

# 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/39 CRD/2

ORIGINAL LANGUAGE ONLY

## JOINT FAO/WHO FOOD STANDARDS PROGRAMME

### CODEX COMMITTEE ON METHODS OF ANALYSIS SAMPLING

Thirty-ninth Session

Budapest, Hungary, 7 – 11 May 2018

#### REPORT OF THE PWG ON ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING

The PWG met on Sunday, 6 May 2018. The PWG considered matters referred for action by CCMAS identified in CX/MAS 18/39/2 Add.1, matters for endorsement in CX/MAS 18/39/3 and CX/MAS 18/39/3 Add.1. and recommended methods of analysis for milk and milk products in CX/MAS 18/39/4 Add.1.

The PWG had the following discussion and made recommendations presented in the Appendix. I

#### CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU39)

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

The PWG reviewed, endorsed and typed the methods referred by CCNFSDU (Table I). Additionally, there were recommendations for revocation of a number of previously listed methods. In the instances of proposed revocation, it was noted that any removal of methods cannot be made unilaterally by CCMAS, but that recommendation should be referred to CCNFSDU prior to removal of the methods from CODEX STAN 234

The revocation of AOAC 992.26 and AOAC 995.05 (as recommended by AOAC, ISO and IDF in MAS/39 CRD/3) for the determination of vitamin D<sub>3</sub> in Ready-to-Feed-Milk-Based Infant Formula, and the determination of vitamin D in Infant Formulas and Enteral products, respectively was proposed and accepted by the PWG. It was discussed that the methods do not use internal standards, which are widely regarded as necessary for analytical methods, that include hot saponification as part of the sample preparation, and that both methods are not validated for a broad range of infant formula products currently on the market. Therefore, in conclusion, both methods are no longer fit-for-purpose.

For the vitamin D methods (AOAC 2016.05 | ISO DIS 20636) it was noted that the AOAC method has been published and that the ISO method has been finalized, but final publication has not yet occurred. Based on comments from ISO that the publication should be finalized prior to the Codex Alimentarius Commission meeting, the PWG recommended endorsement of both methods. However, if the ISO method is not published prior to CAC, the PWG recommends that it not be adopted by CAC2018, but that adoption occur at the first CAC meeting following publication.

A similar recommendation was also made for the Chloride methods (AOAC 2016.03 | ISO DIS 21422 | IDF 242). As with the vitamin D, the AOAC method has been published, while the ISO | IDF method has been finalized and is awaiting final publication.

#### COMMITTEE ON MILK AND MILK PRODUCTS (CCMMP)

##### ***Methods of analysis for dairy permeate powders***

The PWG reviewed, endorsed and typed some of the methods referred by CCMMP with some editorial changes (Table II). Additionally, two methods were not recommended for endorsement based on the following discussions. The method for Lactose, Anhydrous (ISO 22662 | IDF 009) was not endorsed. It was discussed that the method, which has been validated in a number of milk based products, would require a change to the mass of test portion analyzed when applied to Dairy Permeate Powder<sup>1</sup>. Dairy Permeate Powders contain higher concentrations of lactose, therefore the mass for dried powders specified in the method would not allow lactose to fall within the calibration range. The PWG determined that this change must be included in the

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<sup>1</sup> (\*) Test portion size with dairy permeate powders to be between 0.200 g and 0.260 g instead of about 0.300 g.

method prior to endorsement and typing as a Type II, with an alternative as being Type IV while this is being achieved, although a consensus on the action to take at the PWG could not be achieved.

## **CODEX COMMITTEE ON CEREALS, PULSES AND LEGUMES (CCCPL)**

### ***Methods of analysis for quinoa***

The PWG reviewed, endorsed and typed the Moisture content, and Protein Content (N x 6.25); methods referred by CCCPL. Although it was discussed that the methods have not been specifically validated with quinoa, the PWG agreed to recommend endorsement as Type I methods.

The request by CCCPL for suitable internationally validated methods for saponin was communicated to the PWG. Additionally, observer AACCI requested information on any developed/validated methods; even those which had only undergone a single lab validation, and informed the PWG of their interest in undertaking a collaborative study using an appropriate method. There were no methods reported to the PWG, but attendees were encouraged to contact AACCI or the CCCPL if they become aware of potential methods.

### ***Sampling plan for MLs for methylmercury in fish (CXS 193-1995)***

The PWG discussed and endorsed the proposed draft sampling plan for methylmercury contamination in fish, which also included numeric criteria for method performance.

A question about the target audience and that the definition of Lot was unclear and could be clarified with respect to species and other descriptors. It was pointed out that the plan is very similar to an EU method (COMMISSION REGULATION (EC) No 333/2007) which has been utilized internationally for some period of time, and that it addresses food safety concerns.

A question was raised about the proposed revision of GL50 and if endorsement of new plans should be postponed until that revision is complete and the sampling plans can be harmonized with the revised GL. Delegates pointed out that the new work had not yet been approved and that the time line and final outcome was unclear. Therefore, sampling plans should not be held, but that once GL50 is revised, efforts in reviewing previously approved sampling plans and harmonizing them with the revised GL50 should be considered.

There was discussion about the different chemical species of methyl mercury, e.g. mono-methyl and di-methyl mercury and if these are captured in the performance criteria and if so, if a "Sum of Components" approach is more appropriate than the guidelines listed in the Procedural Manual. Delegates pointed out that the standard is for total methyl mercury, that current methods do not measure methyl mercury compounds separately and that there is not a regulatory benefit in speciating. Therefore, the total methyl mercury is determined as a single measurement and the current Procedural Manual Guidelines are applicable.

There was some concern expressed over Table 5 of the Proposed Draft Sampling Plan, which seems to capture the general requirements in establishing numeric criteria. However, this Table, as presented, is different from the Guidelines established by CCMAS and present in the Procedural Manual. The PWG recommended removal of this table and section, which discusses criteria and noted it could be replaced by a reference to the procedural manual. If CCCF chooses to retain this Table it should be corrected to agree with the numeric performance criteria approach included in the Procedural Manual. Additionally, Table 7, which lists the numeric criteria for different fish, is not consistent with the format or information currently used in CODEX STAN 234. A table consistent with CODEX STAN 234 has been drafted, using the guidelines listed in the Procedural Material, for consideration by the Committee (Replacement for Table 7).

## **COMMITTEE ON CONTAMINANTS IN FOODS (CCCF)**

### ***Sampling plan for MLs for methylmercury in fish (CXS 193-1995)***

#### **Response to questions from CCCF**

The PWG agreed that while some delegates may have experience in this area, these questions were outside the remit of PWG and CCMAS. Delegates with the necessary background were encouraged to respond through their CCCF delegations.

#### **Review of Proposed new methods from CX/MAS 18/39/4 Add. 1**

Methods for propionic acid in cheese and emmental were reviewed and endorsed including ISO/TS 19046-1 | IDF/RM 233-1.

#### **ADDRESS QUESTIONS RAISED BY IDF/ISO/AOAC in CX/MAS 18/39/4 Add. 1**

Clarify rules for determining when a defining method should be Type I or Type IV method.

For example:

**Is it necessary to have precision figures for a Type I method?**

There was general agreement that precision figures for Type I methods are an important aspect of assessing the performance of the methods. Moving forward any newly developed/proposed Type I should present precision figures as part of the data reviewed during the endorsement process. However, there was also agreement that while having such data for long standing methods would be beneficial, lack of such data would not cause a change in the method type or revocation of a method.

**If a defining method has been subjected to an international collaborative study involving dairy commodities A, B and C, and the method is generally known to work on commodity D, but this commodity was not included in the study, should the method then be listed as Type I or Type IV in STAN 234 for commodity D?**

It was agreed that a general rule to extend or not extend the typing is not appropriate. Because the decision would depend on the matrices involved as well as the analytical procedure the typing determination should be made on a case-by-case basis.

**Clarify for the situation where there are two defining methods (from different organisations) and the degree of validation differs (i.e. one method has been subjected to an international collaborative study, whereas the other method has not), whether one method be Type I and the other method Type IV, or only one (the best validated) method should be accepted and be listed as Type I.**

There was no consensus reached on this question and delegates suggested that discussion around the terms “technically equivalent” and “technically identical” should be resolved prior to further discussion on this question.

**Clarify for those cases where a provision is not specifically listed in the Commodity Standard, what decision process is to be followed to determine whether or not to include such provision in CXS234 (e.g., see provisions for iron in milk products, lead in edible casein products, and MSNF in cream in the table below).**

It was agreed that some ‘indication’ in the Commodity Standard should exist in order for a provision to be listed in CODEX STAN 234. This ‘indication’ does not have to be a specific Provision in the standard, but may also be general text, for example a referral to contaminants.

**Apply a consistent approach in listing provisions that require a calculation based on two or more analyses. In some cases, all concerned methods are listed; in other cases only a single method.**

It was agreed that all methods should be listed and separated by the word “and”.

**ADDRESS Table in CX/MAS 18/39/4 Add 1 and WORKABLE PACKAGE “DAIRY GROUP”**

The PWG began the review of the Dairy Group Workable Package. This review raised questions of about the applicability of some methods and about previous endorsement and typing decisions.

The PWG considered the case of the designation of Type I and Type IV of methods when applied to a new commodity. For example, methods designated as Type I for a particular commodity were changed to Type IV when applied to a new commodity. This change produced a situation where two methods, both for nitrogen determination by Kjeldahl titrimetry were both listed in STAN 234 as Type IV.

These methods are both empirical (not rational) and they are Type I methods throughout CODEX STAN 234. It is quite possible that they could generate different results for the same sample and delegates expressed concern over this possible outcome. Other delegates expressed support for this approach and referenced a similar, but not identical approach, being taken with the initial endorsement of methods for gluten. Initially, 2 ELISA methods were endorsed as Type IV and when one was successfully collaboratively studied, the typing was changed to Type I and the other ELISA was removed from CODEX STAN 234. Participants also pointed to the definitions for method types in the Procedural Manual to suggest the above approach is consistent with those definitions.

No consensus was reached on this topic.

**CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (CCNFSDU39)**

***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	Proposed Type
Infant Formula	Biotin	EN 15607	HPLC- <u>fluorescence</u>	≠ III
		AOAC 2016.02	HPLC- <u>UV</u>	II
	Vitamin D	<del>AOAC 992.26</del>	HPLC	≠≠
		EN 12821	HPLC-UV	≠ III
		<del>AOAC 995.05</del>	HPLC	≠≠
		AOAC 2016.05   ISO DIS 20636	LC-MS	II
	Chloride	<del>AOAC 986.26</del>	Potentiometry	≠≠
		AOAC 2016.03   ISO DIS 21422   IDF 242	Potentiometry	II

**COMMITTEE ON MILK AND MILK PRODUCTS (CCMMP)****Methods of analysis for dairy permeate powders**

Provisions	Method	Principle	Type
Lactose, anhydrous	ISO 22662   IDF 198:2007 - Milk and milk products - Determination of lactose*	HPLC-refractometry	II
Milkfat	ISO 1736   IDF 009:2008 - Dried milk and dried milk products - Determination of fat content	Gravimetry [Röse-Gottlieb]	I
Nitrogen	ISO 8968-1   IDF 020-1:2014 - Milk and milk products - Determination of nitrogen content - Part 1	Titrimetry, [Kjeldahl principle]	I
Moisture**	ISO 5537   IDF 026:2004 - Dried milk - Determination of moisture content	Gravimetry [drying at 87°C]	I
Ash	NMKL 173 :2005 Ash, gravimetric determination in foods AOAC 930.30 1930 - Ash of Dried Milk	Gravimetry [ashing at 550 °C]	IV

**(\*) Test portion size with dairy permeate powders to be between 0.200 g and 0.260 g instead of about 0.300 g.**

**(\*\*) Moisture content excluding the water of crystallization of lactose.**

**CODEX COMMITTEE ON CEREALS, PULSES AND LEGUMES (CCCPL)****Methods of analysis for quinoa**

Provision	Method	Principle	Type
Moisture content	ISO 712	Gravimetric	I
Protein Content [(N x 6.25) Dry weight basis]	ISO 1871	Titrimetry, Kjeldahl	I

Replacement for Table 7: Calculated Performance Criteria for methylmercury.

Provision	ML (mg/kg)	Min Appl. Range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (%) Not more than	Recovery (%)	Applicable Methods that meet the criteria	Principle
All Tuna	1.2	0.64 – 1.8	0.12	0.24	31	80 – 110		
Alfonsino	1.5	0.82 – 2.2	0.15	0.30	30	80 – 110	988.11	GC-electron capture
All Marlin	1.7	0.95 – 2.5	0.17	0.34	30	80 – 110	988.11	GC-electron capture
Shark	1.6	0.88 – 2.32	0.16	0.32	30	80-110	988.11	GC-electron capture

#### Review of Proposed new methods from CX/MAS 18/39/4 Add. 1

Commodity	Provision	Method	Principle	Type
<b><u>Cheese</u></b>	<b><u>Propionic acid</u></b>	<b><u>ISO/TS 19046-1I IDF/RM 233-1</u></b>	<b><u>Gas chromatography</u></b>	<b><u>IV</u></b>
<b><u>Cheese</u></b>	<b><u>Propionic acid</u></b>	<b><u>ISO/TS 19046-2I IDF/RM 233-2</u></b>	<b><u>Ion exchange chromatography</u></b>	<b><u>IV</u></b>
<b><i>Comment: CXS 283 General Standard for Cheese has a maximum level of 3000 mg/kg for propionic acid.</i></b>				

Commodity	Provision	Method	Principle	Type
<b><u>Emmental</u></b>	<b><u>Propionic acid</u></b>	<b><u>ISO/TS 19046-1I IDF/RM 233-1</u></b>	<b><u>Gas chromatography</u></b>	<b><u>IV</u></b>
<b><u>Emmental</u></b>	<b><u>Propionic acid</u></b>	<b><u>ISO/TS 19046-2I IDF/RM 233-2</u></b>	<b><u>Ion exchange chromatography</u></b>	<b><u>IV</u></b>