

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
United Nations



World Health
Organization

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Agenda Item 2, 3 and 4

MAS/41 CRD/02

ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS SAMPLING

41st Session
17 - 21 May 2021, virtual

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS AND SAMPLING PLANS IN CODEX STANDARDS

The report of PWG on Endorsement of Method of Analysis and Sampling

Agenda Item 2 - Matters Referred to the Committee by the Codex Alimentarius Commission and Other Subsidiary Bodies (CX/MAS 21/41/2)

CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU41)

Microbiological methods¹

The Working Group recommended retention in CXS 234 of the microbiological methods for nicotinamide, niacin, pantothenic acid, pyridoxine, and cobalamin.

The Working Group noted the method for Vitamin D (AOAC 936.14) is not microbiological, but biological (rat bioassay) and scheduled for removal from AOAC Int. *Official Methods of Analysis (OMA)*, and recommended revocation of the method and removal from CXS 234.

Recommendations:

- Retain the methods for nicotinamide, niacin, pantothenic acid, pyridoxine, and cobalamin in CXS 234;
- Revoke AOAC 936.14 for determination of Vitamin D

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

After response from CCNFSDU, The Working Group, recommended the replacement of AOAC 999.15 / EN 14148 for Vitamin K with AOAC 2015.09 / ISO 21446 as Type II. (see Appendix I)

Performance criteria for Type III methods for determination of nine minerals in CXS 72-1981²

The Working Group noted the development of numeric criteria would not solely apply to Type III methods, but also remove the designation of Type II methods. Based on the reply from CCNFSDU, which stated CCNFSDU prefers to identify specific methods for dispute settlement, the Working Group recommended to maintain the typing of methods.

The Working Group noted that based on the decision made by the Committee at CCMAS40 (REP19/MAS, para 10) AOAC 2011.14 / ISO 15151 | IDF 229 will be listed in CXS 234 as Type III.

Recommendation:

- inform CCNSDU accordingly

Methods to measure sweetness in Drink/Product for young children with added nutrients / Drink for young children³

¹ REP20/NFSDU, paras. 198-199

² REP20/NFSDU, para 9

³ REP20/NFSDU, para 49

The Working Group noted that there were no known validated instrumental analytical methods to measure sweetness of carbohydrate sources and therefore no way to determine compliance for such a provision.

Recommendation:

- inform CCNFSDU accordingly

Dietary fibre: Applicable to the Guidelines for Use of Nutrition and Health Claims (CXG 23-1997): Table of Conditions for Claims⁴

The Working Group recommended the endorsement of ICC Standard No. 185 / AOAC 2017.16 as a Type I method to replace AOAC 2009.01 / AACC Intl 32-45.01 (Appendix I).

Recommendation:

- Endorse ICC Standard No. 185 / AOAC 2017.16; and
- Revoke AOAC 2009.01 / AACC Intl 32-45.01.

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA21)

Methods of analysis for laver products (CXS 323R-2017)

The working group reviewed the information provided on the performance of the methods and noted the study represents an extension to a new matrix of a previously validated and therefore does not require 8 or more labs for a full collaborative study.

Recommendations:

- Endorse the methods for acidity and moisture (Appendix II)

Agenda Item 3 - Endorsement of Methods of Analysis and Sampling Plans for Provisions in Codex Standards (CX/MAS 21/41/3)

CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU41)⁵

Methods of analysis for provisions in the Standard for infant formula and formulas for special medical purposes intended for infants (CXS 72-1981)

The Working Group recommended endorsement of methods, as Type II, for thiamine, riboflavin, niacin, Vitamin B₆, choline, and carnitine. Methods previously listed in CXS 234 were retyped to Type III.

The Working Group recommended the endorsement of a method for biotin, which is identical to the existing Type II method. (Appendix I)

The Working Group did not recommend the endorsement of methods for fructans, beta carotene, and lycopene. These were not endorsed because there are no provisions for these analytes in CXS 72-1981 (Appendix I). It was noted that methods for fructans (All foods) are listed in CXS 234 as part of the fibre methods. The methods currently listed in CXS 234 are not sensitive enough to be used for the analysis of fructans in infant formula. There was discussion of adding a footnote to the fibre table, noting these methods were not appropriate for infant formula and then endorsing the proposed method. In the end the Working Group agreed, because of the lack of provision in CXS 72-1981, the fructan method would not be endorsed.

The Working Group recommended the methods be returned to CCNFSDU and along with a reminder that CCMAS40 (Rep 19/MAS para 39-41) had agreed during the development of the information document (CCMAS_INF) and in accordance with the Procedural Manual that *“All proposed methods of analysis must have direct pertinence to the Codex Standard to which they are directed.”*

The Working Group noted the use of ingredients in infant formulas, which are not captured in CXS 72-1981, the desire for accurate analytical methods to determine the concentration of these ingredients. The Working Group recommends CCMAS invite CCNFSDU to work with his committee to identify potential solutions.

Recommendations:

- Endorse as Type II, for thiamine, riboflavin, niacin, Vitamin B₆, choline, carnitine, and biotin (Appendix I);
- Retype Methods previously listed in CXS 234 were retyped to Type III (Appendix I);

⁴ REP20/NFSDU, para 196

⁵ REP20/NFSDU, para. 197

- Inform CCNFSDU that:
 - the methods for fructans, beta-carotene and lycopene were not endorsed as there were no accompanying provisions in CXS 72-1981, however CCMAS would appreciate any rationale from CCNFSDU which could further inform this decision;
 - “All proposed methods of analysis must have direct pertinence to the Codex Standard to which they are directed.”

FAO/WHO COORDINATING COMMITTEE FOR AFRICA (CCAFRICA23)⁶

Methods of analysis for provisions in the draft standard for dried meat

The Working Group recommended endorsement of the method for water activity and determination of ash content (Appendix III).

The Working Group did not recommend endorsement of the remaining methods but did recommend the methods be returned to CCAFRICA and for CCAFRICA consider the following comments and address the following questions (Appendix III)

Only one method can be endorsed for moisture. Is there a preference among CCAFRICA for the AOAC 950.46B or ISO 1442 method?

ISO 1443 is for the determination of total fat and AOAC 960.39 is for the determination of crude fat. CCAFRICA should consider if the determination crude fat or the determination of total fat is the correct provision.

For the determination of crude protein, a conversion factor must be used to convert the nitrogen results measured by the method to a crude protein value. The Working Group identified a conversion factor of 6.25, however CCAFRICA should consider if this is the correct value? Additionally only one method can be endorsed for the determination of crude protein, CCAFRICA should consider which method AOAC 928.08 or ISO 937 is preferred?

There were multiple methods identified for the determination of chloride. One methods can be endorsed as Type II, which would be the method used for disputes and the remaining methods would be listed as Type III. CCAFRICA should consider which method ISO 1841-1 or ISO 1841-2 or AOAC 935.47 and 937.09B is preferred as the Type II method?

Recommendations:

- Endorse the methods for water activity and determination of ash content (Appendix III).; and
- The methods be returned to CCAFRICA Appendix II and for CCAFRICA consider the comments and address the questions above.

FAO/WHO COORDINATING COMMITTEE FOR NORTH AMERICA AND SOUTH WEST PACIFIC (CCNASWP15)

Methods of analysis for provisions in the draft regional standard for fermented noni fruit juice

The Working Group recommended (APPENDIX IV)

- Endorsement of methods for brix value, adding a number of identical methods and adding “soluble solids” to the provision.
- Removal of NMKL 179 for the determination of pH, citing that the validated range (3.6 to 6.9) did not meet the pH necessary for the analysis of noni juice (3.5).
- Replacement of NMKL 179, with AOAC 981.12 as a Type II method for pH.
- Endorsement of 3 methods for the determination of ethanol, all as Type IV. For IFUMA 52 there were and concerns about lack of validation in fermented juices and interferences from other alcohols. For the AOAC methods, there is lack of collaborative study data.

The Working Group did not recommend endorsement of the methods for analysis of fermented non fruit juice

The Working Group did not recommended endorsement of the methods for the identification of scopoletin and deacetylasperulosidic acid. The Working Group recommended changes be made to the methods to give clear indication of the solid phase extraction separation mode needed.

Recommendations:

⁶ REP20/AFRICA, para. 102 ii) and Appendix V

- Endorse them methods for as presented in appendix IV;
- Request CCNASWP to provide clarification on the issue identified above

Methods of analysis for provisions in the regional standard for kava products for use as a beverage when mixed with water

The Working Group recommended endorsement of the method for moisture as AOAC 925.45.

The Working Group did not recommend the endorsement of the methods for Noble Kava Varieties and Flavokavin. The Working Group noted that review of the references did not produce a clear procedure for determining kava lactones or flavokavins. It appeared there were different sections within each reference that needed to be followed and that the 2016 reference may not be required for flavokavins. The work group recommends that CCMAS request CCNASWP consider producing a single stepwise method or SOP which captures the necessary steps for each provision in one easy to follow document.

Recommendations:

- Endorse AOAC 925.45 (Appendix IV);
- Request CCNASWP consider producing a single stepwise method or SOP which captures the necessary steps for each provision in one easy to follow document.

FAO/WHO COORDINATING COMMITTEE FOR NEAR EAST (CCNE10)⁷

Methods of analysis for provisions in the draft regional standard for mixed zaatar

The Working Group recommended endorsement of Type I methods for sodium chloride, moisture, acid-insoluble ash, extraneous matter, and foreign matter.

The Working Group also recommended Type IV methods for insects/insect fragments, mould damage, mammalian excreta (whole), and mammalian excreta (ground) (Appendix V).

For the sodium chloride, and acid insoluble ash methods the Working Group noted that these are on a dry weight basis and therefore require a moisture determination in order to generate a result which meets the provision in the regional standard. The Working Group recommends the same format used for the dairy methods be followed when listing these methods.

Recommendations:

- Endorse the methods as presented in Appendix V

COMMITTEE ON PROCESSED FRUITS AND VEGETABLES (CCPFV29)

Methods of analysis for provisions in the Standard for Gochujang

The Working Group recommended (Appendix VI):

- removal of AOAC 995.03 because the method reports results in scoville units and not concentration of capsaicin as appears in the Standard for Gochujang.
- removal of the gas chromatography method from the Annex
- endorsement of J AOAC method as Type IV, which contains both the HPLC and GC methods, with both reporting in concentration of capsaicin.
- Endorsement of AOAC 984.13 for crude protein, using the conversion factor 6.25, and AOAC 945.43 as Type I to replace AOAC 934.01 for moisture.

Methods of analysis for provisions in the Standard for Chili sauce

The Working Group recommended endorsement of the methods for pH, and fill of containers (CAC/RM46), noting that CAC/RM46 may need to be moved to annex and that this is for glass containers (Appendix VI).

Methods of analysis for provision for the General Standard for Dried Fruits

The Working Group recommended the endorsement of methods for identification of defects and moisture as Type I (Appendix VI).

Sampling Plans for Gochujang, Chili Sauce, Mango Chutney and Dried Fruit

⁷ REP20/NE, para. 87 (ii) and Appendix IV

The Working Group noted the plans presented in CX/MAS 21/41/3 were from an old Codex Standard CXS 233 which was replaced by The General Guidelines on Sampling (GL50). However, the plans were never updated and are not considered appropriate.

The Working Group also noted the on-going work by CCMAS, led by New Zealand, to update GL 50 and the comments and questions related to sampling plans detailed on CRD 7. New Zealand noted that CRD7 represents hypothetical case and additional information would be needed to develop a sampling plan for these commodities.

The Secretariat reminded the Working Group that CCPFV has been adjourned and any information needed to develop a sampling plan would need to be collected in a call for information.

The Working Group did not recommend endorsement of the sampling plans and asked participants to review CRD 7 and to consider possible options for collecting information and developing a plan in line with the update of GL50.

Recommendation

- Endorse methods of analysis for gochujang, chili sauce, dried fruits
- For sampling plans Circulate the proposals in CRD7 for comment and consideration by next session

Agenda Item 3 - Endorsement of Methods of Analysis and Sampling Plans for Provisions in Codex Standards – Addendum 1 (CX/MAS 21/41/3 Add.1)

COMMITTEE ON SPICES AND CULINARY HERBS. (CCSCH5)

Methods of analysis for provisions in the draft standard for dried oregano

The Working Group recommended endorsement of the methods listed for dried basil (Appendix VII). Further discussion of the additional methods from CCSCH was postponed to allow participants to review CX/MAS 21/41/3 Add.1 and to allow time for the discussion of the Dairy Package and the Fats & Oils Package.

Recommendations:

- Endorse the methods presented in Appendix VI; and
- Consider the rest of the methods from CCSCH at the next session of CCMAS.

Agenda Item 4 - Review of methods of analysis in CXS 234: Dairy workable package (CX/MAS 21/41/4)

The Working Group began discussion of the Dairy Workable Package (CX/MAS 21/41/4) by reviewing Methods Not Previously Reviewed (CX/MAS 21/41/4 App II).

The Working Group recommended endorsement of the methods (Appendix VIII).

The Working Group noted that for some methods the provision in the commodity standard did not match the description of the method (e.g. milk fat versus total fat).

The Working Group further recommended that to be consistent with previous decisions, CXS 234 should list the provision which is in the standard. Additionally, the method description should also be listed to avoid confusion. This approach is captured for consideration in Appendix VIII (e.g. Milk Fat (Total Fat)).

The Working Group did not endorse a method for Ash of dairy permeates, but 2 methods are listed for consideration in Appendix VII. As these methods are Type I, only one can be included in CXS 234.

Recommendations:

- Endorse the methods as presented in Appendix VIII; and
- Determine which method for ash should be introduced as the Type I method in CXS 234

Agenda Item 4 - Review of methods of analysis in CXS 234: Fats and oils workable package (CX/MAS 21/41/5)

The Netherlands, Chair of the Electronic Working Group (EWG) for the Fats & Oils Package reviewed the process used by the electronic working group and presented the methods and comments contained in CX/MAS 21/41/5. Given the short time a complete line by line review was not possible, and participants were asked to further review the document and provide comment and feedback where necessary.

The working group noted the line formatting and terminology used by the Dairy Package and recommended that same approach for the Fats and Oils Package.

Note: the fats and oils package will be considered under Agenda item 4.2

APPENDIX I

COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU41)***Dietary fibre: Applicable to the Guidelines for Use of Nutrition and Health Claims (CXG 23-1997):
Table of Conditions for Claims***

All Foods (1)	Method applicable for determining the content of dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches	AOAC 2009.01 / AACC Int# 32- 45.01 <u>ICC Standard No.185 / AOAC 2017.16</u>	Enzymatic-Gravimetry High Pressure Liquid Chromatography	Type I
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Methods of analysis for infant formula

Recommended for Endorsement (Underline = Additions to CXS 234, Strikethrough = removal from CXS 234)

Commodity	Provision	Method	Principle	Proposed Type
Infant Formula	Thiamine	<u>AOAC 2015.14 / ISO 21470</u>	<u>Enzymatic digestion and UHPLC-MS/MS</u>	<u>II</u>
		EN 14122	HPLC with pre- or post-column derivatization to thiochrom	# <u>III</u>
		AOAC 986.27	Fluorimetry	III
	Riboflavin	<u>AOAC 2015.14 / ISO 21470</u>	<u>Enzymatic digestion and UHPLC-MS/MS</u>	<u>II</u>
		EN 14152	HPLC	# <u>III</u>
		AOAC 985.31	Fluorimetry	III
	Niacin	<u>AOAC 2015.14 / ISO 21470</u>	<u>Enzymatic digestion and UHPLC-MS/MS</u>	<u>II</u>
		EN 15652	HPLC	# <u>III</u>
		AOAC 985.34	Microbioassay and turbidimetry	III

	Vitamin B ₆	<u>AOAC 2015.14 / ISO 21470</u>	<u>Enzymatic digestion and UHPLC-MS/MS</u>	II
		AOAC 2004.07 / EN 14164	HPLC	II III
		AOAC 985.32	Microbioassay	III
		EN 14166	Microbioassay	III
	Choline	<u>AOAC 2015.10 / ISO 21468</u>	<u>UHPLC-MS/MS</u>	II
		AOAC 999.14	Enzymatic Colorimetric Method with limitations on applicability due to choline and ascorbate concentration	II III
	<u>Carnitine</u>	<u>AOAC 2015.10 / ISO 21468</u>	<u>UHPLC-MS/MS</u>	II
	Biotin	AOAC 2016.02 / <u>ISO 23305</u>	HPLC-UV	II
		EN 15607	HPLC-fluorescence	III

Not Recommended for Endorsement

Commodity	Provision	Method	Principle	Proposed Type
Infant Formula	Fructans	AOAC 2016.14 / ISO DIS 22579 IDF 241	Enzymatic digestion with HPAEC-PAD	II
	Beta Carotene	AOAC 2016.13 / ISO 23443	UHPLC-	II
	Lycopene	AOAC 2016.13 / ISO DIS 23443	UHPLC-UV	II

APPENDIX II

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA21)

Methods of analysis for laver products (CXS 323R-2017)

<u>Laver products</u>	<u>Acidity: acid value for the extracted oil</u>	<u>ASIA21-CRD2(oil extn)/ and ISO 660 AOCS Cd 3d-63</u>	<u>Extraction of oil</u> <u>Titrimetry</u>	!
<u>Laver Products</u>	<u>Moisture Content</u>	<u>AOAC 925.45</u>	<u>Gravimetry, drying at atmospheric pressure</u>	!

Appendix III**FAO/WHO COORDINATING COMMITTEE FOR AFRICA (CCAFRICA23)⁸****Methods of analysis for provisions in the draft standard for dried meat**

Recommend for Endorsement

Method	Provision	Principle	Type
<u>ISO 936</u>	<u>Determination of Ash Content</u>	<u>Gravimetry</u>	I
<u>ISO 18787</u>	<u>Determination of Water Activity</u>	<u>Electrometry</u>	II

Recommended for return to CCAFRICA

Method	Provision	Principle	Type
AOAC 950.46B	Determination of Moisture Content	Gravimetry	I
ISO 1442	Determination of Moisture Content	Gravimetry	I
ISO 1443	Determination of Total Fat	Gravimetry	I
AOAC 960.39	Determination of Crude Fat	Gravimetry	I
AOAC 928.08	Determination of Crude Protein (Nitrogen x conversion factor 6.25?)	Kjeldahl	I
ISO 937	Determination of Crude Protein (Nitrogen x conversion factor 6.25?)	Kjeldahl	I
ISO 1841-1	Determination of chloride (expressed as sodium chloride-edible Salt)	Volhard method	II or III
ISO 1841-2	Determination of chloride (expressed as sodium chloride-edible Salt)	Potentiometric	II or III
AOAC 935.47 and 937.09 B	Determination of chloride (expressed as sodium chloride-edible Salt)	Volhard method	II or III

⁸ REP20/AFRICA, para. 102 ii) and Appendix V

APPENDIX IV

FAO/WHO COORDINATING COMMITTEE FOR NORTH AMERICA AND SOUTH WEST PACIFIC
(CCNASWP15)

Methods of analysis for provisions in the draft regional standard for fermented noni fruit juice

Provision	Method	Principle	Type	Notes
<u>Brix Values (Soluble solids)</u>	AOAC 983.17 <u>/ EN 12142 /</u> <u>IFUMA 8 /</u> <u>ISO 2173</u>	Refractometry	I	Adopted for fruit juices and nectars
pH value	NMKL 179	Potentiometry	II	Adopted for fruit juices and nectars
<u>pH Value</u>	<u>AOAC 981.12</u>	<u>Potentiometry</u>	<u>II</u>	
Ethanol	IFUMA 52	Enzymatic determination	II <u>IV</u>	Adopted for fruit juices and nectars
<u>Ethanol</u>	<u>AOAC</u> <u>2016.12</u>	<u>Headspace GC-FID</u>	<u>IV</u>	
<u>Ethanol</u>	<u>AOAC</u> <u>2017.07</u>	<u>Enzymatic</u> <u>Determination</u>	<u>IV</u>	
Identification of scopoletin	Annex A*	Thin layer chromatography	IV	
Identification of deacetylasperulosidic acid	Annex B*	Thin layer chromatography	IV	

* *In compliance with the general criteria for testing laboratories laid down in ISO/IEC Guide 17025:2017*

Methods of analysis for provisions in the regional standard for kava products for use as a beverage when mixed with water for consideration by CCNASWP

Provision	Method	Principle	Type
Noble kava varieties (total kava lactones)	<p>Lebot V, Legendre L (2016), Comparison of kava (Piper methysticum Forst.) varieties by UV absorbance of acetononic extracts and high-performance thin-layer chromatography. Journal of Food Composition and Analysis 48:25-33. http://dx.doi.org/10.1016/j.jfca.2016.01.009</p> <p><u>Section 2.3 for UV Absorbance</u></p> <p>and</p> <p>Lebot V, Michalet S, Legendre L. (2019). Kavalactones and flavokavins profiles contribute to quality assessment of kava (Piper methysticumG.Forst.), the traditional beverage of the Pacific. Beverages 2019, 5, 34; https://doi.org/10.3390/beverages5020034</p> <p><u>Sections 2.2, 2.3, and 3.1 for procedures</u></p>	<p>High performance thin layer chromatography</p> <p>and/or UV absorbance of acetononic extracts measured at 440 nm (less or equal to 0.9)</p>	IV
Moisture	<p><u>The Fiji Kava Standard 2017</u>. Section 8.1 AOAC 925.45</p>	Gravimetry	I
[Flavokavins	<p>Lebot V, Legendre L (2016), Comparison of kava (Piper methysticumForst.) varieties by UV absorbance of acetononic extracts and high-performance thin-layer chromatography. Journal of Food Composition and Analysis 48:25-33. http://dx.doi.org/10.1016/j.jfca.2016.01.009</p> <p>and</p> <p>Lebot V, Michalet S, Legendre L. (2019). Kavalactones and flavokavins profiles contribute to quality assessment of kava (Piper methysticumG.Forst.), the traditional beverage of the Pacific. Beverages 2019, 5, 34; https://doi.org/10.3390/beverages5020034</p>	<p>High performance thin layer chromatography</p> <p>and/or UV absorbance of acetononic extracts measured at 440 nm (less or equal to 0.9)]</p>	IV

APPENDIX V

FAO/WHO COORDINATING COMMITTEE FOR NEAR EAST (CCNE10)**Methods of analysis for provisions in the draft regional standard for mixed zaatar**

Provision	Method	Principle	Type*
Sodium chloride (dry weight basis)	AOAC 960.29 <u>ISO 939 and</u> <u>AOAC 971.27</u>	<u>Calculation by moisture and ash</u> <u>Distillation and Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)</u>	I
Moisture	AOAC 925.10	Gravimetry, drying at 130°C	
<u>Moisture</u>	<u>ISO 939</u>	<u>Distillation</u>	<u>I</u>
Acid-insoluble ash (dry weight basis)	<u>ISO 939 and</u> <u>ISO 930</u> <u>AOAC 941.12 (corrected for moisture by ISO 939)</u>	<u>Calculation by moisture and ash</u> <u>Distillation and Gravimetry, Furnace, 550°C (for the HCl insoluble ignited residue)</u>	I
Extraneous Matter	ISO 927	<u>Visual Examination</u> <u>Gravimetry, followed by Volumetry</u>	I
Foreign Matter	ISO 927	<u>Visual Examination,</u> <u>Gravimetry followed by Volumetry</u>	I
Insects/ Excreta /Insect Fragments	ISO 927 Method appropriate for particular spice from AOAC Chapter 16, subchapter 14 [ISPM 08 Determination of Pest Status in an area]	Visual Examination	IV
<u>Insect/Insect Fragments</u>	<u>AOAC 969.44</u>	<u>Visual Examination</u>	<u>IV</u>
<u>Insect/Insect Fragments</u>	<u>AOAC 975.49</u>	<u>Visual Examination</u>	<u>IV</u>
Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA, Technical Bulletin Number 5)	Visual examination (for whole)	IV
Mammalian Excreta Mammalian, Excreta-Other	Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole) <u>AOAC 993.27 (For Ground)</u>	Visual Examination Enzymatic Detection Method	IV IV
<u>Mammalian Excreta</u> <u>Other</u>	<u>AOAC 993.27 (For Ground)</u>	<u>Enzymatic Detection Method</u>	<u>IV</u>

APPENDIX VI

COMMITTEE ON PROCESSED FRUITS AND VEGETABLES (CCPFV29)***Methods of analysis for provisions in the Standard for Gochujang***

Provision	Method	Principle	Type
Capsaicin	<u>AOAC 995.03</u> <u>Journal of AOAC International</u> Vol. 91. No. 2, 2008. pp 387-391	<u>HPLC-Fluorescence</u>	<u>II-IV</u>
Capsaicin	Described in the Standard (Annex I) <u>Journal of AOAC International</u> Vol. 91. No. 2, 2008. Pp 387-391	<u>Gas chromatography-FID</u>	IV
Crude protein	AOAC 984.13 (Nitrogen conversion factor: 6.25)	<u>Titrimetry</u> , Kjeldahl	I
Moisture	AOAC 934.01 ($\leq 70^{\circ}\text{C}$, $\leq 50\text{ mm Hg}$)	<u>Gravimetry</u>	I
Moisture	<u>AOAC 945.43</u>	<u>Gravimetry</u>	I

Methods of analysis for provisions in the Standard for Chili sauce

Commodity	Provision	Method	Principle	Type
Chili Sauce	pH	NMKL 179 (general method)	Potentiometry	II
Chili Sauce	pH	AOAC 981.12	Potentiometry	III
Chili Sauce	Fill of Containers	CAC/ RM 46	Weighing	I

Methods of analysis for provision for the General Standard for Dried Fruits

Commodity	Provision	Method	Principle	Type
Dried fruits	Identification of defects	Described in the standard	Visual inspection	I
Dried fruits (except prunes and raisins)	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I
Dried fruits (prunes and raisins)	Moisture	<u>AOAC 972.20</u>	<u>Electrical Conductance</u>	I

COMMITTEE ON SPICES AND CULINARY HERBS. (CCSCH5)**Methods of analysis for provisions in the draft standard for dried oregano**

Provision	Method	Principle	Type
Moisture	ISO 939	Distillation	I
Total ash (<u>dry weight basis</u>)	ISO 939 and ISO 928	<u>Calculation from moisture and ash</u> Distillation and Gravimetry	I
Acid-insoluble ash (<u>dry weight basis</u>)	ISO 939 and ISO 930	<u>Calculation from moisture and ash</u> Distillation and Gravimetry	I
Volatile oils (<u>dry weight basis</u>)	ISO 939 and ISO 6571	<u>Calculation from moisture and volatile oils</u> Distillation and Distillation	I
Extraneous matter	ISO 927	Visual <u>inspection examination</u> followed by Gravimetry	I
Foreign matter	ISO 927	Visual <u>inspection examination</u> followed by Gravimetry	I
Mammalian excreta Other excreta	Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (For whole) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32	Visual examination	IV
Whole dead insect	ISO 927	Visual examination	IV
	MPM V-8 Spices, Condiments, Flavours 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#v32		
Mould visible	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavors-and-crude-drugs#v32	Visual examination	IV
Insect Damage	ISO 927	Visual Examination	I

Appendix VIII

METHODS NOT PREVIOUSLY REVIEWED

Methods recommended for endorsement

Commodity	Provision	Method	Principle	Type
Milk and Milk Products	Melamine	ISO/TS 15495 IDF/RM 230	LC-MS/MS	IV
<u>Milk and Milk Products</u>	<u>Melamine</u>	<u>ISO DIS 23970 IDF 252</u>	<u>LC-MS/MS</u>	<u>II</u>
Butter	Milkfat	ISO 17189 IDF 194	Gravimetry Direct determination of fat using solvent extraction	I
Butter	Salt	ISO 15648 IDF 179	Potentiometry (determination of chloride, expressed as sodium chloride)	II
Butter	Water ⁹	ISO 3727-1 IDF 80-1	Gravimetry	I
Dairy fat spreads	<u>Milkfat</u> (Total Fat)	ISO 17189 IDF 194	Gravimetry Direct determination of fat using solvent extraction	I
Edible casein products	Acids, free <u>Maximum free acidity¹⁰</u>	ISO 5547 IDF 91	Titrimetry (aqueous extract)	IV I
Edible casein products	Lactose	ISO 5548 IDF 106	Photometry (phenol and H ₂ SO ₄)	IV

⁹ Water content excluding the crystallized water bound to lactose (generally known as “moisture content”)

¹⁰ suggest an editorial amendment in the description of the provision in STAN 290: to change ‘maximum free acid’ to ‘maximum free acidity’

Edible casein products	Milkfat (<u>Total Fat</u>)	ISO 5543 IDF 127	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Edible casein products	pH	ISO 5546 IDF 115	Electrometry	IV II
Emmental	Calcium ≥ 800mg/100g	ISO 8070 IDF 119	Flame atomic absorption	IV III
<u>Emmental</u>	<u>Calcium</u> ≥ 800mg/100g	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP mass spectrometry</u>	II
<u>Emmental</u>	<u>Calcium</u> ≥ 800mg/100g	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP emission spectroscopy</u>	III
Fermented milks	Dry matter (total solids) ¹¹	ISO 13580 IDF 151	Gravimetry (drying at 102 °C)	I
Fermented milks	Total acidity expressed as percentage of lactic acid	ISO/TS 11869 IDF/RM 150	Potentiometry, titration to pH 8.30	† ‡ IV
Fermented milks	Microorganisms constituting the starter culture	ISO 27205 IDF 149 (Annex A)	Colony count at 25 °C, 30 °C, 37 °C and 45 °C according to the starter organism in question	IV
Milk powders and cream powders	Scorched particles	ISO 5739 IDF 107	Visual comparison with standard disks, after filtration	IV
<u>Milk powders and cream powders</u>	<u>Scorched particles</u>	<u>ADPI Scorched Particles, 2016</u>	<u>Visual comparison with standard disks, after filtration</u>	IV

¹¹ Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose

Milk powders and cream powders	Solubility Index	ISO 8156 IDF 129	Centrifugation	I
Whey cheeses by concentration (carbohydrate contents below 5%)	Milkfat (Total Fat)	ISO 1854 IDF 59	Gravimetry (Röse Gottlieb)	I
Whey cheeses by concentration (does not dissolve completely in the ammonia, contains FFA in significant quantities or carbohydrate content >5%)	Milkfat (Total Fat)	ISO 8262-3 IDF 124-3	Gravimetry (Weibull-Berntrop)	I
Whey cheeses by concentration (for carbohydrate content under 5%)	Milk fat in dry matter (Total fat in dry matter)	ISO 1854 IDF 59 and ISO 2920 IDF 58	Calculation from fat content and dry matter content Gravimetry (Röse Gottlieb) Gravimetry, drying at 88 °C	I
Whey cheeses by concentration (does not dissolve completely in the ammonia, contains FFA in significant quantities or carbohydrate content >5%)	Milk fat in dry matter (Total fat in dry matter)	ISO 8262-3 IDF 124-3 and ISO 2920 IDF 58	Calculation from fat content and dry matter content Gravimetry (Weibull-Berntrop) Gravimetry, drying at 88 °C	I
Whey powders	Moisture, "Free"	ISO 2920 IDF 58	Gravimetry (drying at 88°C ±2°C)	IV

Methods not endorsed

Commodity	Provision	Method	Principle	Type
Dairy permeate powders Validated so would be type I	Ash	NMKL 173	Gravimetry (ashing at 550 °C)	I IV
Dairy permeate powders	Ash	AOAC 930.30	Gravimetry	I