CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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**Agenda item 4** **CX/MAS 20/41/4 Add.1**

**July 2020**

**JOINT FAO/WHO FOOD STANDARDS PROGRAMME**

**CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING**

# REVISION OF THE RECOMMENDED METHODS OF ANALYSIS AND SAMPLING (CXS 234 – 1999) DAIRY PACKAGE

**Comments in reply to CL 2020/29/OCS-MAS**

*Comments of Egypt, Honduras, Iraq, Norway, Peru, Syrian Arab Republic, Uruguay, IDF, NMKL*

**NOTE:** CCMAS41 has been postponed to 17 – 21 May 2021. In order to ensure work continuity comments were requested on the two workable packages. See background information in CL 2020/29/OCS-MAS. The comments compiled in this document will be made available to the EWG on the dairy package for discussion and preparation of revised proposals for consideration by CCMAS41.

## Background

1. This document compiles comments received through the Codex Online Commenting System (OCS) in response to CL 2020/29/OCS-MAS issued in March 2020. Under the OCS, comments are compiled in the following order: general comments are listed first, followed by comments on specific sections.

## Explanatory notes on the appendix

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

**ANNEX I**

| GENERAL COMMENTS |
|---|
| **Uruguay**<br>Uruguay appreciates the work carried out by the Dairy Products EWG led by the United States and New Zealand and the work of the Fats and Oils EWG led by the Netherlands and is pleased to present the following general and specific comments to the questions raised in the circular letter CL 2020/29 / OCS-MAS as follows:<br>REVIEW OF METHODS OF ANALYSIS IN CXS 234 MANAGEABLE SET FOR DAIRY PRODUCTS Document CX / MAS 20/41/4<br>COMMENT paragraph 9<br>AOAC METHODS 965.33 Oil and Fat Peroxide Index, ISO 3976 \| IDF 74 (Milk Fat - Determination of Peroxide Index).<br>The eWG reviewed the comparison of methods and the recommendation of the standards development organization (AOAC International), as well as the Information document: Guidance on the method presentation, consideration and ratification process.<br>The eWG recommends that AOAC 965.33 be revoked and ISO 3976 \| IDF 74 maintained as a Type I method.<br>Uruguay comment: Regarding the determination of peroxides index in oils and fats, it is understood that it is necessary to maintain the AOAC 965.33 method considering that the principle of the method is by evaluation, making it an accessible and possible method to implement in any laboratory. On the other hand, it is the method established in the MERCOSUR RES 70/93 Standard applicable in our national regulations.<br>COMMENT paragraph 22<br>Water in butter (ISO 37271-1 \| IDF 80-1): Is the footnote appropriate for butter?<br>Uruguay comment: Referring to the footnote of the water in butter method, it is recommended that the provision be named as the test standard indicates, humidity, and that a note should be included indicating that whenever water is referenced, humidity is read in this provision. to contemplate the different names in Codex product standards.<br>COMMENT paragraph 22<br>Total acidity in fermented milks (ISO / TS 11869 \| IDF / RM 150): Should this be Type I, due to a conversion factor in the method?<br>Uruguay comment: Regarding the acidity typification question in fermented milks, it is understood that the conversion factor in this method does not convert the method into a type I method.<br>COMMENT Appendix II ISO 5547 method \| IDF 91<br>Edible Casein Products Free Acids<br>Maximum free acidity4 ISO 5547 \| IDF 91 Valorimetry (aqueous extract) I<br><br>Uruguay comment: Regarding the question of how the provision should be called for this method, it is understood, as for the previous method, to consider free acidity as in the test standard.<br><br>Dairy Permeate Powder Ash NMKL 173<br>AOAC 930.30 Gravimetry (ash reduction at 550 ° C) IV<br>Uruguay comment: It is suggested to include the AOAC 930.30 method.<br><br>GROUP 3 WATER IN MILK PRODUCTS<br>Context<br>In the context of Dried Milk and by-product Quality measurements, the limited<br>accessibility to the method of determination of moisture content defined in the ISO |

5537 I IDF26: 2004 standard has been identified in Latin American countries. This has been expressed during the revision of milk and milk products standards of the Codex Alimentarius carried out in the framework of the work of the Committee on Analysis and Sampling (CCMAS) at its 40th session and during the Committee of Latin America and the Caribbean (CCLAC) at its 21th session.

The main limitation identified is the applicability of ISO 5537 | IDF 26: 2004, particularly the access to the equipment and test conditions defined in the standard.
Within the necessary equipment to perform the test, is required a specific design oven which is not easily available in South America. Other limitations of use are the number of samples to test by batch which is limited to the number of places available with the oven, the availability of supplies and specific packing which are replaced with a certain frequency together with the generation of waste to the environment.
It also requires measuring air quality and flow, which must be demonstrated through traceable calibrations with international recognition. The verification of the composition of air is unavailable or expensive in our region. The air flow needs to be controlled at 33 ml / min as a critical factor in function of the robustness studies presented in reference 5 of ISO 5537 | IDF 26: 2004. Air flow calibration with
recognized CMC is not available in many National Measurement Institutes including Latin America.

Review of ISO 5537 | IDF 26:2004 within the framework of the Codex Alimentarius

Analyzing Codex general criterion for the selection of methods of analysis, the following comments are identified to the relevance of the method ISO 5537 | IF 26 :2004.
In reference to the preference that should be given to methods of analysis whose reliability have been established in respect of the performance criteria, ISO 5537 | IF 26:2004 has validation data in dried milk powders within the scope of the standard.
Codex endorsed this standard to blend of skimmed milk and vegetable fat in powdered form, reduced fat blend of skimmed milk powder and vegetable fat in powdered form, cream powders, so additional validation data is required.
In reference to methods selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use, limitations with accessibility to the oven, suppliers and control of operating conditions which restrict the use of the method in Latin America as described previously.
In reference to the methods of analysis which are applicable uniformly to various groups of commodities that should be given preference over methods which apply only to individual commodities, general methods of Codex for moisture are gravimetric methods under normal laboratory conditions (normal or reduced pressure).
Below is proposed an alternative method for water content of milk products and single validation data related.

Method description:
Commodity Provision
Whole milk powder, Shimmed milk powder, Dried cream, Dairy permeate powders (Whey powder), Blend of Skimmed Milk and Vegetable Fat in Powdered Form, Baby Dried Milk Water content (or moisture in lines with that CCMAS define for this provision)

DESCRIPTION OF THE METHOD: DETERMINATION OF WATER CONTENT

1. SCOPE This method applies to Whole milk powder, Shimmed milk powder, Dried cream, Dairy permeate powders (Whey powder), Blend of Skimmed Milk and Vegetable Fat in Powdered Form, Baby Dried Milk

2. DEFINITION The water content is the loss of mass determined by the procedure specified in this Standard. It is expressed in g/100g.

3. PROCEDURE
3.1 Preparation of the dish. Heat an uncover dish and its lid in the oven controlled at 102 + 2°C for 1 h. Transfer the cover dish to the desiccator, allow to cool to room temperature and weight to the nearest 0,1mg.
3.2 Put approximately 1-3g of the sample into the dish, cover with de lid and weight to the nearest 0,1mg.
3.3 Uncover the dish and place it with it lid in the oven, well ventilated, controlled at 102 + 2°C for 2 h.
3.4 Transfer the cover dish to the desiccator, allow to cool to room temperature and weight to the nearest 0,1 mg.
3.5 Uncover the dish and heat it again, in the oven for 1 h.
3.6 Repeat this process until the difference in mass between to successive weighing does not exceed 0,5mg.

4. CALCULATION AND EXPRESSION OF RESULTS

$$\frac{m1 - m2 \times 100}{m1 - m0}$$

m0 is the mass, in grams, of the dish and the lid (3.1)
m1 is the mass, in grams, of the dish, the lid and the test portion before drying (3.2)
m2 is the mass, in grams, of the dish, the lid and the test portion after drying (3.6)

Data Validation
Validation procedure: AOAC Guidelines for Standard Method Performance Requirements (2016)

| Commodity (Whey powder) | Whole milk powder Vegetable Fat in Powdered Form | Skimmed milk powder | Dairy permeate powders | |
|---|---|---|---|---|
| Provision | Water content | Water content | Water content | Water content |
| Mean value (%) | 3,21 | 4,11 | 1,88 | 2,09 a 3,57 |
| Repeatability standard deviation, Sr (%) | 0,035 | 0,082 | 0,034 | 0,024 |
| CVr (%) | 1,09 | 2,00 | 1,81 | 2,39 |
| Repeatabilite limit, r (2,8 Sr) (%) | 0,098 | 0,231 | 0,095 | 0,067 |

| Commodity (Whey powder) | Whole milk powder Baby Dried Milk | Skimmed milk powder | Dairy permeate powders | |
|---|---|---|---|---|
| Provision | Water content | Water content | Water content | Water content |
| Mean value (%) | 2,30 a 3,45 | 3,98 a 5,02 | 2,40 a 5,68 | 1,50 a 2,60 |
| Reproducibility CVR (%) | 4,89 | 2,75 | 2,03 | 8,46 |

Reference Material
Commodity Whey
Powder Spray Dried
Milk powder Whole Dried
Milk Whole Dried

Milk
PT provider procedence United
Kington United
Kington New Zeland New Zeland
Certified value assigned by Gravimetric at 102ºC Gravimetric at 102ºC Gravimetric at 102ºC Gravimetric according with NZTM3.12.23
Certified Value (g/100g) 2,42 3,83 3,19 3,32
Result (*)
Satisfactory Satisfactory Satisfactory Satisfactory

(*) Conformity criteria $0 < \sqrt{(|\text{Lab value-reference value}|/(〖U_{(lab value)}〗^2+〖U_{(reference value)}〗^2))} < 1$
Data performed by an accredited ISO/IEC 17025:2017 laboratory since 1998. Calculation using ISO 5725-2:1994

**Norway**

Thanks the members of the working group for their extensive work in reviewing the methods. As we do not possess all methods, we rely upon the experts review of the methods including validation data thereof.

**Syrian Arab Republic**

Approval

**Peru**

1.- Observaciones sobre los cambios propuestos a los métodos relacionados con los productos lácteos de la Norma CXS 234, y brindar puntos de vista o respuestas a las preguntas planteadas en los párrafos 22 y 23-28 del documento CX/MAS 20/41/4

a) Observaciones sobre los cambios propuestos a los métodos relacionados con los productos lácteos de la Norma CXS 234.

El Perú agradece al Grupo de trabajo por medios electrónicos presidido por los Estados Unidos y copresidido por Nueva Zelandia, por el esfuerzo emprendido en la revisión del CXS 234-1999 y la redacción del documento CX/MAS 20/41/4; que nos da la oportunidad de manifestar que nos encontramos conforme con los cambios propuestos a los métodos relacionados con los productos lácteos de la Norma CXS 234.

b) Brindar puntos de vista o respuestas a las preguntas planteadas en los párrafos 22 y 23-28 del documento CX/MAS 20/41/4

En relación a las preguntas planteadas en los párrafos 23 al 28 del CX/MAS 20/41/4, no se presentará opinión al respecto.

**Iraq**

agree with proposed.

**SPECIFIC COMMENTS**

**Egypt**

comments on Appendix I, Appendix II:

- Remove the words (drying) &(ashing) from the principle description.
- Correct (ICP) at principle description to be (Inductively coupled plasma).

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| | **Norway**<br>We accept the proposals made by the working group, and agree to endorse the purposed changes made in Annex I to CXS 234 with the following comments:<br>• Group 2, Table G2.1 and G2.2: It would be helpful to list applicable/suggested methods that meet the criteria as done for natural mineral waters and for histamine in CXS 234.<br>• Group 2, Table G2.3 Lead methods for further review: Methods performance criteria could preferably be applied. |
| Provide comments on whether the proposals in Appendix I can be endorsed | **Egypt**<br>Egypt would like to add the following comments:<br>- We do not support the removal of the commodity “Milk Products” from the commodity description& it should be remained in CXS 234;<br>- We support the development of numeric criteria for lead in butter, edible casein products, and adding numeric criteria for whey powders.<br>- We do not support that “moisture” should replace “water” as the provision in CXS 234 & we recommend that a footnote may be added to note the discrepancy as described in this document.<br>- We support the revocation of AOAC 965.33 (Peroxide Value of Oils and Fats) and the retention of ISO 3976 \| IDF 74 (Milk fat — Determination of peroxide value) as a Type I method;<br>- We support the changing of the commodity name in CXS 234 to align with the commodity name in the Standard for Contaminants and Toxins in Food and Feed (CXS 193 – 1995);<br>- We support the changes to the listing of ISO 20128 \| IDF 192 (Milk products – Enumeration of presumptive Lactobacillus acidophilus on a selective medium – Colony-count technique at 37 °C);<br>- We support the replacement of ISO 17678\|IDF 202 (Milk and milk products – Determination of milk fat purity by gas chromatographic analysis of triglycerides). |
| | **IDF/FIL**<br>Group 1: IDF supports support this recommendation and invite its members to verify that removal of that information would not lead to any loss.<br><br>Group 2: IDF recognizes the value of a criteria approach regarding the endorsement of analytical methods by Codex. IDF is of the view that Type II methods are needed for the purpose of dispute resolution and that in that case, the best candidate method must be designated as such. For other routine purposes (type III), methods adopted under a criteria approach may suffice.<br>While the methods proposed by IDF/ISO are meeting the suggested criteria, IDF |

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| | believes the applicability of the criteria approach is restricted to Type III methods.<br>ISO 21424\|IDF 243 (ICP-MS) would appear to be a suitable type II method for iron and copper. A fully defined method is preferable to a criteria approach in this case. A criteria approach may be suitable for lead if there are no clearly applicable methods available.<br><br>Group 3: IDF agrees with the proposal from the eWG to leave methods as currently listed and supports considering changing the term 'water content' in relevant commodity standards replaced with 'moisture' content.<br>IDF notes that a footnote already exists in CXS 234 to note the discrepancy.<br>However in one case the footnote is not appropriate and shall be removed. There is separate provision for water in milk fat products for which CXS-234 lists ISO 5536\|IDF 23 as type II method that comes with a footnote explaining that moisture content is meant. However, both the Codex standard for milkfat products (CXS 280) the and IDF/ISO standard refer to water determination, therefore the note is not necessary.<br><br>Group 4: IDF supports the eWG recommendation.<br><br>Group 5: IDF confirms there is confusion on the provision in CXS 243 that may result in a change of recommended method(s). IDF is conducting further investigation and discussion among its membership on this topic and hope to bring a recommendation forward to the electronic working group in due course.<br><br>Group 6: IDF supports the eWG recommendation. |
| (ii) METHODS NOT PREVIOUSLY REVIEWED | **Egypt**<br>- Melamine in Milk and Milk Products (ISO/DIS 2370 \| IDF 252):<br>We support changing this to be Type II& we don't support the removal of "Milk Products" from the commodity description.<br>- Water in Butter (ISO 37271-1 \| IDF 80-1):<br>The footnote is appropriate for butter. |
| | **Norway**<br>Paragraph 22 and Appendix II Methods not previously reviewed:<br>• Ash in dairy permeate powders, NMKL 173 (request for validation data): We can inform that NMKL 173 has been validated in two collaborative studies for the following matrices: maize starch, marmalade, mayonnaise, rolled oats, feta cheese, sausage, milk powder, salami, tuna, semi skimmed milk, fromage frais, green beans, apple puree, wheat bran, rye bread. The ash content in study varied from 0.07 g/100 g to 8.0 g/100 g. The relative standard deviation of the reproducibility varied from 1.3% - 11.9%. |

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| Provide comments on the proposals in Appendix II. | **IDF/FIL**<br>IDF supports the proposed changes in Appendix II, with the exception that the method for melmaine should be listed as type II (full validation). |
| Melamine in Milk and Milk Products (ISO/DIS 2370 \| IDF 252): Should this be Type IV or changed to Type II? Should Milk Products be removed from the commodity description? | **IDF/FIL**<br>IDF invites CCMAS members and observers to review the template of submission of EN 16858/ ISO/DIS 2370\|IDF 252 method by IDF/ISO as Type II method and to consider the available precision data, see separate IDF/ISO/<br>AOAC document. The method was fully validated on the proposed matrices.<br>IDF notes that the melamine provision in CXS 193-1995 of 2.5 mg/kg is for 'Food (other than infant formulae) and feed, however the standard EN 16858/ ISO 23970\|IDF 252 scope is for milk, milk products and infants formula.<br>There is a separate entry for infant formula (that may need update too), and therefore IDF recommends keeping the commodity to milk and milk products in CXS 234. |
| Melamina en leche y productos lácteos (ISO/DIS 2370\|IDF 252): ¿Debería ser de Tipo IV o cambiarse a Tipo II? ¿Deberían eliminarse los productos lácteos de la descripción del producto? | **Honduras**<br>No , no deberían eliminarse , dejar tipo IV |
| | **Perù**<br>No se tienen referencias sobre el método ISO/DIS 2370\|IDF 252, no se ha encontrado en la ISO referencia sobre el método, ni en etapa DIS.<br>Sin embargo, se ha encontrado el ISO/TS 15495:2010 MILK, MILK PRODUCTS AND INFANT FORMULAE — GUIDELINES FOR THE QUANTITATIVE DETERMINATION OF MELAMINE AND CYANURIC ACID BY LC-MS/MS, el cual podría ser referenciado. |
| Agua en mantequilla (ISO 37271-1\|IDF 80-1): ¿Es la nota de pie de página apropiada para la mantequilla? | **Perù**<br>Se está conforme con el contenido de la nota 3: Contenido de agua sin el agua cristalizada unida a la lactosa (generalmente conocida como "contenido de humedad"). |
| Water in Butter (ISO 37271-1 \| IDF 80-1): Is the footnote appropriate for butter? | **IDF/FIL**<br>IDF supports keeping the footnote. The IDF/ISO standard is for determination of moisture (type I), and the note is appropriate as it relates to the provision in the butter standard for water. IDF would support revising the Codex standard for butter to change the provision 'water' into 'moisture'.<br>IDF notes that the reference should not read ISO 37271-1\|IDF 80-1 but ISO 3727-1\|IDF 80-1. |
| Calcium in Emmental: Should numeric criteria be developed in place of the three listed methods? | **IDF/FIL**<br>IDF would support retaining the methods as per our comment earlier on criteria approach. |
| Calcio en queso Emmental: ¿Deben desarrollarse criterios numéricos en lugar de los tres ~~métodos enumerados~~criterios numéricos? | **Honduras** |
| | **Perù** |

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| | Se recomienda que no debe haber para un mismo requisito que ya tiene un método tipo II, otros dos métodos del tipo III. Debe elegirse uno entre estos dos últimos basado en los principios que establezca el pleno del Comité sobre Métodos de Análisis y Toma de Muestras para los métodos. |
| Total Acidity in Fermented Milks (ISO/TS 11869 \| IDF/RM 150): Should this be Type I, because of a conversion factor in the method? | **IDF/FIL**<br>There is not reproducibility limit, therefore IDF recommends listing this method as type IV.<br>The conversion factor used relates to expressing the results in terms of lactic acid. It relates to the relative mass of lactic acid (90.08). |
| Acidez total en leches fermentadas (ISO/TS 11869\|IDF/RM 150): ¿Debería este ser Tipo I, debido a un factor de conversión en el método? | **Honduras**<br>deber ser tipo I |
| | **Perù**<br>Teniendo en cuenta que los métodos tipo I, determinan un valor al que puede llegarse sólo mediante la aplicación del método en cuestión y que, por definición, es el único método para establecer el valor aceptado del parámetro medido; consideramos que el método de acidez total en leches fermentadas referido podría considerarse de tipo I. |
| Ash in Dairy Permeate Powders (NMKL 173): No information was received about the applicability of this method. | **NMKL**<br>NMKL 173 is validated for Milk powder with RSDr 0.21 % and RSDR 1.3 %.<br>The method is suitable for up to 12 or 14 % ash.<br>The method should be a type I method not IV. |
| | **IDF/FIL**<br>IDF was not able to confirm applicability of this method. |
| Ceniza en polvos de permeado de lácteos (NMKL 173): No se recibió información sobre la aplicabilidad de este método. | **Perù**<br>No se tiene referencia sobre el método NMKL 173, no se utiliza en el país, se recomienda el método AOAC 945.46 Ash of Milk |
| Scorched Particles in Milk Powders and Cream Powders: New method was inserted (ADPI Scorched Particles, 2016), should this be in own line as Type IV? | **IDF/FIL**<br>IDF supports addition of the ADPI method as type IV in a separate line. |
| Partículas chamuscadas en leches en polvo y natas (cremas) en polvo: Se insertó un nuevo método sobre partículas chamuscadas (ADPI Scorched Particles, 2016), ¿debería estar en una línea propia como Tipo IV? | **Honduras**<br>Si |
| | **Perù**<br>Se está deacuerdo con la inclusión de dicho método y consideramos que se podría mantener como Tipo IV. |
| Verify that the removal of “milk products” will not cause the loss of applicable information. | **IDF/FIL**<br>IDF could not identify a loss of applicable information and supports removal of the lines listed for 'milk products'. |
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| | IDF supports support this recommendation and invite its members to verify that removal of that information would not lead to any loss. |
| Comment on the changing of the commodity name in CXS 234 to align with the commodity name in the *Standard for Contaminants and Toxins in Food and Feed* (CXS 193 – 1995), when the provision is listed in CXS 193 but not the commodity standard (e.g. butter to edible fats and oils for the provision lead). | **IDF/FIL**<br>According to CXS 193, edible fats and oil does not cover CXS 279 which is the Codex Standard for Butter. Therefore IDF recommends to keep coomodity name in that case, and only align with CXS 193 when possible. |
| Recommend retention or replacement of the methods or the development of numeric criteria for lead in butter, edible casein products, and if methods or numeric criteria for whey powders should be added. | **IDF/FIL**<br>IDF recognizes the value of a criteria approach regarding the endorsement of analytical methods by Codex. IDF is of the view that Type II methods are needed for the purpose of dispute resolution and that in that case, the best candidate method must be designated as such. For other routine purposes (type III), methods adopted under a criteria approach may suffice.<br>While the methods proposed by IDF/ISO are meeting the suggested criteria, IDF believes the applicability of the criteria approach is restricted to Type III methods.<br>ISO 21424\|IDF 243 (ICP-MS) would appear to be a suitable type II method for iron and copper. A fully defined method is preferable to a criteria approach in this case. A criteria approach may be suitable for lead if there are no clearly applicable methods available. |
| Consider if “moisture” should replace “water” as the provision in CXS 234 and how changes to the commodity standard could be made. | **IDF/FIL**<br>IDF agrees with the proposal from the eWG to leave methods as currently listed and supports considering changing the term ‘water content’ in relevant commodity standards replaced with ‘moisture’ content.<br>IDF notes that a footnote already exists in CXS 234 to note the discrepancy. |
| Consider suggestions for changes to the provision in CXS 234 to better align with the provision of CXS 243 and the scope of ISO 27205 \| IDF 149. | **IDF/FIL**<br>IDF confirms there is confusion on the provision in CXS 243 that may result in a change of recommended method (s). IDF is conducting further investigation and discussion among its membership on this topic and hope to bring a recommendation forward to the electronic working group in due course. |

**Submissions of methods for Endorsement of Methods of Analysis and Sampling**
**By IDF, ISO, AOAC INTERNATIONAL**

**Executive Summary**

This document provides additional information as per the template agreed on at CCMAS 2019 for the submission of new methods in the framework of the CCMAS eWG on dairy packages, in particular on review of methods Group 2 (methods for copper and iron in edible casein products and milkfat products), and methods not previously reviewed.

AOAC, IDF and ISO recommend the eWG/CCMAS to take the following actions:
1. Endorse AOAC 2015.06 / ISO 21424 | IDF 243 as Type II for the determination of iron in edible casein products and milkfat products,
2. Endorse AOAC 2015.06 / ISO 21424 | IDF 243 as Type II for the determination of copper in milkfat products and change ISO 5738|IDF 76 to type III,
3. Endorse AOAC 2011.14 / ISO 15151 | IDF 229 as Type III for the determination of iron, in edible casein products,
5. Endorse EN 16858 – ISO/DIS 23970 | IDF 252 as Type II for the determination of melamine in milk and milk products,
6. Endorse AOAC 2015.06 / ISO 21424 | IDF 243 as Type II for the determination of calcium in emmental cheese,
7. Endorse AOAC 2011.14 / ISO 15151 | IDF 229 as Type III for the determination of calcium in emmental cheese.

**CCMAS eWG on dairy package – review of methods- Group 2**

Method(s) for determination of iron in edible casein products and milkfat products

There is currently no method listed in CXS 234 for the determination of iron in edible casein products and in milkfat products. As an outcome of CCMAS40, the EWG is requesting new methods or a criteria approach (group 2).

- AOAC, IDF and ISO recently published 2 methods (AOAC 2015.06 / ISO 21424 | IDF 243 and AOAC 2011.14 / ISO 15151 | IDF 229) for determination of trace elements, including iron, for dairy matrices: infant formula, adult nutritional products, milk, milk powder, whey powder, butter and cheese. Butter can be a suitable matrix to represent milkfat products. While edible casein was not a matrix used in the collaborative study, it is considered close enough to tested matrices for the experts to assess the method would perform well.

- Reports published in Journal of AOAC:
  Collaborative study results for AOAC 2015.06 / ISO 21424 | IDF 243 were published in JAOAC Int. (2018) 101, No.2 , 536-561) - Pacquette, L. and Thompson, J., Minerals and Trace Elements in Milk, Milk Products, Infant Formula, and Adult/Pediatric Nutritional Formula, ICP-MS Method: Collaborative Study, AOAC Final Action 2015.06, ISO/DIS 21424, IDF 243, Journal of AOAC INTERNATIONAL (2018), v.101, no. 2, p. 536-561. (AOAC 2015.06) http://doi.org/10.5740/jaoacint.17-0318

  Collaborative study results for AOAC 2011.14 / ISO 15151 | IDF 229 were published in JAOAC Int. (2019) 102, No. 6, 1845-1863.) - Cruijsen, H., Poitevin, E., and Brunelle, S., Determination of Minerals and Trace Elements in Milk, Milk Products, Infant Formula, and Adult Nutrition: Collaborative Study 2011.14 Method Modification, Journal of AOAC INTERNATIONAL (2019), v.102, no. 6, p. 1845-1863. (AOAC 2011.14 modification) **DOI:** https://doi.org/10.5740/jaoacint.19-0073

- Description of the principle for AOAC 2015.06 / ISO 21424 | IDF 243: it employs rapid closed vessel microwave digestion of samples in inert Teflon™ vessels with nitric acid and hydrogen peroxide to a final temperature of 200°C to completely digest fat and other organic material. An internal standard (germanium for these nine elements) is added into the microwave vessel before digestion (specifically not on-line, post digestion) to improve accuracy and robustness. An ICP-MS with a modern collision/reaction cell is employed to simultaneously determine all nine elements (chromium, molybdenum, and selenium are also determined at the same time; see AOAC 2011.19 /ISO 20649 | IDF 235).

- Description of the principle for AOAC 2011.14 / ISO 15151 | IDF 229: this is another recently validated, collaboratively studied and published method for mineral analysis. This method uses microwave digestion of the sample followed by determination of elemental concentrations by ICP-AES detection. It is a "sister" technique to AOAC 2015.06 / ISO 21424 | IDF 243 in that many laboratories employ one or the other (or both) of these kinds of techniques for routine elemental analysis.

| **Iron in edible casein products and milkfat products** | AOAC 2015.06 / ISO 21424 \| IDF 243 | AOAC 2011.14 / ISO 15151 \| IDF 229 |
|---|---|---|
| Matrices, samples used in collaborative study | 18 infant/adult nutritional formula (6 liquid, 12 powder) + 6 milk products | 18 infant/adult nutritional formula (6 liquid, 12 powder) + 6 milk products |
| Concentration range of dairy matrices validated | 0,17 – 172.7 mg/kg | 0,16 – 172.7 mg/kg |
| Repeatability ($RSD_r$) (%) | mg/kg / $RSD_r$<br>Whole milk liquid: 0.171 / 3.71<br>Whey powder: 3.00 / 5.10<br>Whey protein concentrate: 6.557 / 1.38<br>Whole milk powder (as RTF 25g in 200g water): 0.274 / 1.37<br>Butter: 0.279 / 18.70 | mg/kg / $RSD_r$<br>0.16 / 16<br>3.38 / 37<br>6.62 / 3.13<br>0.33 / 21<br>- |
| Reproducibility ($RSD_R$) (%) | mg/kg / $RSD_R$ / HorRat<br>Whole milk liquid: 0.171 / 24 / 1.17<br>Whey powder: 3.00 / 27 / 2.01<br>Whey protein concentrate: 6.557 / 2.60 / 0.22<br>Whole milk powder (as RTF 25g in 200g water): 0.274 / 5.53 / 0.28<br>Butter: 0.279 / 38 / 2.07 | mg/kg / $RSD_R$ / HorRat<br>0.16 / 35 / 1.69<br>3.38 / 59 / 4.4<br>6.62 / 16.3 / 1.35<br>0.33 / 49 / 2.58<br>- |
| Recovery range from SLV/MLT | NA for dairy matrices | NA for dairy matrices |
| Accuracy (Certified materials): NIST SRM 1849a:Infant/adult nutritional Formula (milk-based | 172.7 mg/kg (certified 175.6 ± 2.9) | 174.0 mg/kg (certified 175.6 ± 2.9) |
| Limit of Quantitation | 0.25 mg/kg | 3.3 mg/kg |
| CODEX STAN 290 edible casein products | 20 mg/kg (50 mg/kg in roller dried caseinates) | |
| CODEX STAN 280 milkfat products | 0.2 mg/kg | |

[Note: SLV refers to Single Laboratory Validation. MLT refers to Multi-Laboratory Testing studies (i.e., collaborative studies).]

| **Commodity** | **Provision** | **Method** | **Principle** | **Codex STAN** | **Proposed Type** |
|---|---|---|---|---|---|
| **Edible casein products** | **Iron** | **AOAC 2015.06 / ISO 21424 \| IDF 243** | **ICP mass spectrometry** | **290** | **II** |
| **Edible casein products** | **Iron** | **AOAC 2011.14 / ISO 15151 \| IDF 229** | **ICP emission spectroscopy** | **290** | **III** |
| **Milkfat products** | **Iron** | **AOAC 2015.06 / ISO 21424 \| IDF 243** | **ICP mass spectrometry** | **280** | **II** |

Method(s) for determination of copper in milkfat products

As an outcome of CCMAS40, the EWG is requesting new methods or a criteria approach (group 2).

AOAC, IDF and ISO recently published AOAC 2015.06 / ISO 21424 | IDF 243 for the determination of trace elements, including copper, for dairy matrices: infant formula, adult nutritional products, milk, milk powder, whey powder, butter and cheese. Butter can be a suitable matrix to represent milkfat products.

- Collaborative study results for AOAC 2015.06 / ISO 21424 | IDF 243 was published in *J. AOAC Int*. 2018 Mar 1; 101(2):536-561. DOI: http://doi.org/10.5740/jaoacint.17-0318

- Description of the principle - AOAC 2015.06 / ISO 21424 | IDF 243, employs rapid closed vessel microwave digestion of samples in inert Teflon™ vessels with nitric acid and hydrogen peroxide to a final temperature of 200°C to completely digest fat and other organic material. An internal standard (germanium for these nine elements) is added into the microwave vessel before digestion (specifically not on-line, post digestion) to improve accuracy and robustness. An ICP-MS with a modern collision/reaction cell is employed to simultaneously determine all nine elements (chromium, molybdenum, and selenium are also determined at the same time); see AOAC 2011.19 /ISO 20649 | IDF 235).

The current methods listed for this provision are ISO 5738|IDF 76 (currently type II) and AOAC 960.40 (type IV). The method previously cited is more commonly used now and show better performance, therefore the method AOAC 2015.06 / ISO 21424 | IDF 243 is proposed as type II, and the ISO 5738|IDF 76 as type II. The AOAC 960.40 remains a type IV.

| **Copper in milkfat products** | AOAC 2015.06 / ISO 21424 \| IDF 243 | ISO 5738\|IDF 76 |
|---|---|---|
| Matrices, samples used in collaborative study | 18 infant/adult nutritional formula (6 liquid, 12 powder) + 6 milk products | Milk, Skimmed milk, Whole milk powder, Skimmed milk powder, Caseins, caseinates and coprecipitates, Butter, Butterfat (milkfat) |
| Concentration range of dairy matrices validated | 0.040-19.60 mg/kg | 0.03 – 1.8 mg/kg (0.04 mg/kg for butter and milkfat) |
| Repeatability ($RSD_r$) (%) | mg/kg / $RSD_r$<br>Whole milk liquid 0.052 0.97<br>Whey powder 0.345 6.31<br>Whey protein concentrate 1.172 1.40<br>Whole milk powder (as RTF 25g in 200g water) 0.040 4.66<br>Butter 0.042 36 | Level mg/kg / $RSD_r$ (%)<br>Milk 0.03 4.76<br>Skimmed milk 0.03 4.76<br>Whole milk powder 0.3 5.95<br>Skimmed milk powder 0.8 2.23<br>Caseins, caseinates 1.8 2.98<br>Butter 0.04 3.57<br>Butterfat 0.04 3.57 |
| Reproducibility ($RSD_R$) (%) | mg/kg / $RSD_R$ / HorRat<br>Whole milk liquid 0.052 5.49 0.22<br>Whey powder 0.345 28.59 1.52<br>Whey protein concentrate 1.172 4.16 0.27<br>Whole milk powder (as RTF 25g in 200g water) 0.040 7.47 0.29<br>Butter 0.042 99 3.86 | Level mg/kg / RSDR (%) / HorRat<br>Milk 0.03 11.90 0.44<br>Skimmed milk 0.03 11.90 0.44<br>Whole milk powder 0.3 11.9 0.62<br>Skimmed milk powder 0.8 4.46 0.27<br>Caseins, caseinates 1.8 5.95 0.41<br>Butter 0.04 17.86 0.69<br>Butterfat 0.04 17.86 0.69 |
| Recovery range from SLV/MLT | NA for dairy matrices | N.A. |

| | | |
|---|---|---|
| Accuracy (Certified materials): NIST SRM 1849a:Infant/adult nutritional Formula (milk-based | 19.60 mg/kg (certified 19.78 ± 0.26) | N.A |
| Limit of Quantitation | 0.016 mg/kg | N.A |
| CODEX STAN 280 milkfat products | 0.2 mg/kg | |

[Note: SLV refers to Single Laboratory Validation. MLT refers to Multi-Laboratory Testing studies (i.e., collaborative studies).]

| Commodity | Provision | Method | Principle | Codex STAN | Proposed Type |
|---|---|---|---|---|---|
| **Milkfat products** | **Copper** | **AOAC 2015.06 / ISO 21424 \| IDF 243** | **ICP mass spectrometry** | **280** | **II** |
| Milkfat products | Copper | ISO 5738\|IDF 76 | Photometry, diethyldithiocarbamate | **280** | ~~**II**~~ **III** |
| Milkfat products | Copper | AOAC 960.40 | Photometry, diethyldithiocarbamate | **280** | IV |

**methods not previously reviewed**

Method(s) for determination of melamine in milk and milk products

- EN 16 858/ISO/DIS 23970|IDF 252 - Foodstuffs – Determination of melamine and cyanuric acid in foodstuffs by liquid chromatography and tandem mass spectrometry (LC-MS/MS)
- The method was validated on infant formula, infant formula soy based, milk powder, whole milk, soy drink and milk chocolate
- A summary of precision data is included in the method. Full report is available: RIKILT report 2011.014 “Method validation study on determination of melamine and cyanuric acid in food” project leader W.A. Traag; http://www.rikilt.wur.nl/UK/publications/Reports
- Description of the principle:
  A test portion of the homogenous food sample is fortified with $^{13}C$ labelled internal standards (melamine and cyanuric acid). After incubation for at least one hour, water is added to the sample and after shaking, the slurry is dissolved in a mixture of acetonitrile and water. The sample is shaken and centrifuged. After separation of supernatant from sediments benzoguanamine is added as a recovery standard. An aliquot of the aqueous supernatant is injected into a LC-MS/MS system. The triple quadrupole mass spectrometer is coupled either to high performance liquid chromatography (HPLC) or to ultra-performance liquid chromatography (UHPLC). Chromatography is based on hydrophilic interaction liquid chromatography (HILIC). Ionization is achieved by electrospray ionization (ESI) in multiple reaction monitoring (MRM).
- EN 16858 in the process of standardisation within IDF and ISO, currently under voting at DIS stage (Draft International standard), which is currently available as ISO/DIS 23970|IDF 252.
- The document ISO/TS 15495 | IDF/RM 230 contains three protocols but no validation data and it is proposed to be removed and replaced with the new proposed method.

| **Melamine – milk and milk products** | **EN 16858/ ISO/DIS 23970 \|IDF252** | ISO/TS 15495 \| IDF/RM 230 |
|---|---|---|
| Matrices, samples used in collaborative study | Infant formula, infant formula soy based, milk powder, whole milk, soy drink and milk chocolate Values for whole milk below. | Not validated |
| Concentration range of matrices validated | Milk powder 1.04 mg/kg Whole milk 1.43 mg/kg | - |

| | | |
|---|---|---|
| Repeatability ($RSD_r$) | Milk powder 6.51 %<br>Whole milk 3.00 % | - |
| Reproducibility ($RSD_R$) | Milk powder 21.2 % HorRat 1,33<br>Whole milk 11,7 % HorRat 0,77 | - |
| Recovery range from SLV/MLT | n.a. | - |
| Accuracy (Certified materials) | n.a. | - |
| Limit of Quantitation | 0.05 mg/kg | - |
| CODEX STAN 193 | 2.5 mg/kg | |

[Note: SLV refers to Single Laboratory Validation. MLT refers to Multi-Laboratory Testing studies (i.e., collaborative studies).]

| | | | | |
|---|---|---|---|---|
| ~~Milk and Milk Products~~ | ~~Melamine~~ | ~~ISO/TS 15495 \| IDF/RM 230~~ | ~~LC-MS/MS~~ | ~~IV~~ |
| **Milk and Milk Products** | **Melamine** | **EN 16858/ ISO/DIS 23970\|IDF 252** | **LC-MS/MS** | **II** |

Method(s) for determination of calcium in emmental cheese

- AOAC, IDF and ISO recently published 2 methods (AOAC 2015.06 / ISO 21424 | IDF 243 and AOAC 2011.14 / ISO 15151 | IDF 229) for determination of trace elements, including calcium, for dairy matrices: infant formula, adult nutritional products, milk, milk powder, whey powder, butter and cheese.

- Reports published in Journal of AOAC:
  Collaborative study results for AOAC 2015.06 / ISO 21424 | IDF 243 were published in *J. AOAC Int*. 2018 Mar 1; 101(2):536-561. DOI: http://doi.org/10.5740/jaoacint.17-0318
  Collaborative study results for AOAC 2011.14 / ISO 15151 | IDF 229 were published in *J. AOAC Int.* 2019, 102, (6):1845-1863. **DOI:** https://doi.org/10.5740/jaoacint.19-0073

- Description of the principle - AOAC 2015.06 / ISO 21424 | IDF 243, employs rapid closed vessel microwave digestion of samples in inert Teflon™ vessels with nitric acid and hydrogen peroxide to a final temperature of 200°C to completely digest fat and other organic material. An internal standard (germanium for these nine elements) is added into the microwave vessel before digestion (specifically not on-line, post digestion) to improve accuracy and robustness. An ICP-MS with a modern collision/reaction cell is employed to simultaneously determine all nine elements (chromium, molybdenum, and selenium are also determined at the same time); see AOAC 2011.19 /ISO 20649 | IDF 235).

- Description of the principle - AOAC 2011.14 / ISO 15151 | IDF 229 is another recently validated, collaboratively studied and published method for mineral analysis. This method uses microwave digestion of the sample followed by determination of elemental concentrations by ICP-AES detection. It is a “sister” technique to AOAC 2015.06 / ISO 21424 | IDF 243 in that many laboratories employ one or the other (or both) of these kinds of techniques for routine elemental analysis.

The current method listed for this provision is ISO 8070|IDF 119. IDF is unsure why this method is currently listed as type IV as it was validated for cheese. Therefore, we propose to retype it to type III. The two methods previously cited are more commonly used now and show better performance than the AAS technique, therefore the method AOAC 2015.06 / ISO 21424 | IDF 243 is proposed as type II, and the AOAC 2011.14 / ISO 15151 | IDF 229 as another type III.

| **Calcium in emmental cheese** | AOAC 2015.06 / ISO 21424 \| IDF 243 | AOAC 2011.14 / ISO 15151 \| IDF 229 | ISO 8070\|IDF 119 |
|---|---|---|---|
| Matrices, samples used in collaborative study | infant formula, adult nutritional products, milk, milk powder, whey powder, butter and cheese | infant formula, adult nutritional products, milk, milk powder, whey powder, butter and cheese | milk and whey, buttermilk, yogurt, cream, dried milk, butter, cheese, casein and caseinate |
| Concentration range in validated matrix cheese | 4.98 g/kg | 4.94 g/kg | 2.73 - 4.04 g/kg |
| Repeatability ($RSD_r$) | 1.42 | 1.69 | 1.6 2.2 |
| Reproducibility ($RSD_R$) | 5.22<br>(HorRat 1.18) | 3.88<br>(HorRat 0.87) | 6.4 7.4<br>(HorRat 1.5-1.4) |
| Accuracy (Certified materials) Mean NIST SRM 1849a, result from 10 labs (AOAC 2015.06 / ISO 21424 \| IDF 243) or from 12 labs (AOAC 2011.14 / ISO 15151 \| IDF229) | 5279 mg/kg<br>(certified 5253 ± 51) | 5302 mg/kg<br>(certified 5253 ± 51) | NA |
| Limit of Quantitation | 17.7 mg/kg | 6.7 mg/kg | - |
| CODEX STAN 269 | minimum 8 g/kg | | |

[Note: SLV refers to Single Laboratory Validation. MLT refers to Multi-Laboratory Testing studies (i.e., collaborative studies).]

| **Commodity** | **Provision** | **Method** | **Principle** | **Codex STAN** | **Proposed Type** |
|---|---|---|---|---|---|
| Emmental | Calcium<br>>= 800mg/100g | **AOAC 2015.06 / ISO 21424 \| IDF 243** | **ICP mass spectrometry** | **269** | **II** |
| Emmental | Calcium<br>>= 800mg/100g | **AOAC 2011.14 / ISO 15151 \| IDF 229** | **ICP emission spectroscopy** | **269** | **III** |
| Emmental | Calcium<br>>= 800mg/100g | ISO 8070 \| IDF 119 | Flame atomic absorption spectrometry | **269** | ~~IV~~ **III** |

**Summary of proposed changes in CXS 234, including retyping of existing methods and recommendations to CCMAS**

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

| **Commodity** | **Provision** | **Method** | **Principle** | **Codex STAN** | **Proposed Type** |
|---|---|---|---|---|---|

| | | | | | |
|---|---|---|---|---|---|
| ~~Milk and Milk Products~~ | ~~Melamine~~ | ~~ISO/TS 15495 \| IDF/RM 230~~ | ~~LC-MS/MS~~ | ~~193~~ | ~~IV~~ |
| **Milk and Milk Products** | **Melamine** | **EN 16858 or ISO 23970\|IDF 252** | **LC-MS/MS** | **193** | **II** |
| **Edible casein products** | **Iron** | **AOAC 2015.06 / ISO 21424 \| IDF 243** | **ICP mass spectrometry** | **290** | **II** |
| **Edible casein products** | **Iron** | **AOAC 2011.14 / ISO 15151 \| IDF 229** | **ICP emission spectroscopy** | **290** | **III** |
| **Milkfat products** | **Iron** | **AOAC 2015.06 / ISO 21424 \| IDF 243** | **ICP mass spectrometry** | **280** | **II** |
| **Milkfat products** | **Copper** | **AOAC 2015.06 / ISO 21424 \| IDF 243** | **ICP mass spectrometry** | **280** | **II** |
| Milkfat products | Copper | ISO 5738\|IDF 76 | Photometry, diethyldithiocarbamate | **280** | **~~II~~ III** |
| **Emmental** | **Calcium >= 800mg/ 100g** | **AOAC 2015.06 / ISO 21424 \| IDF 243** | **ICP mass spectrometry** | **269** | **II** |
| **Emmental** | **Calcium >= 800mg/ 100g** | **AOAC 2011.14 / ISO 15151 \| IDF 229** | **ICP emission spectroscopy** | **269** | **III** |
| Emmental | Calcium >= 800mg/ 100g | ISO 8070 \| IDF 119 | Flame atomic absorption spectrometry | **269** | **~~IV~~ III** |