

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
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World Health
Organization

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Agenda Item 2 and 3

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

Thirty-seventh Session

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REPORT OF THE WORKING GROUP ON THE ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING

The Working Group on the endorsement of methods of analysis and sampling was held on Saturday 21 February 2016, prior to the 37th Session of the Committee on Methods of Analysis and Sampling. The Working Group was chaired by Dr Roger Wood (ICUMSA) and co-chaired by Dr Gregory Noonan (USA) with Hilde Norli (NMKL) as rapporteur. The WG considered the methods of analysis and sampling submitted for endorsement (CX/MAS 16/37/3 and CX/MAS 16/37/3 Add.1), follow ups from the previous CCMAS meeting and matters related to methods of analysis referred to the Committee by the CAC and others Codex committees (CX/MAS 16/37/2).

The recommendations are tabulated in the Appendix.

A. Endorsement of methods of analysis and sampling

Committee on Contaminants in Foods (CCCF9)

Fumonisin in Maize Grain, Maize Flour and Maize Meal

The WG recommended to include “methods of analysis” into the title as it deals with both sampling and methods of analysis. The title will then be “Sampling plan and methods of analysis for fumonisins (FB1+FB2) in maize grain and maize flour and maize meal”.

Sampling Plans

WG recognized that the sampling plans had been revised to remove inconsistencies as requested by CCMAS and that the performance criteria had been adjusted in accordance with the “Guidelines for establishing numeric values for criteria.”

There were some discussions regarding the size of the test portion, as some methods of analyses prescribes test portions of far less than 25 g. It was agreed that the term “should” in para 19 opens up for the use of different amounts of the test portion.

Some suggested that aggregate sample should be included in the table as aggregate samples is included in the definition and in the text. However, last year, CCMAS suggested to delete the row of aggregate sample as it caused confusion.

The WG recommended endorsement of the Sampling Plans.

Methods of Analysis

The WG recommended endorsement of the criteria approach for the analytical methods.

Deoxynivalenol (DON) in cereal-based foods for infants and young children; in flour, meal, semolina and flakes derived from wheat, maize or barley; and in raw cereal grains (wheat, maize and barley) including sampling plans for raw cereal grains

The WG recommended to include “methods of analysis” into the title as it deals with both sampling and methods of analysis.

The title will then be “Sampling plan and methods of analysis for deoxynivalenol (DON) in cereal-based foods for infants and young children, in flour, meal, semolina and flakes derived from wheat, maize or barley; and in grains (wheat, maize and barley) destined for further processing.”

Sampling Plans

The discussions regarding test portion and aggregate sample discussed under sampling plan for fumonisins were also relevant for DON. The WG recommended endorsement of the sampling plan.

Methods of Analysis

The WG recommended endorsement of the criteria approach for the analytical methods.

Committee on Spices and Culinary Herbs (CCSCH2)

Methods of Analysis for Cumin and Dried Thyme, respectively

Determination of Moisture

The WG recommended endorsement of the Karl Fisher titration methods; AOAC 2001.12 and ISO 760:1978 as Type III methods.

It was recognized that the ISO method had not been collaboratively studied. The AOAC method is collaboratively studied, but not for cumin. However, the Karl Fisher titration is a general method, and it is not practical (impossible) to include all types of food in a method validation. If the two methods provide equivalent results, the CCSCH might consider to recommend one of the method as a Type II and the other as Type III.

The WG recognized that there was a typing error; the CCSCH suggested ISO 938:1980 should be ISO 939:1980. The WG recommended not endorsement of ISO 939:1980, nor ASTA 2.0.

Determination of Total Ash

The WG recommended endorsement of the ISO 928:1997 as Type I.

The WG recognized that there can only be one Type I methods, no alternative ones.

The AOAC 950.49 contains two different procedures for the determination of ash. It could not be confirmed whether one of the procedures in AOAC 950.49 and the ASTA 3.0 were equivalent to ISO 928:1997.

Determination of Acid-insoluble Ash

The WG recommended endorsement of ISO 930:1997 as Type I.

The WG recognized that there can only be one Type I method specified in a Standard, with no alternative procedures endorsed.

It could not be confirmed that ASTA 4.0 was equivalent to ISO 930:1997.

Determination of Volatile Oils

The WG recommended endorsement of ISO 6571:2008 as Type I.

The WG recognized that there can only be one Type I method specified in a Standard, with no alternative procedures endorsed.

It could not be confirmed that AOAC 962.17 and ASTA 5.0 were equivalent to ISO 6571:2008.

Determination of Extraneous Vegetable Material

The WG recommended endorsement of ISO 927:2009 as Type I.

The WG recognized that there can only be one Type I method specified in a Standard, with no alternative procedures endorsed.

It could not be confirmed that ASTA 14.1 was equivalent to ISO 927:2009.

Determination of Foreign Matter

The WG recommended endorsement of ISO 927:2009 as Type I.

Determination of Insect Damage

The WG recommended endorsement of method published in the FDA Technical Bulletin Number 5 as Type IV. The method is not collaboratively studied.

COMMITTEE ON FISH AND FISHERY PRODUCTS (CCFFP34)

Amendments to Section 7.4 of the Standard for Quick Frozen Fish Sticks (Fish Fingers), Fish Portions and Fish Fillets – Breaded or in Batter

The WG recommended endorsements of the Amendments to Section 7.4; i.e. inclusion of the calculation of the Fish Content and two links to leading to the same web site (which is overseen by FAO) listing nitrogen factors.

There were some concerns having information on the conversion factors linked to Websites instead of including the factors in the standard. Whether conversion factors should be discussed within the framework of CCMAS was also raised; see discussion at the last page.

The WG recommended to inform CCFFP that the factors might be reassessed when reviewing the standards.

COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU37)

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

The WG recognized that there was not a consensus whether to recommend CCNFSDU to convert the suggested method into method performance criteria, or to endorse the suggested methods as Type II.

The reasoning for endorsing the suggested method as Type II were:

- there have been very many disputes in this area, therefore these methods have been carefully chosen by an international community, and thereafter extensively validated in international studies with participants from all over the world
- IDF, ISO and AOAC International have agreed on these methods
- the standard method performance requirements (SMPRs) are stricter than the method performance criteria given in the Procedural Manual. It was suggested distributing a paper prepared to IAM by AOAC International and IDF to the delegates regarding this matter.
- it is important that the methods determine the same entity

The reasons for the suggestion of converting the specified methods into method performance criteria were:

- these are rational methods, and the SMPRs or the performance of the suggested methods can easily be converted into criteria,
- the laboratories are given freedom of choice in the use of method,
- some of the suggested methods includes expensive instrumentation that not all countries possess. For instance, it was questioned whether it is necessary to use ICP-MS for the determination of chromium and selenium, or if GF-AAS and HGAAS, respectively, still provide satisfactory results. GF-AAS and HGAAS are the principles of the current Type II method for infant formula and in widely use. The TC noted the request for documentation, including results for PT-schemes, on the fact that the current Type II methods given in Codex Stan 234 are not fit for purpose according the Codex requirements.

The WG recommended endorsement of the suggested AOAC methods equivalent to the ISO or ISO/IDF methods for the determination of **Vitamin B12, Chromium, Selenium, Molybdenum, Iodine** and **Pantothenic Acid**. Provided that documentation show that the current Type II methods are no longer fit in dispute situations, the WG recommended the methods endorsed as Type II. The current Type II methods should then become Type III.

Determination of myo-inositol

The WG recommended to request CCNSFDU to confirm that the AOAC 2011.18 and ISO 20637 determine the measures defined in the Codex Stan 72 for myo-inositol. Provided that the definition and the methods' scope harmonize, the WG recommended endorsement of AOAC 2011.18 and ISO 20637 as Type II. (It does not need to come back for re-endorsement by CCMAS.)

Determination of Nucleotides

The WG recommended to request CCNSFDU to confirm that the AOAC 2011.20 and ISO 20638 determine the compound given in the provision in the Codex Stan 72. The provision in the standard is total nucleotides but the methods determine 5'-Mononucleotides. Provided that the provision and the methods' scope harmonize, the WG recommended endorsement of AOAC 2011.20 and ISO 20638 as Type II. (It does not need to come back for re-endorsement by CCMAS.)

Determination of vitamins A and E

The WG recommended to request CCNSFDU to confirm that the scope of AOAC 2012.10 and ISO 20633 is in line with the provision for the isomers of Vitamin E in the Codex Stan 72. Provided that the provision and the methods' scope harmonize, the WG recommended endorsement of 2012.10 and ISO 20633 as Type II. (It does not need to come back for re-endorsement by CCMAS.)

The WG recommended to request CCNSFDU to include the conversion of IU to µg, and conversion from kg to 100 calories for all the vitamins of interest, not only for vitamin A, in Codex Stan 72.

Determination of fatty acid profile

The WG recognized that that the provision in the Codex Stan 72 is Total Fat and Linoleic Acid, that the scope of the AOAC 2012.13 and ISO 16958 | IDF 231 are Determination of Fatty Acids Composition and that the scope in the table is Total Fatty Acid Profile. Provided that the provision and the methods' scope harmonize, the WG recommended endorsement of the methods as Type II. (It does not need to come back for re-endorsement by CCMAS.)

The CRD 6 provides extensive information about the proposed methods, the results of the validations and other different methods considered.

A summary of the agreed discussion on which methods to include in the appropriate Standard is given in CRD 24.

B. OTHER MATTERS

Follow up from last CCMAS session

REP15/MAS, paragraph 37: ISO was requested to provide information on the equivalence of the ISO method with the COI/T.20 doc. No. 30-2013 (Determination of sterols in olive oils and olive pomace oils)

The WG recognized that ISO 12228-2 (Determination of individual and total sterols contents - Gas chromatographic method - Part 2: Olive oils and olive pomace oils) is equivalent to IOC Standard COI/T.20/Doc. No. 30-2013. (See CRD 19)

REP15/MAS, paragraph 39: Information on the applicability of ISO 1211|IDF 1:2010 for lipid determination in tempe and information on whether this method had been tested on tempe products

As the ISO 1211|IDF 1:2010 methods are not validated for tempe, it has been reported that it is not appropriate to replace the current method, AOAC 983.23. As the AOAC method uses chloroform, Indonesia

proposed to replace this method with the AOAC 963.15. The WG did not recommend endorsement of AOAC 963.15 as the method is for determination of fat in cacao.

NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU37)

Examination of “ELISA G12” as a potential additional method for inclusion in Standard for Foods for Special Dietary Use for Persons Intolerant to Gluten (CODEX STAN 118-1979)

The WG recommended to inform CCNFSDU that (regrettably) the two methods (the R5 and G12 methods) for the determination of gluten are not comparable. Comparison data for the two methods are not available, and mixed matrixes are not included in the scope of either of the methods obtained during their validation. Users confirmed that the two methods provide different results, and that both methods are used in official control. The developers of these proprietary methods might be able to provide further information on the applicability of the methods.

Nitrogen Conversion Factor

REP 15/CAC, para 20 and

Rep 16/NFSDU, para 57; Review of the Standard for Follow-Up Formula (CODEX STAN 156-1987)

The WG discussed if conversion factors are in the scope of CCMAS. The FAO secretariat confirmed that it is most probably not in its scope as the factors do not influence on the analytical methodology. However, other Codex Committees do ask CCMAS for advice.

The WG recognized that there was no consensus on the nitrogen factors. It was however agreed that it is important that the conversion factors are scientifically based and that the factors should be harmonized between different standards. WG also recognized that the factors have severe economic aspects.

The WG recommends CAC to return the request to the commodity committee.

The WG requests the FAO secretariat to assist in moving forward on this matter, e.g. requesting FAO/WHO for arranging an expert panel meeting reviewing the literature and updating their report from 2002.

Appendix

COMMITTEE ON SPICES AND CULINARY HERBS (CCSCH)**METHODS OF ANALYSIS FOR CUMIN**

Provision	Method	Principle	Notes and Type Proposed
Moisture	ISO 938:1980 ISO 939:1980 Alternative:- ISO 760:1978 AOAC 2001.12 ASTA 2.0	Titration	III
Total ash	ISO 928:1997 Alternative:- AOAC 950.49 ASTA 3.0	Gravimetry	I
Acid-insoluble ash	ISO 930:1997 Alternative:- ASTA 4.0	Gravimetry	I
Volatile oils	ISO 6571:2008 Alternative:- AOAC 962.17 ASTA 5.0	Distillation/Volumetric	I
Extraneous vegetable Material-matter	ISO 927:2009 Alternative:- ASTA 14.1	Visual examination / Gravimetry	I
Foreign matter	ISO 927:2009	Visual examination/Gravimetry	IV
Insect damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)	Visual examination	

METHODS OF ANALYSIS FOR DRIED THYME

Provision	Method	Principle	Notes and Type Proposed
Moisture	ISO 939:1980 Alternative: ISO 760:1978 AOAC 2001.12 ASTA 2.0	Distillation Titration	I
Total ash	ISO 928:1997 Alternative: AOAC 950.49 ASTA 3.0	Gravimetry	I
Acid-insoluble ash	ISO 930:1997 Alternative: ASTA 4.0	Gravimetry	I
Volatile oils	ISO 6571:2008 Alternative: AOAC 962.17 ASTA 5.0	Distillation Volumetric	I
Extraneous vegetable Material/matter	ISO 927:2009 Alternative: ASTA 14.4	Visual examination Gravimetry	I
Foreign matter	ISO 927:2009	Visual examination Gravimetry	I
Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)	Visual examination	IV

Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5)	Visual examination	IV
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METHODS OF ANALYSIS IN THE STANDARD FOR INFANT FORMULA AND FORMULAS FOR SPECIAL MEDICAL PURPOSES INTENDED FOR INFANTS (CODEX STAN 72-1981)

Commodity	Provision	Method	Principle	Proposed Type
Infant Formula	Vitamin B12	AOAC 2011.10 ISO 20634	High Performance Liquid Chromatography (HPLC)	II
Infant Formula	Myo-Inositol	AOAC 2011.18 ISO 20637	Liquid Chromatography (LC)-pulsed amperometry	II
Infant Formula	Chromium	AOAC 2011.19 ISO 20649 IDF 235	Inductive Coupled Plasma-Mass Spectrometry (ICP-MS)	II
Infant Formula	Selenium	AOAC 2011.19 ISO 20649 IDF 235	ICP-MS	II
Infant Formula	Molybdenum	AOAC 2011.19 ISO 20649 IDF 235	ICP-MS	II
Infant Formula	5'-Mononucleotides	AOAC 2011.20 ISO 20638	LC	II
Infant Formula	Vitamin A Palmitate (Retinyl Palmiate), Vitamin A Acetate (Retinyl Acetate), Total Vitamin E (dl- α -Tocopherol and	AOAC 2012.10 ISO 20633	HPLC	II
Infant Formula	Total Fatty Acid Profile	AOAC 2012.13 ISO 16958 IDF 231	Gas Chromatography	II
Infant Formula	Iodine	AOAC 2012.15 ISO 20647 IDF 234	ICP-MS	II
Infant Formula	Pantothenic Acid	AOAC 2012.16 ISO 20639	Ultra HPLC-MS/MS	II