u>	<u>Gravimetry</u>	!
<u>Cheonggukjang</u>	<u>Protein</u>	<u>AOAC 988.05</u>	<u>Titrimetry, (Kjeldahl)</u>	!
<u>Cheonggukjang</u>	<u>Lipid Content</u>	<u>AOAC 963.15</u>	<u>Gravimetry (Soxhlet)</u>	!
<u>Thua Nao</u>	<u>Moisture</u>	<u>AOAC 925.09</u>	<u>Gravimetry</u>	!
<u>Thua Nao</u>	<u>Protein</u>	<u>AOAC 988.05</u>	<u>Titrimetry, (Kjeldahl)</u>	

Sampling plan not recommended for endorsement**Methods of analysis and sampling for provisions in the draft Regional Standard for Cooked Rice Wrapped in Plant Leaves****10. METHODS OF ANALYSIS AND SAMPLING⁷**

For checking the compliance with this standard, the methods of analysis and sampling contained in the *Recommended Methods of Analysis and Sampling* (CXS 234-1999) relevant to the provisions in this standard, shall be used.

10.1. Determination of Peroxide Value**10.1.1. Extraction of Oils from the Product****10.1.1.1. Apparatus**

- (a) Rotary evaporator
- (b) Water bath

10.1.1.2. Extraction

Remove the product package and plant leaves, etc., take out the edible part of the representative sample, crush it and put it in a homogenizer or glass mortar, and grind it continuously to make the sample fully mashed and mixed well, and then put it in the wide-mouth bottle, and add 2 to 3 times the sample volume of petroleum ether (boiling range: 30°C-60°C). After fully mixing, stopper the bottle and leave for more than 12 hours. Filter all the solution with a funnel filled with anhydrous sodium sulphate into a round-bottom flask. Rinse the residue in the wide-mouth bottle with petroleum ether. Repeat the filtration once with a new anhydrous sodium sulphate funnel, if the filtrate is not clear enough. Evaporate the petroleum ether in the round-bottom flask under reduced pressure on a rotary evaporator at below 40°C, and the residue is the test sample. A sufficient number of

⁷ The analytical methods will be removed when the standard is adopted by CAC and included in CXS 234-1999.

representative samples should be selected to ensure that not less than 8 grams of the test sample can be obtained. The test sample should be tested as soon as possible.

10.1.2. Determination

Table II

Commodity	Provision	Method	Principles	Type
<u>Cooked rice wrapped in plant leaves</u>	<u>Peroxide Value</u>	<u>ISO 3960 / AOCS Cd 8b-90</u>	<u>Titrimetry</u>	<u>IV</u>

Appendix VII

**FAO/WHO COORDINATING COMMITTEE FOR NORTH AMERICA AND SOUTHWEST PACIFIC
(CCNASWP16)**

Methods of analysis and sampling for provisions in the draft Regional Standard for Fermented Noni Fruit Juice

Provision	Method	Principle	Type	Notes
Brix value (Soluble solids)	AOAC 983.17 EN 12143 IFUMA 8 ISO 2173	Refractometry	†	Adopted for fruit juices and nectars
<u>Identification of scopoletin</u>	<u>Annex A*</u>	<u>Solid phase extraction and thin layer chromatography</u>	<u>IV</u>	
<u>Identification of deacetylasperulosidic acid</u>	<u>Annex B*</u>	<u>Thin layer chromatography</u>	<u>IV</u>	

IDENTIFICATION OF SCOPOLETIN**1. PREPARATION OF SAMPLES**

Noni fruit juice is filtered through a 0.45 µm membrane filter and then purified by solid-phase extraction (SPE) with Waters OASIS® HLB 6cc 200 mg extraction cartridges (or similar solid-phase extraction cartridge), after first equilibrating with **methanol (5 mL) followed by deionized water (5 mL)**. The **filtered juice** samples (3 mL) are then loaded onto the **equilibrated** cartridge and washed with 5% methanol (MeOH) **in deionized water (5 mL)**. **The cartridges are allowed to dry under flow of air for 5 mins and then, eluted with MeOH (3mL)**. The MeOH eluate is retained for TLC analysis. **The SPE flow rates of equilibration, wash and elution solvents through the cartridge is approximately 1 drop per second.**

2. PREPARATION OF REFERENCE STANDARD

- 2.1 A reference standard is prepared by dissolving **0.1 mg** Scopoletin in 1 milliliter of methanol.
- 2.2 Alternately, certified *Morinda citrifolia* reference plant material may be prepared in the same manner as the samples to be analyzed. The certified *Morinda citrifolia* reference material should be from the same part of the plant as the samples to be analyzed.

3. IDENTIFICATION**3.1 THIN LAYER CHROMATOGRAPHY**

Spot 5 microliters of sample solutions and reference standard solution on a silica gel 60 F254 thin layer chromatography (TLC) plate. After spotting the plates are dried at 110°C for 15 minutes in a drying oven. Develop the plate with a mobile phase of dichloromethane:methanol (19:1, v/v). View bright fluorescent blue colours on developed plate under UV lamp, 365 nm. Identify Scopoletin in samples by comparing Rf values and colours to the standard.

REFERENCES

1. Deng S, West BJ, Jensen J. A Quantitative Comparison of Phytochemical Components in Global Noni Fruits and Their Commercial Products. *Food Chemistry* 2010, 122 (1): 267-270.
2. Potterat O, et al. Identification of TLC markers and quantification by HPLC-MS of various constituents in noni fruit powder and commercial noni-derived products. *Journal of Agricultural and Food Chemistry* 2007, 55(18):7489–7494.
3. Basar S, Westendorf J. Identification of (2E, 4Z, 7Z)-Decatrienoic Acid in Noni Fruit and Its Use in Quality Screening of Commercial Noni Products. *Food Analytical Methods* 2011, 4(1):57-65. DOI: 10.1007/s12161-010-9125-9.
4. Chan-Blanco Y, et al. The ripening and aging of noni fruits (*Morinda citrifolia* L.): microbiological flora and antioxidant compounds. *Journal of the Science of Food and Agriculture* 2007, 87:1710 – 1716.
5. West BJ, Deng S. Thin layer chromatography methods for rapid identity testing of *Morinda citrifolia* L. (noni) fruit and leaf. *Advance Journal of Food Science and Technology* 2010, 2(5):298-302.

IDENTIFICATION OF DEACETYLASPERULOSIDIC ACID

1. PREPARATION OF SAMPLES

Noni fruit juice is filtered through a 0.45 µm membrane filter and diluted 1:1 with MeOH and then purified by solid phase extraction (SPE) with Waters OASISS® extraction cartridges, or similar solid phase extraction cartridge. [SPE cartridges is first equilibrated with water, followed by methanol. The samples are then loaded onto the cartridge and washed with 5% MeOH, followed by 100% MeOH. The MeOH eluate is retained for TLC analysis.]

2. PREPARATION OF REFERENCE STANDARD

2.1 A reference standard is prepared by dissolving 1 mg deacetylasperulosidic acid in 1 milliliter of methanol.

2.2 Alternately, certified *Morinda citrifolia* reference plant material may be prepared in the same manner as the samples to be analyzed. The certified *Morinda citrifolia* reference material should be from the same part of the plant as the samples to be analyzed.

3. PREPARATION OF p-ANISALDEHYDE SOLUTION

Anisaldehyde solution was prepared by dissolving 2g of p-anisaldehyde in 96 mL of ethanol with stirring. The solution was then acidified through dropwise addition of concentrated sulfuric acid (4 mL).

4. IDENTIFICATION

4.1 THIN LAYER CHROMATOGRAPHY

Spot 5 microliters of sample solutions and reference standard solution on a silica gel 60 F254 thin layer chromatography (TLC) plate, previously dried at 110 °C for 15 minutes in a drying oven. After spotting samples are again dried at 110°C or through application of heat via a heat gun for a period of 8-10 seconds. The TLC plates are developed with a mobile phase of dichloromethane: methanol: water (13:6:1, v/v/v). Upon completion of elution, the plate is air dried and developed by spraying with 2% anisaldehyde / 4% sulfuric acid in ethanol (EtOH) solution and then heat in oven at 110 °C for 1-5 minutes to reveal and maximise the blue colour. Identify deacetylasperulosidic in samples by comparing spot Rf values and colour with reference standard solution on same TLC plate.

REFERENCES

1. Potterat O, et al. Identification of TLC markers and quantification by HPLC-MS of various constituents in noni fruit powder and commercial noni-derived products. Journal of Agricultural and Food Chemistry 2007, 55(18):7489–7494.
2. Deng S, et al. Determination and comparative analysis of major iridoids in different parts and cultivation sources of *Morinda citrifolia*. Phytochemical Analysis 2011, 22(1):26-30.
3. West BJ, Deng S. Thin layer chromatography methods for rapid identity testing of *Morinda citrifolia* L. (noni) fruit and leaf. Advance Journal of Food Science and Technology 2010, 2(5):298-302

ANNEX C

SINGLE LABORATORY VERIFICATION / VALIDATION FOR IDENTIFICATION OF SCOPOLETIN AND DEACETYASPERULOSIDIC ACID IN FERMENTED NONI JUICE

The performance characteristics validation for an 'Identification test' is usually limited to 'Selectivity'. Where the capability of an analytical procedure to identify an analyte can be confirmed by obtaining positive results comparable to a known reference material with samples containing the analyte, along with negative results from samples which do not contain the analyte. In addition, the identification test can be applied to materials structurally similar to or closely related to the analyte to confirm that an undesired positive response is not obtained. Specificity/selectivity can be verified by demonstrating that the measured result of an analyte is comparable to the measured result of a second, well characterized analytical procedure (e.g., an orthogonal procedure).

Thus,

- a) the colour response with the TLC visualization technique with standards, and a relative response for increasing standard concentration tested was confirmed,
- b) the coloured TLC spot with samples with a R_f similar to the standard was confirmed for different Fermented Noni juices from a range of pacific island locations (supplied by Scientific Research Organisation of Samoa (SROS)-Apia),
- c) various juices observed mixed in commercial Noni products were tested along with a Noni Juice by TLC to confirm a negative result for other juices.
- d) an orthogonal HPLC technique based on Choi et al (2022)¹¹ was used to measure concentrations or absence of the identity analytes for selected samples, and PDA spectra along with R_t used to confirm HPLC peak identity.

For Scopoletin Identification

- a) Colour response under UV@365nm and relative intensity/response for Scopoletin TLC standards at 0.001, 0.01, 0.1 and 1.0 mg/mL in MeOH. We thus suggest that a 0.10 mg/mL Scopoletin standard may be more appropriate in the Scopoletin TLC identification.

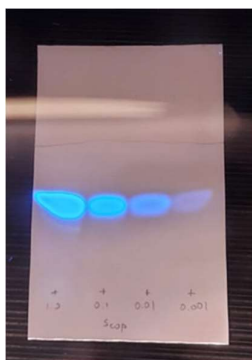


Figure 1 TLC for Scopoletin standards at 1.0, 0.1, 0.01 and 0.001 mg/mL in MeOH at 365nm.

- b) Colour response under UV@365nm and R_f relative to standard Scopoletin for various Pacific Island samples.

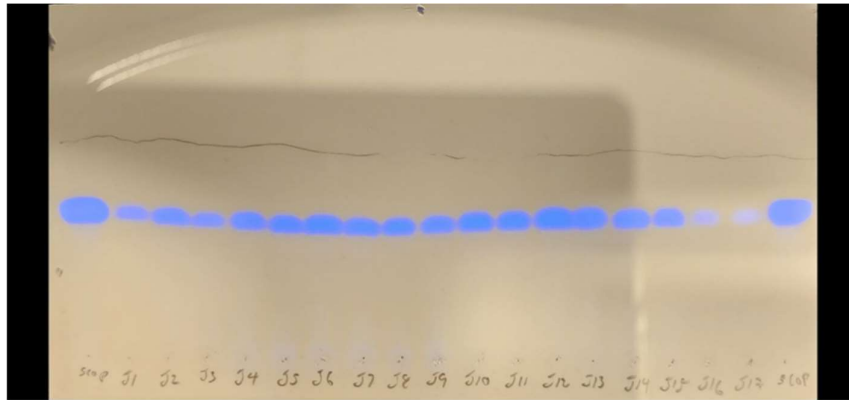


Figure 2. Scopoletin standard plus SPE extraction & TLC with UV@365nm visualization of fermented Noni juice samples, with left to right, standard; fermented Noni juice samples J1-17; standard.

Standard and Pacific Island samples J18-J19.

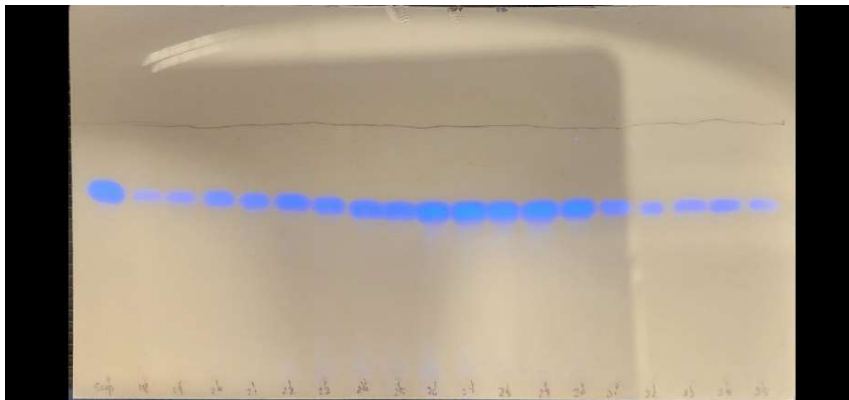


Figure 3. Scopoletin standard plus SPE extraction & TLC with UV@365nm visualization of fermented Noni juice samples, with left to right, standard; fermented Noni juice samples 18-35.

- c) Following is the Scopoletin TLC Identification test applied to various juices observed mixed in commercial Noni products, including commercial pineapple juice, apple and blackcurrant juice, grape juice, pear juice, and coconut juice.

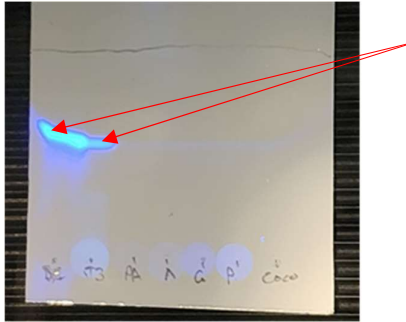


Figure 4. Scopoletin ID TLC for a Noni juice plus various other juices; from left to right, Scopoletin (0.1mg/mL), Noni Juice#3(J3), Pineapple juice (PA), Apple and Blackcurrant juice(A), Grape juice(G), Pear juice(P), and Coconut juice (Coco). Scopoletin band for standard and Noni Juice#3 indicated by red arrows, where the absence of similar band for the other samples gives a negative Scopoletin Identification.

- a) An orthogonal HPLC technique based on Choi et al (2022)¹¹ used to confirm 'presence' or 'absence' of the identity analytes for selected samples, and PDA spectra along with peak at Rt=22.8min(approx.) used to confirm HPLC peak identity.

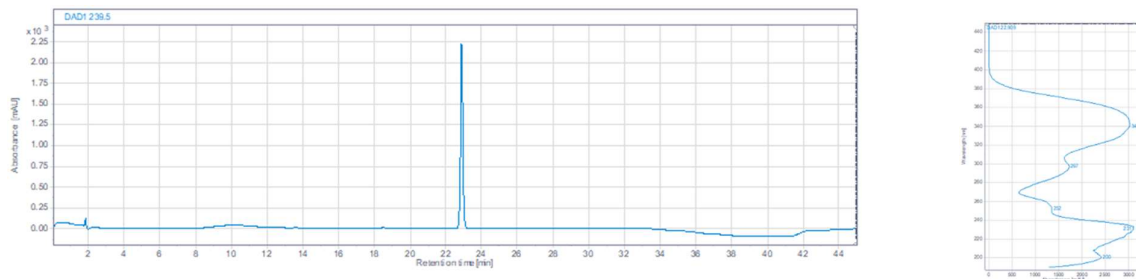


Figure 5. Scopoletin standard, HPLC-DAD chromatogram, 10 μ L injection, @ 239.5nm and peak UV spectra.

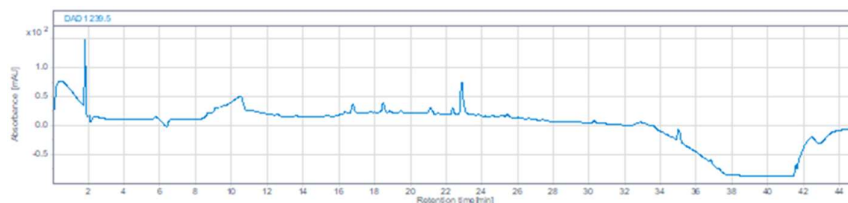


Figure 6. Juice#3, filtered, HLB-SPE 100% MeOH elution solution and injected 10 μ L on HPLC-DAD @ 239.5nm

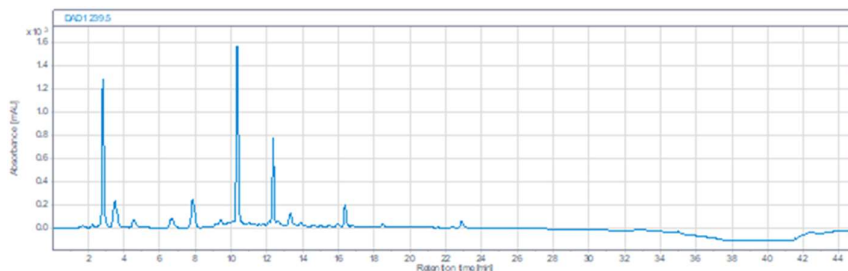


Figure 7. Juice#3, filtered 0.45 μ m, no SPE, and direct injected 10 μ L on HPLC-DAD @ 239.5nm. See section f) for HPLC-DAD conditions.

For Deacetylasperulosidic acid Identification

- a) Colour response with 2% anisaldehyde / 10% sulfuric acid-ethanol (EtOH) solution then heating for visualisation, and relative intensity/response at 1.0, 0.5, 0.25 and 0.1 mg/mL Deacetylasperulosidic acid.

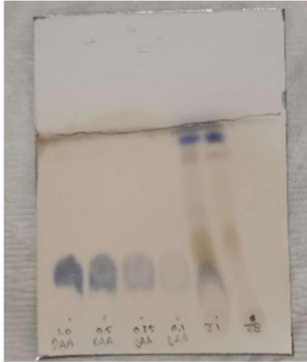


Figure 8. TLC standard solutions spots 1.0 mg/mL, 0.5 mg/mL, 0.25 mg/mL, 0.1 mg/mL; Juice 1; Juice 8.

- b) Colour response with 2% anisaldehyde / 10% sulfuric acid-ethanol (EtOH) solution then heating for visualisation, and R_f relative to standard Deacetylasperulosidic acid for various Pacific Island samples.

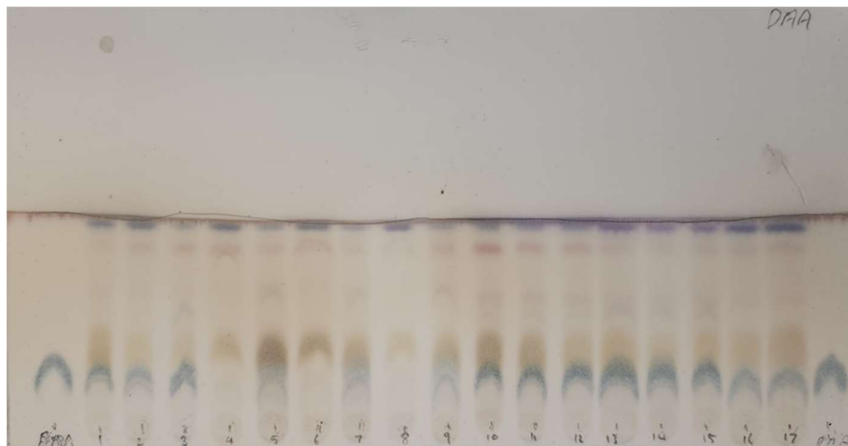


Figure 9. From left to right, DAA standard; fermented Noni juice samples 1-17; DAA standard; with TLC visualised with 2% anisaldehyde / 4% sulfuric acid-ethanol (EtOH) solution then heating.

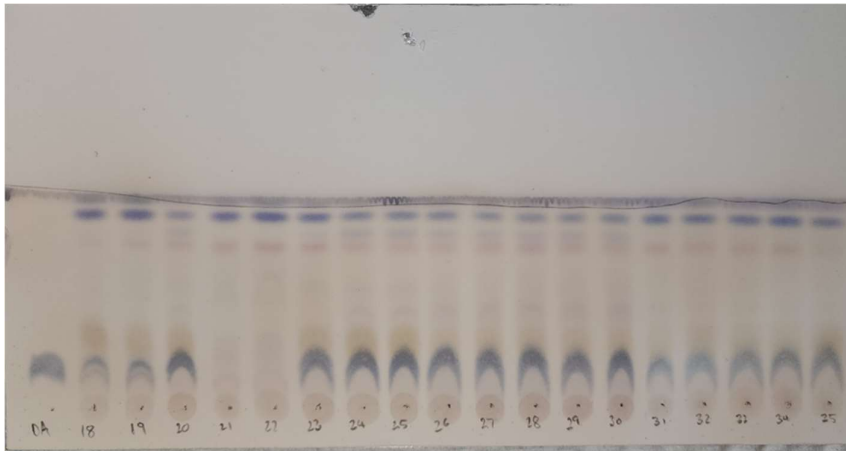


Figure 10. From left to right, DAA standard, fermented Noni juice samples 18-35; with TLC visualisation with 2% anisaldehyde / 4% sulfuric acid-ethanol (EtOH) solution then heating.

Note: Samples sourced from SROS-Apia for purpose of verification of TLC method for Scopoletin and DAA. Samples may have been subjected to adverse conditions during transport or pre-sampling prior to shipment to Australia. No conclusion can be inferred for Juices, 4, 6, 8, 21, 22 other than HPLC-DAD and TLC are in alignment in the absence or scarcity of DAA analyte. Further investigation would be required on non-compliant sample to determine the reason behind these atypical or non-compliant findings.

- c) Following is the Deacetylasperulosidic acid TLC Identification test applied to various juices observed mixed in commercial Noni products, including commercial pineapple juice, apple and blackcurrant juice, grape juice, pear juice, and coconut juice.

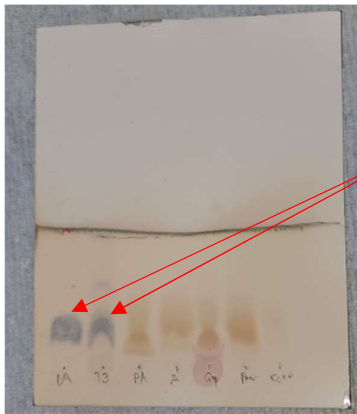


Figure 11. Deacetylasperulosidic acid ID TLC for a Noni juice plus various other fruit juices; from left to right, Deacetylasperulosidic acid (0.5mg/mL DA), Noni Juice#3(J3), Pineapple juice (PA), Apple and Blackcurrant juice(A), Grape juice (Gp), Pear juice (Pear), and coconut juice (Co). Deacetylasperulosidic acid blue band indicated by red arrow in standard and Juice#3, where the absence of similar blue bands for the other samples gives a 'negative' identification.

- d) An orthogonal HPLC technique based on Choi et al (2022)¹¹ used to confirm 'presence' or 'absence' of the identity analytes for selected samples, and PDA spectra along with Rt used to confirm HPLC peak identity.

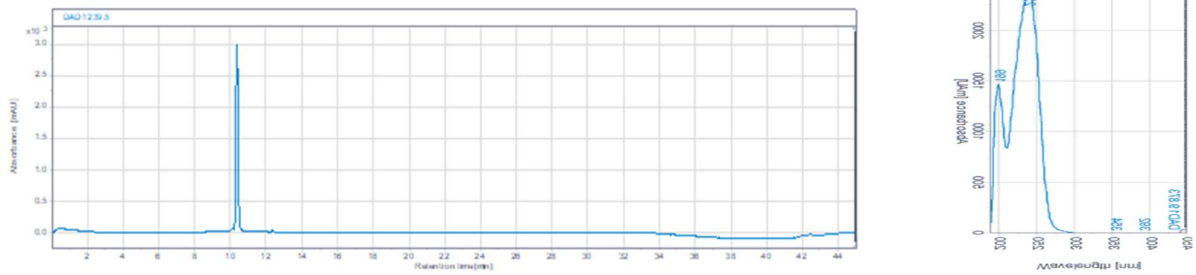


Figure 12. Deacetylasperulosidic acid 2 mg/mL; HPLC-DAD chromatogram, 10µL injection, @ 239.5nm; and peak UV spectra.

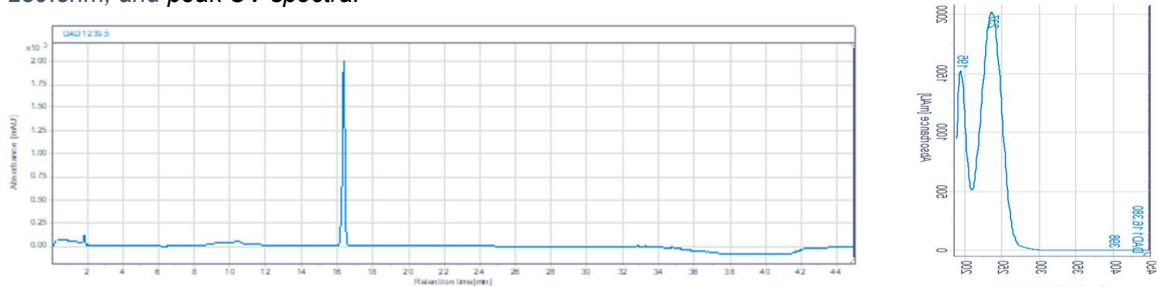


Figure 13. Asperulosidic acid; HPLC-DAD chromatogram, 10µL injection, @ 239.5nm; and peak UV spectra.

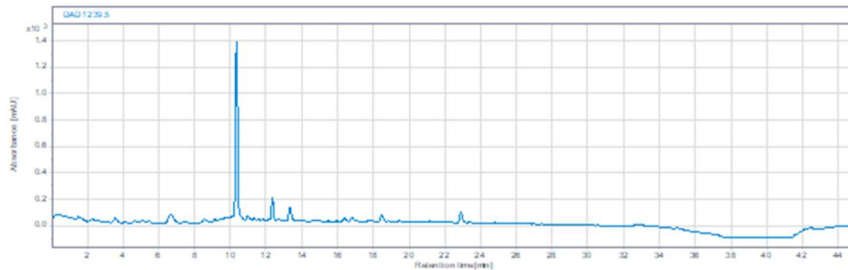


Figure 14. Juice 1, 0.45µm filtered & no SPE, direct injected 10µL on HPLC-DAD @ 239.5nm
See section f) for HPLC-DAD conditions.

- e) HPLC confirmation of the Deacetylasperulosidic acid ID by TLC for selected Pacific Island Noni Juice samples where 'negative' and 'positive' DAA IDs were observed.

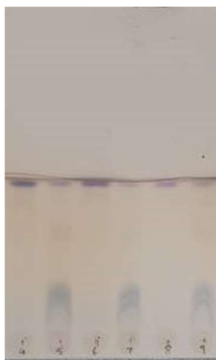


Figure 15 Cropped section of Fig 10, highlighting Deacetylasperulosidic acid ID by TLC for a selection of various Pacific Island Noni Juice samples where 'negative' and 'positive' DAA IDs were observed.

The juices in the following chromatograms were 0.45µm filtered and injected directly onto the HPLC-DAD with 10µL injection. The specific pattern to note is that according to the TLC, juices 4, 6 and 8 show a 'negative' DAA identification; while juice 5, 7, 9 show a 'positive' DAA identification. As observed in the following the HPLC-DAD chromatograms confirm the TLC results, with 'presence' or 'absence' of a sharp DAA peak at approximately 9.9 min, with 10uL injection, using 239.5 nm wavelength detection. Note, all these juices have a peak at $R_t=22.8$ mins, thus positive ID for Scopoletin. Note the 10x reduction in absorbance scale for the negative results for DAA.

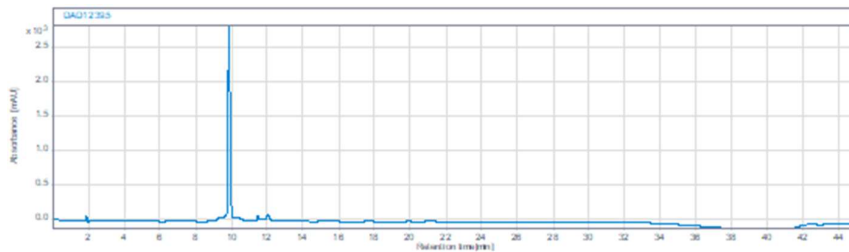


Figure 16. HPLC of DAA standard 2mg/mL with peak at 9.9mins.

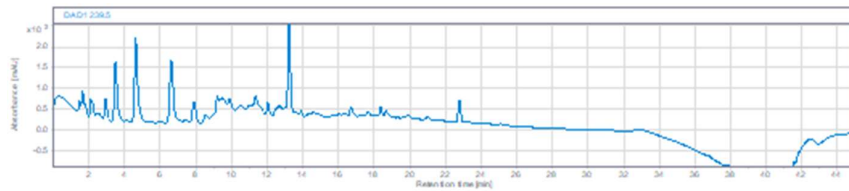


Figure 17. HPLC injection of Pacific Island juice#4, confirming 'negative' result for DAA.

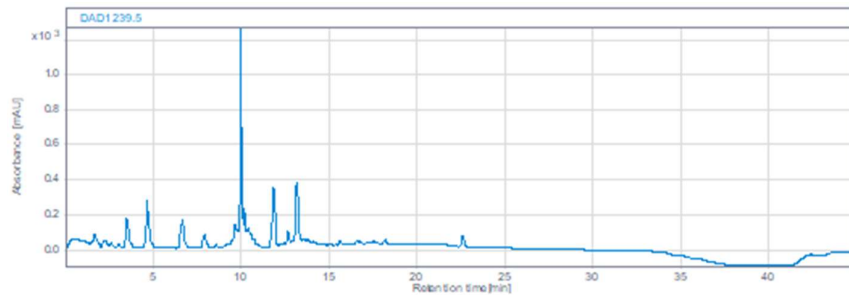


Figure 18. HPLC injection of Pacific Island juice#5, confirming 'positive' result for DAA.

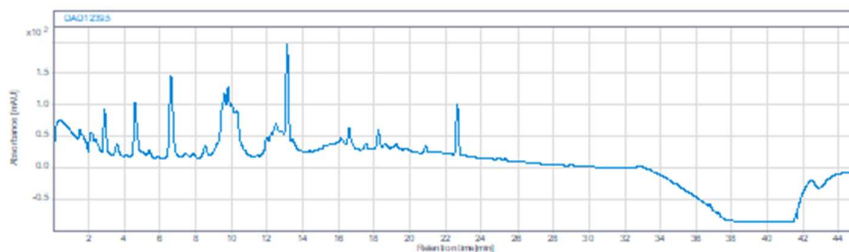


Figure 19. HPLC injection of Pacific Island juice#6, confirming 'negative' result for DAA.

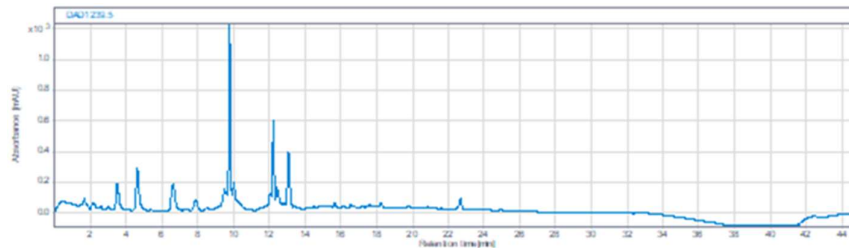


Figure 20. HPLC injection of Pacific Island juice#7, confirming 'positive' result for DAA.

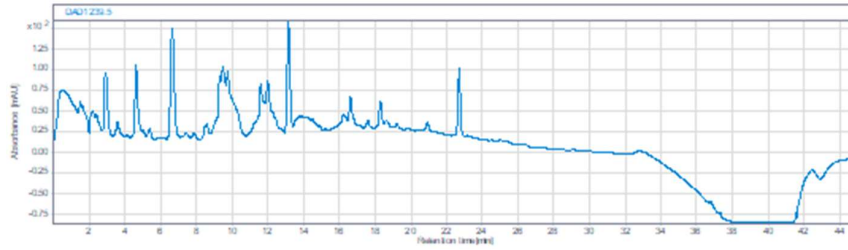


Figure 21. HPLC injection of Pacific Island juice#8, confirming 'negative' result for DAA.

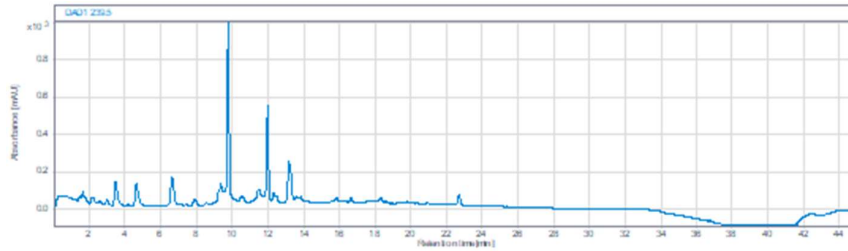


Figure 22. HPLC injection of Pacific Island juice#9, confirming 'positive' result for DAA.

f) Conditions for HPLC-DAD

HPLC-DAD was performed on an Agilent 1260 Infinity HPLC

Instrument: Agilent 1260 Infinity

Gradient:	Time(min)	0.1% Formic acid in deionised water	Acetonitrile
	0	100	0
	5	100	0
	30	65	35
	35	0	100
	39	0	100
	40	100	0
	45	100	0

Run time: 45 minutes

Wavelengths monitored: 239.5 nm (Deacetylasperulosidic Acid); 344 nm (Scopoletin),

Peak width: >0.2 min (4s response time) 1.25Hz

Injection volume: 10µL

Mobile phase flow rate: 1.0 mLs/minute

Column temperature: 25 °C

Column: Agilent, Zorbax Eclipse Plus C18. 5um, 4.6 x 150 mm, (PN:959993-902, SN:USUXB20707, LN:B20104)

Guard Column: Agilent, Zorbax Eclipse Plus C18 2.1 x5, 1.8 micron (PN:821725-901, SN:USEDP03464)

Standard Operating Procedure for the Identification of Kavalactones and Flavokavains in Fresh and Dried Kava Products by High Performance Thin Layer Chromatography in the Regional Standard for Kava Products for Use as a Beverage when Mixed with Water (CXS 336R-2020)

1.0 Introduction

Piper methysticum G. Forst. (*Piperaceae*) rhizomes and roots are peeled, grinded, macerated in cold water, and pressed through a cloth strainer to prepare kava, a non-alcoholic beverage. The composition and quality of kava can be highly variable, depending on the age of the plant, the variety, and the part used to prepare the beverage: roots, rhizomes, or basal stems. The six major kavalactones (KLs: yangonin = Y, dihydrokavain = DHK, desmethoxyyangonin = DMY, kavain = K, dihydromethysticin = DHM and methysticin = M) are responsible for the physiological effect and are usually quantified with HPLC. There is a second group of molecules flavokavins (FKs: A, B, C). The chemical composition of the kava extract is strongly influenced by the extraction solvent and extraction technique. This procedure is based on analytical procedure using High Performance Thin Layer Chromatography (HPTLC). The HPTLC is a validated procedure for 174 varieties of kava.

Scope: Identification of Kavalactones and Flavokavins by High Performance Thin Layer Chromatography

2.0 Materials and methodology

2.1 Preparation of Samples

- Wash by hand under cold running water the kava roots and peeled rhizomes.
- Cut into small pieces the kava organs with a knife.
- Sun-dry the kava pieces for 3 days (similar to traditional practises).
- Grind the dried kava matter into powder using a Forplex F00 1218 hammer mill to achieve <2 mm particle size and pack into labelled zip-log plastic bags.
- Further ground the kava powder to very fine kava flour texture using a coffee grinder.
- Weigh the kava flour sample then dry in an oven at 60°C for 6 hours.

2.2 Preparation of Reference Standard

- Make available Six kavalactone and three flavokavain standards of analytical grade possibly available from Sigma-Aldrich including standards of:

Six kavalactones:

- o methysticin (M),
- o dihydromethysticin (DHM),
- o kavain (KAV),
- o dihydrokavain (DHK),
- o yangonin (Y),
- o desmethoxyyangonin (DMY),

Three flavokavain:

- o flavokavain A (FKA),
 - o flavokavain B (FKB) and
 - o flavokavain C (FKC).
- Accurately weigh 1.0mg individually the pure kava standard powder into 1ml acetone
 - store in dark at 4°C if analysed later.

Checking Purity of Standards:

- Conduct peak purity tests for the kava standards using the UV Vis spectrophotometer and compare the UV spectra.

2.3 Sample extraction

- Weigh 10g of kava powder,
- Transfer to a clean 50ml polypropylene centrifuge tube and add 30ml acetone.
- Sonicate the tubes in a water bath for 30min
- Transfer to a centrifuge instrument and set at 4500 rpm for 10min.
- Transfer the supernatant to a 9mm wide opening screw thread vial of 2ml amber glass.
- Store vials in refrigerator at 4°C in dark till required for analysis.

2.4 Identification by High Performance Thin Layer Chromatography (HPTLC)

2.4.1 Chemicals and reagents for HPTLC analysis

- Analytical grade solvent (acetone, dioxane, hexane and methanol).
- Silica gel 60 F254 plates (dimension; 20 x 10cm) using Camag HPTLC system with an automatic TLC sampler (ATS 4) coupled to an automatic developing chamber (ADC 2) and a visualizer as well as a TLC Scanner 4 controlled with winCATS software.

2.4.2 Check standards and prepare Sample Run

- Prepare standards and sample solutions at bands (length of 8 mm, 250 nL/s delivery speed, track distance 8.0 mm and distance from the edge of 15 mm).
- Conduct standard linearity curve check by using the HPTLC plates. Apply different stock solutions (0.1, 0.2, 0.4, 0.6, 0.8, 1.0 µL) of the six KLs and three FKs scan at 240nm (for M, DHM, K, DHK) and scan at 355nm (for Y, DMY, FKA, FKB, FKC).
- Add 10 mL mobile phase to develop the plates using hexane:dioxane (8:2 v/v) with a migration distance of 80 mm at room temperature after 30 s of pre-drying and no tank saturation.
- Visual documentation of the plates is carried out at 254 nm and 366 nm.
- Scan the plates in reflectance mode at 240 nm (for M, DHM, K and DHK) and at 355 nm (for Y, DMY, FKA, FKB, FKC) with D2 and W lamp slit dimension 8.00 mm × 0.20 mm, scanning speed 20 mm/s, and data resolution 100 µm/step.
- Identify the Peak area measurements (in area units, AU).
- Ensure that the total analytical time is 50 min for 20 samples and 10 mL of mobile phase (corresponding to 2.5 min and 0.5 mL per sample).

3.0 References

Lebot, V., Michalet, S., Legendre, L. (2019). Kavalactone and Flavokavins Profile Contribute to Quality Assessment of Kava (*Piper methysticum* G. Forst), the Traditional Beverage of the Pacific. *Beverages*. 2019, 1-14.

CODEX COMMITTEE ON CONTAMINANTS IN FOODS (CCCF16)**Appendix VIII****Sampling Plans Provisions in the General Standard for Contaminants in Food and Feed (CXS 193-1995)**

Commodity	Analyte	ML (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	Precision (%)	Minimal applicable range (µg/kg)	Recovery (%)
Maize grain	AF B1+B2+G1+G2	15	≤ 3	≤ 6	≤44	8.4 - 21.6	60-115
	AFB1	-	≤0.75 1.5	≤ 1.5 3.0	≤44	2.1 - 5.4 4.2 - 10.8	40-120 60-115
	AFB2	-	≤0.75 0.5*	≤1.5 1*	≤44	2.1 - 5.4 1.4 - 3.6	40-120
	AFG1	-	≤0.75 0.5*	≤ 1.5 1*	≤44	2.1 - 5.4 1.4 - 3.6	40-120
	AFG2	-	≤0.75 0.5*	≤ 1.5 1*	≤44	2.1 - 5.4 1.4 - 3.6	40-120
Maize flour, meal, semolina and flakes derived from maize; Sorghum grain; cereal-based foods for infants and young children for food aid programs	AF B1+B2+G1+G2	10	≤2	≤4	≤44	5.6 - 14.4	60-115
	AFB1	-	≤0.5 1.0	≤1.0 2.0	≤44	1.4 - 3.6 2.8 - 7.2	40-120 60-115
	AFB2	-	≤0.5 0.33*	≤1.0 0.67*	≤44	1.4 - 3.6 0.9 - 2.4	40-120
	AFG1	-	≤0.5 0.33*	≤1.0 0.67*	≤44	1.4 - 3.6 0.9 - 2.4	40-120
	AFG2	-	≤0.5 0.33*	≤1.0 0.67*	≤44	1.4 - 3.6 0.9 - 2.4	40-120
Husked Rice	AF B1+B2+G1+G2	20	≤4	≤8	≤44	11.2 - 28.8	60-115
	AFB1	-	≤1.0 2.0	≤2.0 4.0	≤44	2.8 - 7.2 5.6 - 14.4	40-120 60-115
	AFB2	-	≤1.0 0.67*	≤2.0 1.33*	≤44	2.8 - 7.2 1.9 - 4.8	60-115
	AFG1	-	≤1.0 0.67*	≤2.0 1.33*	≤44	2.8 - 7.2 1.9 - 4.8	60-115
	AFG2	-	≤1.0 0.67*	≤2.0 1.33*	≤44	2.8 - 7.2 1.9 - 4.8	60-115
Polished Rice; Cereal-based food for infants and young children	AF B1+B2+G1+G2	5	≤1	≤2	≤44	2.8 - 7.2	40-120
	AFB1	-	≤0.25 0.5	≤0.5 1	≤44	0.7 - 1.8 1.4 - 3.6	40-120
	AFB2	-	≤0.25 0.17*	≤0.5 0.33*	≤44	0.7 - 1.8 0.5 - 1.2	40-120
	AFG1	-	≤0.25 0.17*	≤0.5 0.33*	≤44	0.7 - 1.8 0.5 - 1.2	40-120
	AFG2	-	≤0.25 0.17*	≤0.5 0.33*	≤44	0.7 - 1.8 0.5 - 1.2	40-120

Summary of proposed changes in CXS 234, including update to references of existing methods and recommendations to CCMAS

Table 1. Recommended Methods of Analysis and Sampling (CXS 234-1999)

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737 IDF 13 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	250	I
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat ¹³ (MSNF)	ISO 6731 IDF 21 and ISO 1737 IDF 13 ISO 23318 IDF 249	Calculation from total solids content and fat content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb)	250	I
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ¹³	ISO 6731 IDF 21 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	250	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ¹³	ISO 6731 IDF 21 and ISO 1737 IDF 13 ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	250	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737 IDF 13 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	250	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF) ¹³	ISO 6731 IDF 21 and ISO 1737 IDF 13 ISO 23318 IDF 249	Calculation from total solids content and fat content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb)	250	I

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ¹³	ISO 6731 IDF 21 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	250	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ¹³	ISO 6731 IDF 21 and ISO 1737 IDF 13 ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	250	IV
Blend of skimmed milk and vegetable fat in powdered form	Total fat	ISO 1736 IDF 9 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	251	I
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF ¹³	ISO 5537 IDF 26 and ISO 1736 IDF 9 ISO 23318 IDF 249 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	251	IV
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF ⁸	ISO 5537 IDF 26 and ISO 1736 IDF 9 ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	251	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Total fat	ISO 1736 IDF 9 ISO 23318 IDF 249	Gravimetry (Röse -Gottlieb)	251	I

⁸ Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF ¹³	ISO 5537 IDF 26 and ISO 1736 IDF 9 ISO 23318 IDF 249 and ISO 8968 - 1 IDF 20 - 1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87 °C and Gravimetry (Röse -Gottlieb) and Titrimetry (Kjeldahl)		IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF ¹³	ISO 5537 IDF 26 and ISO 1736 IDF 9 ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 87 °C and Gravimetry (Röse -Gottlieb) and Titrimetry (Kjeldahl)	251	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737 IDF 13 ISO 23318 IDF 249	Gravimetry (Röse -Gottlieb)	252	I
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids -not - fat ¹³ (MSNF)	ISO 6734 IDF 15 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 2911 IDF 35 and	Calculation from total solids content, fat content and sucrose content Gravimetry, drying at 102 °C and Gravimetry (Röse -Gottlieb) and Polarimetry	252	IV
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF ¹³	ISO 6734 IDF 15 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 2911 IDF 35 and ISO 8968 -1 IDF 20 - 1	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse -Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	252	IV

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF ¹³	ISO 6734 IDF 15 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 2911 IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	252	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737 IDF 13 ISO 23318 IDF 249	Gravimetry (Röse -Gottlieb)	252	I
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids -not - fat ¹³ (MSNF)	ISO 6734 IDF 15 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 2911 IDF 35	Calculation from total solids content, fat content and sucrose content Gravimetry, drying at 102 °C and Gravimetry (Röse -Gottlieb) and Polarimetry	252	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF ¹³	ISO 6734 IDF 15 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 2911 IDF 35 and ISO 8968 -1 IDF 20 - 1	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse -Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	252	IV

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF ¹³	ISO 6734 IDF 15 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 2911 IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	252	IV
Cheese	Milkfat	ISO 1735 IDF 5 ISO 23319 IDF 250	Gravimetry (Schmid-Bondzynski - Ratzlaff)	283	I
Cheeses, individual	Milkfat in dry matter	ISO 5534 IDF 4 ISO 1735 IDF 5 ISO 23319 IDF 250	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry	263 to 278	I
Cheeses in brine	Milkfat in dry matter (FDM)	ISO 5534 IDF 4 ISO 1735 IDF 5 ISO 23319 IDF 250	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid- BondzynskiRatzlaff)	208	I
Cottage cheese	Fat-free dry matter	ISO 5534 IDF 4 and ISO 1735 IDF 5 ISO 23319 IDF 250	Calculation from dry matter content and fat content Gravimetry, drying at 102 °C Gravimetry (Schmid-Bondzynski-Ratzlaff)	273	I
Cottage cheese (for samples containing lactose up to 5%)	Milkfat in dry matter	ISO 5534 IDF 4 and ISO 1735 IDF 5 ISO 23319 IDF 250	Calculation from dry matter content and fat content Gravimetry, drying at 102 °C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	273	I

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Cottage cheese (for samples containing lactose up to 5%)	Milkfat	ISO 1735 IDF 5 ISO 23319 IDF 250	Gravimetry (Schmid-Bondzynski-Ratzlaff)	273	I
Cream cheese	Moisture on fat-free basis	ISO 5534 IDF 4 ISO 1735 IDF 5 ISO 23319 IDF 250	Calculation from fat content and moisture content Gravimetry drying at 102°C (forced air oven) Gravimetry (Schmid-Bondzynski-Ratzlaff)	275	I
Dairy permeate powders	Milkfat	ISO 1736 IDF 9 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	331	I
Dairy permeate powders	Lactose	ISO 22662 IDF 198	High performance liquid chromatography	331	II
Edible casein products	Milkfat (Total fat)	ISO 5543 IDF 127 ISO 23318 IDF 249	Gravimetry (Schmid-Bondzynski-Ratzlaff)	290	I
Milk powders and cream powders	Milkfat	ISO 1736 IDF 9 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	207	I
Mozzarella	Milkfat in dry matter – with high moisture	ISO 5534 IDF 4 and ISO 1735 IDF 5 ISO 23319 IDF 250	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	262	I

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Mozzarella	Milkfat in dry matter – with low moisture	ISO 5534 IDF 4 and ISO 1735 IDF 5 ISO 23319 IDF 250	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	262	I
Whey cheeses by coagulation	Milkfat	ISO 1735 IDF 5 ISO 23319 IDF 250	Gravimetry (Schmid-Bondzynski-Ratzlaff)	284	I
Whey cheeses by coagulation	Milkfat in dry matter	ISO 1735 IDF 5 ISO 23319 IDF 250 and ISO 5534 IDF 4	Calculation from fat content and dry matter content Gravimetry (Schmid-Bondzynski-Ratzlaff) Gravimetry, drying at 102°C	284	I
Fermented milks	Milkfat	ISO 1211 IDF 1 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	243	I
Cream	Milkfat	ISO 2450 IDF 16 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	288	I
Creams lowered in milkfat content	Milkfat	ISO 2450 IDF 16 ISO 23318 IDF 249 / AOAC 995.19	Gravimetry (Röse-Gottlieb)	288	I
Evaporated milks	Milkfat	ISO 1737 IDF 13 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	281	I
Evaporated milks	Milk Protein in MSNF ¹³	ISO 1737 IDF 13 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	281	I
Sweetened condensed milk	Milkfat	ISO 1737 IDF 13 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	282	I
Sweetened condensed milks (for products sweetened with sucrose only)	Milk Protein in MNSF ¹³	ISO 6734 IDF 15 and ISO 1737 IDF 13 ISO 23318 IDF 249 and ISO 2911 IDF 35 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content, sucrose and protein content Gravimetry, drying at 102 °C and Polarimetry Gravimetry (Röse-Gottlieb) Titrimetry (Kjeldahl)	282	I

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Whey cheeses by concentration (carbohydrate contents below 5%)	Milkfat (Total fat)	ISO 1854 IDF 59 ISO 23318 IDF 249	Gravimetry (Röse Gottlieb)	284	I
Whey cheeses by concentration (for carbohydrate content under 5%)	Milkfat in dry matter (total fat in dry matter)	ISO 1854 IDF 59 ISO 23318 IDF 249 and ISO 2920 IDF 58	Calculation from fat content and dry matter content Gravimetry (Röse Gottlieb) Gravimetry, drying at 88 °C	284	I
Whey powders	Milkfat	ISO 1736 IDF 9 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	289	I
Infant formula	Total fat	AOAC 989.05 ISO 8381 IDF 123 ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	72	I

Other updates necessary in CXS 234 to be considered

- 2 lines for aqueous coconut products refer to ISO 1211 | IDF 1, and shall need to be updated to ISO 23318|IDF 249
- 2 lines on page 56 of the 2021 version of CXS 234 - * DETERMINATION OF WATER CAPACITY OF CONTAINERS (CAC/RM 46)

Appendix X

Section A – Matters agreed by CCFO27

Commodity	Provision	Method	Principle	Type	Comment
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, & propyl gallate	AOAC 983.15; or AOCS Ce 6-86	Liquid chromatography	II	
Fats and oils	<u>Synthetic phenolic antioxidants</u>	<u>AOCS Ce 6-86 AOCS Ce 6a-2021</u>	Liquid chromatography	II	
Fats and oils	<u>Synthetic phenolic antioxidants</u>	AOAC 983.15	Liquid chromatography	III	
Fish oils	Fatty acid composition	AOCS Ce 1a-13	Capillary GLC	III	
Fish oils	Fatty acid composition	AOCS Ce 2-66	Preparation of methyl esters by fatty acids	III	
Fish oils	Fatty acid composition	AOCS Ce 2b-11	Alkali hydrolysis	III	
Fish oils	Fatty acid composition	AOCS Ce 2b-11 and AOCS Ce 1j-07	Gas Chromatography of methyl esters	III	
Fish oils	Fatty acid composition	AOCS Ce 1j-07	Capillary GLC	III	
Fish oils	Fatty acid composition	ISO 12966-2	Gas chromatography	III	
Fish oils	Fatty acid composition	ISO 5508	Gas chromatography	III	
Fish oils	Fatty acid composition	AOCS Ce 2-66 and AOCS Ce 1i-07	<u>Preparation of methyl esters and Gas Chromatography</u>	III II	
Fish oils	Fatty acid composition	AOCS Ce 2-66 and AOCS Ce 1a-13	<u>Gas Chromatography of methyl esters</u>	III	Remove from CXS 234
Fish oils	Fatty acid composition	AOCS Ce 2b-11 Ce 2c-66 and AOCS Ce 1i-07 / AOCS Ce 1j-07	<u>Preparation of methyl esters and Gas Chromatography</u>	III	
Fish oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4	<u>Preparation of methyl esters and Gas Chromatography</u>	III	
Fish oils	Fatty acid composition	AOCS Ce 1b-89	GLC	III	Remove from CXS 234
Named Animal Fats	GLC ranges of fatty acid composition	ISO 5508 and ISO 12966-2; or AOCS Ce 2-66 and Ce 1e-91 or Ce 1f-96	Gas chromatography of methyl esters	II	

Named Animal Fats	Fatty acid composition	ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and Ce 1f-96 1j-07	Gas Chromatography of methyl esters	#	
Named Animal Fats	Fatty acid composition	<u>AOCS Ce 2-66 and Ce 1j-07</u>	<u>Preparation of methyl esters and Gas Chromatography</u>	##	
Named Animal Fats	Fatty acid composition	AOCS Ce 2-66 and Ce 1f-96	Gas Chromatography of methyl esters	#	<i>Remove from CXS 234</i>
Named Animal Fats	Fatty acid composition	<u>ISO 12966-2 and ISO 12966-4</u>	<u>Preparation of methyl esters and Gas Chromatography</u>	# III	
Named Animal Fats	Titre	ISO 935; or AOCS Cc 12-59	Thermometry	‡	
Named Animal Fats	Titre	<u>ISO 935</u>	Thermometry	I	
Named Animal Fats	Titre	<u>AOCS Cc 12-59^a</u>	Thermometry	‡ IV	
Named Vegetable Oils	Crismer value	AOCS Cb 4-35 and AOCS Ca 5a-40	Calculation from individual fatty acid composition (gas chromatography of methyl esters) and turbidity	I	
Named Vegetable Oils	Halphen test	AOCS Cb 1-25	Colorimetry	I	
Named Vegetable Oils	Unsaponifiable matter	ISO 3596; or ISO 18609; or AOCS Ca 6b-53	Gravimetry	‡	
Named Vegetable Oils	Unsaponifiable matter	<u>ISO 3596 / AOCS Ca 6b-53</u>	<u>Diethyl ether extraction and gravimetry, drying at 103 °C and titrimetry (colorimetry) and correction for free fatty acids titrimetry (colorimetry)^c</u>	I	
Named Vegetable Oils	Unsaponifiable matter	<u>ISO 18609^b</u>	<u>Hexane extraction and Gravimetry, drying at 103 °C and titrimetry (colorimetry) and correction for free fatty acids titrimetry (colorimetry)^c</u>	‡ IV	

^a **AOCS Cc 12-59** is the preferred method in certain regions. Due to differences in practical application of AOCS Cc 12-59 compared to ISO 935, it is listed as a Type IV method.

^b Results obtained from ISO 18609 are systematically lower. In case of limitations due to climate or regulations that prohibit the use of diethyl ether, ISO 18609 can be used instead of the Type I method.

^c The technique according to ISO is gravimetric. The correction by titration and colorimetry is only when it is necessary to correct for free fatty acids.

Section B – Fish oil - Vitamin A and Vitamin D review

Commodity	Provision	Method	Principle	Type
Fish Oil	Vitamin A^a	European Pharmacopeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29	<u>LC Liquid Chromatography</u>	III
Fish Oil	Vitamin A^a	EN 12823-1 (Determination of vitamin A by high performance liquid chromatography – Part 1: Measurement of all-E-retinol and 13-Z-retinol)	<u>LC Liquid Chromatography</u>	III II
Fish Oil	Vitamin D^b	NMKL 167 / EN 12821 (Determination of vitamin D by high performance liquid chromatography – Measurement of cholecalciferol (D3) or ergocalciferol (D2))	<u>LC Liquid Chromatography</u>	III II

^a The respective standard on fish oils CXS 329-2017 states that Vitamin A is expressed as 'Retinol equivalents'(RE) where RE takes into account the fact that different vitamers of vitamin A differ in activity. ISO/TR 23304:2021 "Food products – Guidance on how to express vitamins and their vitamers" may give clarity on this matter, for example for the relevant activities of the all-E-retinol levels and 13-Z-retinol levels.

^b The provision accounts for Vitamin D2 and D3.

Appendix XII

Group 1. Methods reviewed by CPL EWG with decisions

Cereals, Pulses and Legumes and Derived Products							
Commodity	Provision	Codex Standard	Method	Principle	Type	Committee	Comments
Certain pulses	Moisture	CXS 171-1989 (2019)	ISO 665	Gravimetry (oven drying at 103°C)	I	CCCPL	soybeans
Certain pulses	Moisture	CXS 171-1989 (2019)	ISO 24557 / AACC 44-17.01	Gravimetry (oven drying at 130°C)	I	CCCPL	except soybeans
Degermed maize (corn) meal and maize (corn) grits	Ash	CXS 155-1985 (2019)	AOAC 923.03 / ISO 2171 and ICC 110/1 ICC Method No 104/4	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	
Degermed maize (corn) meal and maize (corn) grits	Fat, crude	CXS 155-1985 (2019)	AOAC 945.38F; and 920.39C and ICC 110/1	Calculation from moisture and Gravimetry (ether extraction)	I	CCCPL	
Degermed maize (corn) meal and maize (corn) grits	Moisture	CXS 155-1985 (2019)	ISO 712 ICC Method No 110/4 ICC 110/1	Gravimetry (oven drying at 130 – 133°C)	I	CCCPL	
Degermed maize (corn) meal and maize (corn) grits	Particle size (granularity)	CXS 155-1985 (2019)	AOAC 965.22 ¹ and ISO 3310-1	Sieving	I	CCCPL	
Degermed maize (corn) meal and maize (corn) grits	Protein	CXS 155-1985 (2019)	ICC Method No 105/4 ICC 105/2 and ICC 110/1	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCCPL	
Durum wheat semolina and durum wheat flour	Ash (semolina)	CXS 178-1991 (2019)	AOAC 923.03 / ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry	I	CCCPL	

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				(incineration at 550°C)		
Durum wheat semolina and durum wheat flour	Moisture	CXS 178-1991 (2019)	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 – 133°C)	I	CCCPL
Durum wheat semolina and durum wheat flour	Protein (N x 5.7)	CXS 178-1991 (2019)	ICC 105/1 2 and ISO 712 / ICC 110/1	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCCPL
Instant Noodles	Extraction of oil from instant noodles	CXS 249-2006 (2019)	described in the standard	Gravimetry (ether extraction)	I	CCCPL
Instant Noodles	Acid Value	CXS 249-2006 (2019)	described in the standard, will be moved to 234	Titrimetry (ether extraction)	I	CCCPL
Instant Noodles	Moisture	CXS 249-2006 (2019)	described in the standard, will be moved to 234	Gravimetry (oven drying at 105°C)	I	CCCPL
Maize (corn)	Moisture	CXS 153-1985 (2019)	ISO 6540 / ICC 110/1	Gravimetry (oven drying at 130 – 133°C)	I	CCCPL
Aflatoxin methods to be replaced with performance criteria once correct criteria are established						
Peanuts (raw)	Aflatoxins, total	CXS 200-1995 (2019) CXS 193-1995 (2019)	AOAC 991.31 (A – G)	Immunoaffinity column (IAC); (Aflatest); fluorometry	II III	CCCPL/CC CF
Peanuts (raw)	Aflatoxins, total as Σ of aflatoxins, B1 B2 G1 and G2	CXS 200-1995 (2019) CXS 193-1995 (2019)	AOAC 991.31 (A – F, H)	Immunoaffinity column (Aflatest) IAC (Aflatest) and HPLC-Post column	II	CCCPL/CC CF

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				<u>derivatization (PCD)</u>			
Peanuts (raw) <u>(intended for further processing)</u>	<u>Aflatoxins, total</u>	<u>CXS 200-1995 (2019)</u> <u>CXS 193-1995 (2019)</u>	AOAC 993.17	Thin layer chromatography	III <u>IV</u>	CCCPL /CCCF	Method uses hazardous reagents (benzene/chloroform) not all aflatoxins captured by method, recommend removal
Peanuts (intended for further processing)	Aflatoxins, total	<u>CXS 200-1995 (2019)</u> <u>CXS 193-1995 (2019)</u>	AOAC 975.36	<u>IAC</u> (Romer minicolumn)	III <u>IV</u>	CCCPL/CCCF	Qualitative/ semi-quantitative screening method; does not meet performance criteria in Procedural Manual; recommend removal
Peanuts (Cereals, shell fruits and derived products (including peanuts))	Sum of aflatoxins B ₁ , B ₂ , G ₁ and G ₂	<u>CXS 200-1995 (2019)</u> <u>CXS 193-1995 (2019)</u>	EN 12955 / ISO 16050	<u>IAC</u> , HPLC-PCD	III	CCCPL	EN 12955 withdrawn
Peanuts (intended for further processing)	Aflatoxins, total	<u>CXS 200-1995 (2019)</u> <u>CXS 193-1995 (2019)</u>	AOAC 979.18	<u>IAC</u> (Holaday-Velasec minicolumn)	III <u>IV</u>	CCCPL/CCCF	Qualitative/ semi-quantitative screening method; does not meet performance criteria in Procedural Manual; recommend removal also uses benzene
Pearl millet flour	Ash	CXS 170-1989 (2019)	AOAC 923.03 / <u>ISO 2171</u> and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (<u>incineration at 550°C</u>)	I	CCCPL	
Pearl millet flour	Colour	CXS 170-1989 (2019)	<i>Modern Cereal Chemistry</i> , 6th Ed., D.W. Kent Jones and A.J. Amos (Ed.), pp. 605-612,	Colorimetry using (specific colour grader)	IV	CCCPL	Colour-grading equipment used in method is no longer available, possible use of other item capable of results of the style of the original;

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				Food Trade Press Ltd, London, 1969.				sample is affected by bleach and method requires benzene; there does not appear to be a conversion factor from Kent Jones units to the more commonly used CIE Lab color space, making it difficult to determine whether or not the products comply with the limit/range listed in the Standard. reconsideration of provision/method suggested by reviewers
Pearl millet flour	Fat, crude	CXS 170-1989 (2019)	AOAC 945.38F; and 920.39C and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (ether extraction)	I	CCCPL		
Pearl millet flour	Fibre, crude	CXS 170-1989 (2019)	ISO 5498 (B-5 Separation) and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (extraction and filtration)	I	CCCPL		
Pearl millet flour	Moisture	CXS 170-1989 (2019)	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 – 133°C)	I	CCCPL		
Pearl millet flour	Protein	CXS 170-1989 (2019)	AOAC 920.87 ISO 20483 and ISO 712 / ICC 110/1	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCCPL		
Quinoa	Moisture content	CXS 333-2019 (2020)	ISO 712 / AACCI 44-15.02	Gravimetry (oven drying at 130 – 133°C)	†	CCCPL	Moved to Appendix II, for further consideration	

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Cereals, Pulses and Legumes and Derived Products							
Quinoa	Protein (N x 6.25 in dry weight basis)	CXS 333-2019 (2020)	ISO 20483 ISO 1871 and ISO 712	Calculation from moisture and Titrimetry (Kjeldahl digestion)	IV !	CCCPL	Moved to Appendix II, for further consideration Suggest that N factor be prescribed in commodity standards if internationally agreed, but not in CXS 234.
Sorghum flour	Ash	CXS 173-1989 (2019)	AOAC 923.03 / ISO 2171 ICC 104/1 and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	
Sorghum flour	Colour	CXS 173-1989 (2019)	<i>Modern Cereal Chemistry</i> , 6th Ed., D.W. Kent Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry using (specific colour grader)	IV	CCCPL	Colour-grading equipment used in method is no longer available, possible use of other item capable of results of the style of the original; sample is affected by bleach and method requires benzene; there does not appear to be a conversion factor from Kent Jones units to the more commonly used CIELab color space, making it difficult to determine whether or not the products comply with the limit/range listed in the Standard. reconsideration of provision/method suggested by reviewers
Sorghum flour	Fat, crude	CXS 173-1989 (2019)	AOAC 945.38F; and 920.39C and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (ether extraction)	I	CCCPL	
Sorghum flour	Fibre, crude	CXS 173-1989 (2019)	ICC 113 / ISO 6541 and ISO 712 / ICC 110/1	Calculation from moisture and	I	CCCPL	

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				Gravimetry (separation, incineration)			
Sorghum flour	Moisture	CXS 173-1989 (2019)	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 – 133°C)	I	CCCPL	
Sorghum flour	Particle size (granularity)	CXS 173-1989 (2019)	AOAC 965.22 ¹ and ISO 3310-1	Sieving	I	CCCPL	
Sorghum flour	Protein	CXS 173-1989 (2019)	ICC 105/1 ² and ISO 712 / ICC 110/1	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCCPL	
Sorghum flour	Protein ¹ (N x 6.25)	CXS 173-1989 (2019)	ISO 1871	Titrimetry (Kjeldahl digestion)	†	CCCPL	ISO 1871 Type IV listed in CXS-173, not CXS 234, review of ICC 105/2 completed in 2021/22 and accepted
Sorghum flour	Tannins	CXS 173-1989 (2019)	ISO 9648 and ISO 712 / ICC 110/1	Calculation from moisture and Spectrophotometry	I	CCCPL	Method established for sorghum grains, samples to be crushed, not milled as occurs for flour
Sorghum grains	Ash	CXS 172-1989 (2019)	AOAC 923.03 / ISO 2171 ICC 104/1 and ISO 6540	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL	
Sorghum grains	Fat, crude	CXS 172-1989 (2019)	AOAC 945.38F ₇ and 920.39C and ISO 6540	Calculation from moisture and Gravimetry (ether extraction)	I	CCCPL	
Sorghum grains	Moisture	CXS 172-1989 (2019)	ISO 6540	Gravimetry (oven drying at 130 – 133°C)	I	CCCPL	

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Sorghum grains	Protein	CXS 172-1989 (2019)	ICC 105/42 and ISO 6540	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCCPL	
Sorghum grains	Protein ¹ (N x 6.25)	CXS 172-1989 (2019)	ISO 1871	Titrimetry, Kjeldahl digestion	†	CCCPL	ISO 1871 Type IV listed in CXS-172, not CXS 234, review of ICC 105/2 completed in 2021/22 and accepted
Sorghum grains	Tannins	CXS 172-1989 (2019)	ISO 9648 and ISO 6540	Calculation from moisture and Spectrophotometry	I	CCCPL	
Soy protein products	Ash	CXS 175-1989 (2019)	AOAC 923.03 / ISO 2171: (Method B) and AOAC 925.09	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCVP	
Soy protein products	Fibre, crude	CXS 175-1989 (2019)	ISO 5498 and AOAC 925.09	Calculation from moisture and Gravimetry (separation) (extraction and filtration)	I	CCVP	
Soy protein products	Moisture	CXS 175-1989 (2019)	AOAC 925.09	Gravimetry (vacuum oven at 98 – 100°C)	I	CCVP	
Vegetable protein products	Ash	CXS 174-1989 (2019)	AOAC 923.03 / ISO 2171 (Method B) and AOAC 925.09	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCVP	
Vegetable protein products	Fibre, crude	CXS 174-1989 (2019)	AACC 32-17 32-10.01 and AOAC 925.09	Calculation from moisture and Gravimetry	I	CCVP	

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				(Ceramic filter filtration)			
Vegetable protein products	Moisture	CXS 174-1989 (2019)	AOAC 925.09	Gravimetry (vacuum oven at 98 – 100°C)	I	CCVP	
Wheat flour	Ash	CXS 152-1985 (2019)	AOAC 923.03 / ISO 2171 ICC 104/1	Gravimetry (incineration at 550°C)	I	CCCPL	
Wheat flour	Fat acidity	CXS 152-1985 (2019)	AOAC 939.05 ISO 7305 and ISO 712 / ICC 110/1	Calculation from moisture and Titrimetry (extraction)	I	CCCPL	
Wheat flour	Moisture	CXS 152-1985 (2019)	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 – 133°C)	I	CCCPL	
Wheat flour	Particle size (granularity)	CXS 152-1985 (2019)	AOAC 965.22 ¹ and ISO 3310-1	Sieving	I	CCCPL	
Wheat flour	Protein	CXS 152-1985 (2019)	ICC 105/1 ² and ISO 712: / ICC 110/1	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCCPL	
Wheat flour	Protein¹ (N x 5.7)	CXS 152-1985 (2019)	ISO 1871	Titrimetry (Kjeldahl digestion)	I	CCCPL	ISO 1871 Type IV listed in CXS-152, not CXS 234, review of ICC 105/2 completed in 2021/22 and accepted
Wheat protein products including wheat gluten	Fibre, crude ⁴	CXS 163-1987 (2001)	AOAC 962.09 and AOAC 925.09	Calculation from moisture and Gravimetry Ceramic fibre (ceramic fibre filtration)	I	CCVP	

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<u>Wheat protein products including wheat gluten</u>	<u>Moisture</u>	<u>CXS 163-1987 (2001)</u>	<u>AOAC 925.09</u>	<u>Gravimetry (vacuum oven at 98 – 100°C)</u>	<u>I</u>	<u>CCVP</u>	
Wheat protein products including wheat gluten	Crude Protein ⁺ ; excluding added vitamins, minerals, amino acids and optional ingredients	CXS 163-1987 (2001)	Vital wheat gluten and devitalized wheat gluten AOAC 979.09 (wheat protein in grain N x 5.7) <u>ISO 20483 and AOAC 925.09</u>	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCVP	Suggest that N factor be prescribed in commodity standards if internationally agreed, but not in CXS 234.
			Solubilized wheat protein AOAC 920.87 (wheat protein in flour N x 5.7) <u>ISO 20483 and AOAC 925.09</u>	Calculation from moisture and Kjeldahl Titrimetry (Kjeldahl digestion) (wheat protein in flour N x 5.7)	I	CCVP	
Wheat protein products including wheat gluten	Ash	CXS 163-1987 (2001)	AOAC 923.03 / ISO 2171: method B and AOAC 925.09	Calculation from moisture and Gravimetry (<u>incineration at 550°C</u>)	I	CCVP	
Whole and decorticated pearl millet grains	Ash	CXS 169-1989 (2019)	AOAC 923.03 / <u>ISO 2171</u> and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (<u>incineration at 550°C</u>)	I	CCCPL	
Whole and decorticated pearl millet grains	Fat, crude	CXS 169-1989 (2019)	AOAC 945.38F; <u>and</u> 920.39C and ISO 712 / ICC 110/1	Calculation from moisture and	I	CCCPL	

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					Gravimetry (ether extraction)			
Whole and decorticated pearl millet grains	Fibre, crude	CXS 169-1989 (2019)	ISO 5498 (B.5 separation) and ISO 712 / ICC 110/1	Calculation from moisture and Gravimetry (filtration through filter paper)	I	CCCPL		
Whole and decorticated pearl millet grains	Moisture	CXS 169-1989 (2019)	ISO 712 / ICC 110/1	Gravimetry (oven drying 130 – 133°C)	I	CCCPL		
Whole and decorticated pearl millet grains	Protein	CXS 169-1989 (2019)	AOAC 920.87 ISO 20483 and ISO 712 / ICC 110/1	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCCPL		
Whole maize (corn) meal	Ash	CXS 154-1985 (2019)	AOAC 923.03 / ISO 2171 ICC 104/1 and ICC 110/1	Calculation from moisture and Gravimetry (incineration at 550°C)	I	CCCPL		
Whole maize (corn) meal	Crude -Fat, crude	CXS 154-1985 (2019)	AOAC 945.38F; and 920.39C and ICC 110/1	Calculation from moisture and Gravimetry (ether extraction)	I	CCCPL		
Whole maize (corn) meal	Moisture	CXS 154-1985 (2019)	ISO 712 ICC 110/1 / ISO 6540	Gravimetry (oven drying 130 – 133°C)	I	CCCPL		
Whole maize (corn) meal	Particle size (granularity)	CXS 154-1985 (2019)	AOAC 965.22 ¹ and ISO 3310-1	Sieving	I	CCCPL	AACC 66-20.01 not identical, different sample size	
Whole maize (corn) meal	Protein	CXS 154-1985 (2019)	ICC 105/1 2 and ICC 110/1	Calculation from moisture and Titrimetry (Kjeldahl digestion)	I	CCCPL		

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<u>Gari</u>	<u>Total acidity</u>	<u>CXS 151-1989 (2019)</u>	<u>ISO/DP 7305</u> <u>AOAC 1975 14.064 –</u> <u>14.065 (AOAC 939.05)</u> <u>and ISO 712</u>	<u>Titrimetry (ethanol</u> <u>extraction)</u>	<u>!</u>	<u>CCCPL</u>	<u>Reviewer supported 7305;</u> <u>AOAC 1975 14.064 –</u> <u>14.065 is AOAC 939.05,</u> <u>using an earlier</u> <u>numbering system. AOAC</u> <u>939.05 reviewed 2021/2022</u> <u>and proposed for wheat</u> <u>flour, but was</u> <u>recommended for</u> <u>replacement due to</u> <u>hazardous chemical</u> <u>usage</u>
Gari	Crude fibre	CXS 151-1989 (2019)	ISO 5498 <u>and ISO 712</u>	Gravimetry (separation)		CCCPL	General method
Gari	Ash	CXS 151-1989 (2019)	ISO 2171 <u>and ISO 712</u>	Calculation from moisture Gravimetry (incineration <u>at</u> <u>550°C</u>)		CCCPL	
Gari	Moisture	CXS 151-1989 (2019)	ICC 109/1 ISO 712	Gravimetry (<u>oven</u> <u>drying 130 –</u> <u>133°C</u>)		CCCPL	No method given in CXS 151, listed in CXS 234: ISO 712 accepted for other commodities, ICC 109/1 states not to be used for commercial disputes
Edible Cassava flour	Moisture	CXS 176-1989 (2019)	ISO 712	Gravimetry (<u>oven</u> <u>drying at 98 –</u> <u>100°C</u>)	!	CCCPL	No method given in CXS 176, listed in CXS 234: ISO 712 accepted for other commodities
Edible Cassava flour	Crude fibre	CXS 176-1989 (2019)	ISO 5498 (B.5 separation)	Gravimetry (separation)		CCCPL	General method
Edible Cassava flour	Ash	CXS 176-1989 (2019)	ISO 2171 <u>and ISO 712</u>	Calculation from moisture Gravimetry (incineration <u>at</u> <u>550°C</u>)		CCCPL	

Group 2. Methods requiring additional follow up action**Cereals, Pulses and Legumes and Derived Products**

Commodity	Provision	Codex Standard	Method	Principle	Type	Committee	Comments
Pearl millet flour	Colour	CXS 170-1989 (2019)	<i>Modern Cereal Chemistry</i> , 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry using (specific colour grader)	IV	CCCPL	Colour-grading equipment used in method is no longer available, reconsideration of provision/method suggested by reviewers.
Quinoa	Moisture content	CXS 333-2019 (2020)	ISO 712 + AACCI 44-15.02	Gravimetry (<u>oven drying at 130 – 133°C</u>)	I	CCCPL	Methods are not identical, both methods endorsed by CCMAS as identical in a previous meeting. Further consideration may be needed, given the larger sample size with AACCI 44-15.02
Quinoa	Protein (N x 6.25 in dry weight basis)	CXS 333-2019 (2020)	<u>ISO 20483</u> ISO 1871 and ISO 712	Calculation from moisture and Titrimetry (Kjeldahl digestion)	IV I	CCCPL	Validation information for ISO 1871 required, data are available and anticipated to be shared for review. While ISO 20483 is acceptable for cereals, quinoa is a pseudocereal and it is not considered as a good fit.
Sorghum flour	Colour	CXS 173-1989 (2019)	<i>Modern Cereal Chemistry</i> , 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry using (specific colour grader)	IV	CCCPL	Colour-grading equipment used in method is no longer available, reconsideration of provision/method suggested by reviewers.
Soy protein products	Fat	CXS 175-1989 (2019)	CAC/RM 55 - Method 1	Gravimetry (extraction)	I	CCVP	Method is not available

Group 2. Methods requiring additional follow up action**Cereals, Pulses and Legumes and Derived Products**

							Replacement requested, none identified to date
Soy protein products	Protein; <u>excluding added vitamins, minerals, amino acids and food additives (N-x 6.25)</u>	CXS 175-1989 (2019)	AOAC 955.04D	Titrimetry (Kjeldahl digestion)	I	CCVP	Recommend revoke method and replace – mercury used Replacement requested, none identified to date
Vegetable protein products	Fat	CXS 174-1989 (2019)	CAC/RM 55 - Method 1	Gravimetry (extraction)	I	CCVP	Method is not available Replacement requested, none identified to date
Vegetable protein products	<u>Crude Protein; excluding added vitamins, minerals, amino acids and food additives</u>	CXS 174-1989 (2019)	AOAC 955.04D	Titrimetry (Kjeldahl digestion)	II	CCVP	Recommend revoke method and replace – mercury used Replacement requested, none identified to date
Gari	Particle size (classification)	CXS 151-1989 (2019)	ISO 2591-1	Sieving	I	CCCPL	Recommended for removal, however, classification determined by sieve size used. ISO 2591 provides general guidance on sieving protocols, but is not specific to CPL.
Edible Cassava flour	Particle size	CXS 176-1989 (2019)	ISO 2591-1	Sieving	I	CCCPL	Recommended for removal, however, classification determined by sieve size used. ISO 2591 provides general guidance on sieving protocols, but is not specific to CPL.

Group 3: Methods proposed by SDOs as updates and/or replacements for methods currently in CXS 234

Cereals, Pulses and Legumes and Derived Products							
Commodity	Provision	Codex Standard	Original Method entry	Original Principle	Type	Committee	Comments
Degermed maize (corn) meal and maize (corn) grits	Ash ¹	CXS 155-1985 (2019)	AOAC 923.03 <u>l</u> ISO 2171	Gravimetry (<u>incineration</u>)	I	CCCPL	<i>C&G recommends addition of AACC 08-01.01</i>
Degermed maize (corn) meal and maize (corn) grits	Fat, crude ¹	CXS 155-1985 (2019)	AOAC 945.38F ₃ ; and 920.39C	Gravimetry (ether extraction)	I	CCCPL	<i>ISO recommends addition of ISO 11085 C&G recommends addition of AACC 30-25.01</i>
Degermed maize (corn) meal and maize (corn) grits	Moisture	CXS 155-1985 (2019)	ISO 712 ICC Method No 110/1 ICC 110/1	Gravimetry (<u>oven drying</u>)	I	CCCPL	<i>ISO recommends addition of ISO 6540</i>
Degermed maize (corn) meal and maize (corn) grits	Protein ¹	CXS 155-1985 (2019)	ICC 105/1 <u>2</u>	Titrimetry (Kjeldahl digestion)	I	CCCPL	<i>ISO recommends addition of ISO 20483 C&G recommends addition of AACC 46-16.01 (copper sulfate catalyst)</i>
Durum wheat semolina and durum wheat flour	Ash ¹ (semolina)	CXS 178-1991 (2019)	AOAC 923.03 <u>l</u> ISO 2171	Gravimetry (<u>incineration</u>)	I	CCCPL	<i>C&G recommends addition of AACC 08-12.01 (semolina)</i>
Durum wheat semolina and durum wheat flour	Protein ¹ (N x 5.7)	CXS 178-1991 (2019)	ICC 105/1 <u>2</u>	Titrimetry (Kjeldahl digestion)	I	CCCPL	<i>ISO recommends addition of ISO 20483 C&G recommends addition of AACC 46-16.01 (copper sulfate catalyst)</i>
Pearl millet flour	Ash ¹	CXS 170-1989 (2019)	AOAC 923.03 <u>l</u> ISO 2171	Gravimetry (<u>incineration</u>)	I	CCCPL	<i>C&G recommends addition of AACC 08-01.01</i>
Pearl millet flour	Fat ¹	CXS 170-1989 (2019)	AOAC 945.38F ₃ ; and 920.39C	Gravimetry (ether extraction)	I	CCCPL	<i>ISO recommends addition of ISO 11085</i>

Cereals, Pulses and Legumes and Derived Products							
Sorghum flour	Ash ¹	CXS 173-1989 (2019)	AOAC 923.03 <u>I</u> ISO 2171	Gravimetry (incineration)	I	CCCPL	<i>C&G recommends addition of AACC 08-01.01</i>
Sorghum flour	Fat, crude ¹	CXS 173-1989 (2019)	AOAC 945.38F; and 920.39C	Gravimetry (ether extraction)	I	CCCPL	<i>ISO recommends addition of ISO 11085</i>
Sorghum flour	Protein ¹ (N x 6.25)	CXS 173-1989 (2019)	ICC 105/4 <u>2</u>	Titrimetry (Kjeldahl digestion)	I	CCCPL	<i>ISO recommends addition of ISO 20483 C&G recommends addition of AACC 46-16.01 (copper sulfate catalyst)</i>
Sorghum grains	Ash ¹	CXS 172-1989 (2019)	AOAC 923.03 <u>I</u> ISO 2171 ICG 104/4	Gravimetry (incineration)	I	CCCPL	<i>C&G recommends addition of AACC 08-01.01</i>
Sorghum grains	Fat, crude ¹	CXS 172-1989 (2019)	AOAC 945.38F; and 920.39C	Gravimetry (ether extraction)	I	CCCPL	<i>ISO recommends addition of ISO 11085 C&G recommends addition of AACC 30-25.01</i>
Sorghum grains	Protein ¹ (N x 6.25)	CXS 172-1989 (2019)	ICC 105/4 <u>2</u>	Titrimetry (Kjeldahl digestion)	I	CCCPL	<i>ISO recommends addition of ISO 20483 C&G recommends addition of AACC 46-16.01 (copper sulfate catalyst)</i>
Soy protein products	Ash ¹	CXS 175-1989 (2019)	AOAC 923.03 <u>I</u> ISO 2171; (Method B)	Gravimetry (incineration)	I	CCVP	<i>C&G recommends addition of AACC 08-01.01</i>
Soy protein products	Moisture	CXS 175-1989 (2019)	AOAC 925.09	Gravimetry (vacuum oven)	I	CCVP	<i>ISO recommends addition of ISO 771 AACC recommends addition of 44-40.01</i>
Vegetable protein products	Moisture	CXS 174-1989 (2019)	AOAC 925.09	Gravimetry (vacuum oven)	I	CCVP	<i>AACC recommends addition of 44-40.01</i>

Cereals, Pulses and Legumes and Derived Products							
Wheat flour	Ash	CXS 152-1985 (2019)	AOAC 923.03 <u>I</u> ISO 2171 ICC 104/4	Gravimetry (incineration)	I	CCCPL	<i>C&G recommends addition of AACC 08- 01.01</i>
Wheat flour	Protein ¹ (<u>N x 5.7</u>)	CXS 152-1985 (2019)	ICC 105/12	Titrimetry (Kjeldahl digestion)	I	CCCPL	<i>ISO recommends addition of ISO 20483 C&G recommends addition of AACC 46- 16.01(copper sulfate catalyst)</i>
Wheat protein products including wheat gluten	Ash ¹	CXS 163-1987 (2001)	AOAC 923.03 <u>I</u> ISO 2171	Gravimetry (incineration)	I	CCVP	<i>C&G recommends addition of AACC 08- 01.01</i>
Wheat protein products including wheat gluten	Moisture	CXS 163-1987 (2001)	AOAC 925.09	Gravimetry (vacuum oven)	I	CCVP	<i>C&G recommends addition of AACC 44- 40.01</i>
Whole and decorticated pearl millet grains	Ash ¹	CXS 169-1989 (2019)	AOAC 923.03 <u>I</u> ISO 2171	Gravimetry (incineration)	I	CCCPL	<i>C&G recommends addition of AACC 08- 01.01</i>
Whole and decorticated pearl millet grains	Fat ¹	CXS 169-1989 (2019)	AOAC 945.38F ₂ and 920.39C	Gravimetry (ether extraction)	I	CCCPL	<i>ISO recommends addition of ISO 11085</i>
Whole maize (corn) meal	Ash ¹	CXS 154-1985 (2019)	AOAC 923.03 <u>I</u> ISO 2171	Gravimetry (incineration)	I	CCCPL	<i>C&G recommends addition of AACC 08- 01.01</i>
Whole maize (corn) meal	Crude fat ¹	CXS 154-1985 (2019)	AOAC 945.38F ₁ and 920.39C	Gravimetry (ether extraction)	I	CCCPL	<i>ISO recommends addition of ISO 11085 C&G recommends addition of AACC 30- 25.01</i>
Whole maize (corn) meal	Moisture	CXS 154-1985 (2019)	ICC 110/1	Gravimetry (oven drying)	I	CCCPL	<i>ISO recommends addition of ISO 6540</i>

Cereals, Pulses and Legumes and Derived Products							
Whole maize (corn) meal	Protein ¹ (N x 6.25)	CXS 154-1985 (2019)	ICC 105/42	Titrimetry, (†) Kjeldahl digestion)	I	CCCPL	<i>ISO recommends addition of ISO 20483 C&G recommends addition of AACC 46-16.01 (copper sulfate catalyst)</i>

¹ A correction for moisture content is frequently required for reporting results of the proximate methods (i.e., ash, protein and fat). 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.

Appendix XI Methods of Analysis for “Processed Fruits and Vegetables” commodity

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Processed fruits and vegetables* <u>(Jams, Jellies, Marmalades, pickled cucumbers, mango chutney, Coconut Milk and Coconut Cream)</u>	Benzoic acid	NMKL 124	Liquid Chromatography <u>(UV)</u>	II	CXS 192	Benzoic acid falls under CXS 192 – Food Additives. Numeric performance criteria developed 2024: these changes remain until then.
Processed fruits and vegetables* <u>(Jams, Jellies, Marmalades, pickled cucumbers, mango chutney, Coconut Milk and Coconut Cream)</u>	Benzoic acid	NMKL 103; AOAC 983.16	Gas Chromatography <u>(Flame ionization)</u>	III	CXS 192	NMKL 103 withdrawn because of the use of hazardous solvent
Processed fruits and vegetables* <u>(Canned strawberries, pickled cucumbers, preserved tomatoes, canned citrus fruits, certain canned vegetables)</u>	Calcium	AOAC 968.31	Complexometry / Titrimetry	II	CXS 192 CXS 62 CXS 115 CXS 13 CXS 254 CXS 297	Calcium firming agents listed in CXS 192 – food additives. Numeric performance criteria to be developed for 2024: these changes remain until then.
Processed fruits and vegetables	Drained Weight	AOAC 968.30 (Codex General Method)	Sieving Gravimetry <u>(Sieving)</u>	I		
Processed fruits and vegetables	Fill of <u>glass</u> containers	CAC/RM 46 (reference to “metal containers” deleted and refer to ISO 90-1 for determination of water capacity in metal containers) <u>ISO 8106</u>	Weighing <u>Gravimetry</u>	I		CCMAS36 (2015) agreed to replace CAC/RM 46 with ISO 8106
<u>Processed fruits and vegetables</u>	<u>Fill of metal containers</u>	<u>ISO 90-1</u>	Weighing <u>Gravimetry</u>	I		

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Processed fruits and vegetables* <u>(Canned Fruits, Jams, Jellies and Marmalades, Mango Chutney, Canned Vegetables, Preserved Tomatoes, Table Olives, Pickled Cucumbers)</u>	Lead	AOAC 972.25 (Codex general method)	AAS (Flame absorption) <u>Atomic Absorption Spectrophotometry (Flame)</u>	III <u>II</u>	CXS 193	Codex general method type II for other commodities. All PFV commodities are covered in CXS 193. Numeric performance criteria were developed at CCMAS42 (2023); this row to be struck and replaced with the criteria.
Processed fruits and vegetables	Packing medium Canned berry fruits (raspberry, strawberry)	AOAC 932.12 ISO 2173	Refractometry	†		AOAC 932.12 and ISO 2173 both determine soluble solids which is already listed below. Recommend striking this row.
Processed fruits and Vegetables* <u>(Pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney, and aqueous coconut products)</u> except canned bamboo shoots, pH determined by AOAC 981.12)	pH	ISO 1842	Potentiometry	IV	CXS 115 CXS 66 CXS 57 CXS 13 CXS 160 CXS 240	
<u>Canned bamboo shoots</u>	<u>pH</u>	<u>AOAC 981.12</u>	<u>Potentiometry</u>	<u>IV</u>	CXS 24	Validated in pimientos, marinated pimientos, 2 pH buffer solutions, chocolate syrup

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Processed fruits and vegetables* <u>(Pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney, and aqueous coconut products)</u>	pH	AOAC 981.12	Potentiometry	III	CXS 115 CXS 66 CXS 57 CXS 13 CXS 160 CXS 240	
Processed fruits and vegetables* <u>(Pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney, and aqueous coconut products)</u>	pH	NMKL 179	Potentiometry	II	CXS 115 CXS 66 CXS 57 CXS 13 CXS 160 CXS 240	
Processed fruits and vegetables* <u>(Pickled cucumbers, processed tomato concentrates, preserved tomatoes, canned applesauce, jams, jellies and marmalades, mango chutney, and certain canned fruit)</u>	Soluble solids <u>(packing medium)</u>	ISO 2173 AOAC 932.12	Refractometry	I	CXS 115 CXS 57 CXS 13 CXS 17 CXS 296 CXS 160 CXS 319	These methods are not identical. Suggest retaining ISO method which contains more detailed procedures.
Processed fruits and vegetables* <u>(Jams, Jellies, Marmalades, pickled cucumbers)</u>	Sorbates	NMKL 103 AOAC 983.16	Gas Chromatography (Flame ionization)	III	CXS 192	NMKL 103 withdrawn because of the use of hazardous solvent. Numeric performance criteria to be developed for 2024: these changes remain until then.

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Processed fruits and vegetables* <u>(Jams, Jellies, Marmalades, pickled cucumbers)</u>	Sorbates	NMKL 124	Liquid Chromatography <u>(UV)</u>	II	CXS 192	Sorbate falls under CXS 192 – Food Additives. Numeric performance criteria to be developed for 2024: these changes remain until then.
Processed fruits and vegetables	Tin	AOAC 980.19 (Codex general method)	AAS <u>Atomic Absorption Spectrophotometry (Flame)</u>	II	CXS 193	Relevant Codex commodity standards include CXS 62-1981, CXS 254-2007, CXS 296-2009, CXS 242-2003, CXS 297-2009, CXS 78-1981, CXS 159-1987, CXS 42-1981, CXS 60-1981, CXS 99-1981, CXS 160-1987, CXS 66-1981, CXS 13-1981, CXS 115-1981, CXS 57-1981, CXS 145-1981, CXS 98-1981, CXS 96-1981, CXS 97-1981, CXS 88-1981, CXS 89-1981. Numeric performance criteria to be developed for 2024: these changes remain until then.
Processed fruits and vegetables	Total solids	AOAC 920.151	Gravimetry	I		
Aqueous Coconut Products	Total Fats	ISO 1211 IDF 4 <u>ISO 23318 IDF 249</u>	Gravimetry (Röse-Gottlieb)	I	CXS 240	2009 CCMAS report: “Standard for Aqueous Coconut Products: <i>The Committee considered the information on the validation studies carried out on ISO 1211:1999 for total fats and ISO 6731:1989 for total solids in coconut milk and agreed to endorse both methods as Type I.</i> ”
Aqueous Coconut Products	Total solids	ISO 6731 IDF 21	Gravimetry	I	CXS 240	Validated on milk, cream, and evaporated milk

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Aqueous Coconut Products	Non-fat solids	ISO 1211 IDF 4 ISO 23318 IDF 249 and ISO 6731 IDF 21	Calculation: Gravimetry (Röse-Gottlieb) Gravimetry	I	CXS 240	2009 CCMAS report: “Standard for Aqueous Coconut Products: <i>The Committee considered the information on the validation studies carried out on ISO 1211:1999 for total fats and ISO 6731:1989 for total solids in coconut milk and agreed to endorse both methods as Type I.</i> ”
Aqueous Coconut Products	Moisture	ISO 6731 IDF 21	Calculation: Gravimetry	I	CXS 240	Validated on milk, cream, and evaporated milk
Canned Apple Sauce	Fill of glass containers	CAC/RM 46* (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables). ISO 8106	Weighing Gravimetry	I	CXS 17	CAC/RM 46 resides in the Standard for certain canned vegetables (CXS 297). CCMAS 36 (2015) agreed to replace CAC/RM 46 with ISO 8106
Canned Apple Sauce	Fill of metal containers	ISO 90-1	Weighing Gravimetry	I	CXS 17	
Canned Apple Sauce	Soluble solids (packing medium)	AOAC 932.12 ISO 2173 (Codex general method for processed fruits and vegetables)	Refractometry	I	CXS 17	These methods are not identical. Suggest retaining ISO method which contains more detailed procedures
Canned green beans and wax beans	Tough Strings	CAC/RM 39	Stretching	I	CXS 297	CAC/RM 39 currently in CXS 297 – will be moved to CXS 234. This row to be struck after method is moved.
Canned green peas	Fill of glass containers	ISO 8106	Weighing	I	CXS 297	CCPFV 24 (2008) agreed to revoke CAC/RM 45

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Canned green peas	Proper fill (in lieu of drained weight) <u>Fill of metal containers</u>	CAC/RM 45 <u>ISO 90-1</u>	Pouring and measuring <u>Gravimetry</u>	I	CXS 297	CCPFV 24 (2008) agreed to revoke CAC/RM 45
Canned green peas	Types of peas, distinguishing	CAC/RM 48	Visual inspection <u>examination</u>	I	CXS 297	CAC/RM 48 currently in CXS 297 – will be moved to CXS 234. This row to be struck after method is moved.
Canned mangoes	Syrup <u>Soluble Solids (packing medium)</u>	AOAC 932.14C	Brix spindle method <u>(refractometry)</u>	I	CXS 319	Method is “solids in syrups”
Canned mushrooms	Washed Drained weight	CAC/RM 44 <u>AOAC 968.30</u>	<u>Gravimetry</u> (Sieving)	I	CXS 297	CCPFV25 (2010) revoked CXS 55 (Standard for canned mushrooms) containing CAC/RM 44. Annex on mushrooms now included in CXS 297, containing provision for drained weight. Suggest replacing CAC/RM 44 with AOAC 968.30
Canned palmito	Mineral impurities	ISO 762	Gravimetry	I	CXS 297	
Canned Stone Fruits	Drained weight	AOAC 968.30 ISO:2173	Gravimetry <u>(sieving)</u>	I	CXS 242	ISO 2173 is a method for soluble solids, not drained weight. Wrong provision
Canned Stone Fruits	Soluble solids <u>(packing medium)</u>	AOAC 932.14C <u>ISO 2173</u>	Refractometry	I	CXS 242	Methods are not identical. Suggest retaining ISO method which contains more detailed procedures

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Canned strawberries	Calcium	AOAC 968.31	Complexometric titrimetry	II	CXS 62	Validated for canned tomatoes, lima beans, potatoes. Numeric performance criteria to be developed for 2024: these changes remain until then.
Canned strawberries	Mineral impurities	AOAC 971.33 <u>ISO 762</u>	Gravimetry	I	CXS 62	AOAC 971.33 is acid-insoluble residue. Recommend replacing with ISO 762
Certain canned citrus fruits	Calcium	NMKL 153	<u>Atomic Absorption Spectrophotometry (Flame)</u>	II	CXS 254	Calcium firming agents listed in CXS 192 – food additives. Numeric performance criteria to be developed for 2024: these changes remain until then.
Certain canned citrus fruits	Calcium	AOAC 968.31	Complexometry Titrimetry	III	CXS 254	Calcium firming agents listed in CXS 192 – food additives. Numeric performance criteria to be developed for 2024: these changes remain until then.
Certain Canned Vegetables (palmito)	Mineral impurities (sand)	AOAC 971.33 ISO 762	Gravimetry	I		Methods are not identical and AOAC 971.33 is acid-insoluble residue. Mineral impurities in canned palmito already listed above.
Citrus marmalade	Calcium	AOAC 968.31	Complexometric titrimetry	II	CXS 296	Calcium firming agents listed in CXS 192 – food additives. Numeric performance criteria to be developed for 2024: these changes remain until then.
Dates	Identification of defects	Described in the Standard <u>CXS 143</u>	Visual inspection <u>examination</u>	I	CXS 143	Method in CXS143 will be moved to CXS 234
Dates	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I	CXS 143	

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Desiccated coconut	Total acidity of the extracted oil	ISO 660 or AOCS Cd 3d-63 <u>ISO 660 / AOCS Cd 3d-63</u>	Potenciometry / Titrimetry	I	CXS 177	Changed method format to maintain consistency with previous decisions, e.g. named vegetable oils
Desiccated coconut	Ash	AOAC 950.49	Gravimetry (<u>Ashing</u>)	I	CXS 177	
Desiccated coconut	Extraneous vegetable matter	Described in the Standard <u>CXS 177</u>	Counting extraneous material with the naked eye	IV	CXS 177	Method in CXS177 will be moved to CXS 234
Desiccated coconut	Moisture	AOAC 925.40	Gravimetry (loss on drying)	I	CXS 177	
Desiccated coconut	Oil content	AOAC 948.22	Gravimetry	I	CXS 177	Titled "Fat (Crude)" in method title
Dried apricots	Identification of defects	Described in the Standard	Visual inspection (weighing)	I	CXS 130	n.b. CCPFV29 (2020) forwarded proposed draft standard for dried fruits to CAC43 at Step 5/8. CAC43 adopted this Standard, pending certain endorsements. This Standard once published will supersede CXS 130. Method to be moved to CXS234
Dried apricots	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I	CXS 130	
Dried apricots	Sulphur dioxide	AOAC 963.20	Colorimetry	II	CXS 130	
Jams (fruit preserves) and jellies <u>Jams, Jellies, and Marmalades</u>	Fill of <u>Glass</u> Containers	CAC/RM 46 <u>ISO 8106</u>	Weighing <u>Gravimetry</u>	I	CXS 296	CCMAS 36 (2015) agreed to replace CAC/RM 46 with ISO 8106
Jams (fruit preserves) and jellies <u>Jams, Jellies, and Marmalades</u>	Soluble solids	ISO 2173 AOAC 932.12	Refractometry	I	CXS 296	Methods are not identical. Suggest retaining ISO method which contains more detailed procedures
Mango chutney	Ash insoluble in HCl	ISO 763	Gravimetry	I	CXS 160	

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Pickled cucumbers	Acidity, total	AOAC 942.15	Titrimetry	I	CXS 115	
Pickled cucumbers	Drained weight	AOAC 968.30	Gravimetry	I	CXS 115	
Pickled cucumbers	Mineral impurities	AOAC 971.33 ISO 762	Gravimetry	I	CXS 115	AOAC 971.33 is acid-insoluble residue. Recommend replacing with ISO 762
Pickled cucumbers	Salt in brine (NaCl)	AOAC 971.27 (Codex general method)	Potentiometry	II	CXS 115	
Pickled cucumbers	Volume fill by displacement	Described in the Standard	Displacement	I	CXS 115	
Preserved tomatoes	Calcium	AOAC 968.31	Complexometric titrimetry	III	CXS 13	Calcium firming agents listed in CXS 192 – food additives. Numeric performance criteria to be developed for 2024: these changes remain until then.
Preserved tomatoes	Calcium	NMKL 153	Atomic Absorption Spectrophotometry (Flame)	II		Calcium firming agents listed in CXS 192 – food additives. Numeric performance criteria to be developed for 2024: these changes remain until then.
Preserved tomatoes	Minimum Drained Weight	AOAC 968.30	Gravimetry (sieving) note: Use a No. 14 screen instead of '7/16' or No. 8	I	CXS 13	
Preserved tomatoes	Mould count	AOAC 965.41	Howard mould count	I	CXS 13	Mould count for preserved tomatoes to be set according to the legislation of the country of retail sale
Processed tomato concentrates	Lactic acid	EN 2631 EN 12631	Spectrometry (Enzymatic determination)	II	CXS 57	Should be EN 12631. EN 2631 is "Evaluation of human exposure to whole-body vibration"

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Processed tomato concentrates	Mineral impurities (sand)	AOAC 971.33 <u>ISO 762</u>	Gravimetry	IV I	CXS 57	AOAC 971.33 is acid-insoluble residue. Recommend replacing with ISO 762
Processed tomato concentrates	Mould count	AOAC 965.41	Howard mould count	I	CXS 57	Mould count for processed tomato concentrates to be set according to the legislation of the country of retail sale.
Processed tomato concentrates	Natural tomato soluble solids	AOAC 970.59	Refractometry	↓		Redundant of “Tomato soluble solids” below
Processed tomato concentrates	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	II	CXS 57	
Processed tomato concentrates	Tomato soluble solids	AOAC 970.59	Refractometry	I	CXS 57	
Raisins	Mineral impurities	CAC/RM 51 <u>ISO 762</u>	<u>Gravimetry</u> (Ashing)	I	CXS 67	CCPFV29 (2020) forwarded proposed draft Standard for dried fruits to CAC43 at Step 5/8. CAC43 adopted the Standard, pending certain endorsements. This Standard once published will supersede CXS 67. Recommend replacing with ISO 762
Raisins	Mineral oil	CAC/RM 52	Extraction and separation on alumina	II	CXS 67	Cannot find CAC/RM 51 or 52 in CXS 67. CXS 67 will be superseded by the Standard for dried fruits once it is published. Retain until new standard is published?
Raisins	Moisture	AOAC 972.20	Electrical conductance	I	CXS 67	
Raisins	Sorbitol	AOAC 973.28	Gas chromatography (<u>flame ionization</u>)	II	CXS 67	Numeric performance criteria to be developed for 2024: these changes remain until then.
Raisins	Sulphur dioxide	AOAC 963.20	Colorimetry	II	CXS 67	

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Table olives	Drained weight	AOAC 968.30 (Codex general method for processed fruits and vegetables)	Sieving Gravimetry (<u>sieving</u>)	I	CXS 66	
Table olives	Fill of <u>glass</u> containers	CAC/RM 46* (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables) <u>ISO 8106</u>	Weighing <u>Gravimetry</u>	I	CXS 66	CCMAS 36 (2015) agreed to replace CAC/RM 46 with ISO 8106
<u>Table olives</u>	<u>Fill of metal containers</u>	<u>ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)</u>	Weighing <u>Gravimetry</u>	I	CXS 66	
Table olives	pH of brine	NMKL 179 (Codex general method for processed fruits and vegetables)	Potentiometry	II	CXS 66	
Table olives	pH of brine	AOAC 981.12 (Codex general method for processed fruits and vegetables)	Potentiometry	III	CXS 66	
Table olives	pH of brine	ISO 1842	Potentiometry	IV	CXS 66	
Table olives	Salt in brine	AOAC 971.27 NMKL 178 (Codex general method)	Potentiometry	II	CXS 66	

Processed Fruits and Vegetables – Group 1						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Table olives	Lead	AOAC 999.11 NMKL 139 (Codex general method)	<u>Atomic Absorption Spectrophotometry (Flame)</u> AAS (Flame absorption)	II	CXS 66	Pb in table olives is covered in CXS 193. Numeric performance criteria were developed at CCMAS42 (2023); this row to be struck and replaced with the numeric criteria.
Table olives	Tin	NMKL 190 EN 15764	<u>Atomic Absorption Spectrophotometry (Flame)</u> AAS	II	CXS 66	Numeric performance criteria to be developed for 2024: these changes remain until then.

***FOOTNOTE:**

The processed fruit & vegetable commodities listed in parenthesis suggests those where the relevant provision is either an allowed food additive, or 'applies to' by a commodity standard. This is not an exhaustive list; and does not necessarily represent specific commodities included in method validation.

Appendix XII: Group 2 Items for future consideration

Processed Fruits and Vegetables						
Commodity	Provision	Method	Principle	Type	Standard	Comments
Processed fruits and vegetables	Tin	AOAC 980.19 (Codex general method)	AAS <u>Atomic Absorption Spectrophotometry (Flame)</u>	II	CXS 193	Relevant Codex commodity standards include CXS 62-1981, CXS 254-2007, CXS 296-2009, CXS 242-2003, CXS 297-2009, CXS 78-1981, CXS 159-1987, CXS 42-1981, CXS 60-1981, CXS 99-1981, CXS 160-1987, CXS 66-1981, CXS 13-1981, CXS 115-1981, CXS 57-1981, CXS 145-1981, CXS 98-1981, CXS 96-1981, CXS 97-1981, CXS 88-1981, CXS 89-1981. Numeric performance criteria to be developed for 2024: these changes remain until then.
Raisins	Mineral oil	CAC/RM 52	Extraction and separation on alumina	II	CXS 67	Cannot find CAC/RM 51 or 52 in CXS 67. CXS 67 will be superseded by the Standard for dried fruits once it is published. Retain until new standard is published?