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World Health Organization

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Agenda Item 4.1 **CX/MAS 20/41//4**

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

41st Session
Budapest, Hungary, 11 - 15 May 2020

REVIEW OF METHODS OF ANALYSIS IN CXS 234 DAIRY WORKABLE PACKAGE

(Prepared by the EWG led by the United States of America and co-chaired by New Zealand)

Codex members and Observers wishing to submit comments on the methods in Appendix I, Appendix II and the questions highlighted in paragraphs 22 and 23-28 should do so as instructed in CL 2020/29/OCS-MAS available on the Codex webpage/Circular Letters:
http://www.fao.org/fao-who-codexalimentarius/resources/circular-letters/en/

INTRODUCTION

1. At its 38th session, CCMAS (CCMAS38) agreed to continue efforts on the workable packages for the review and update of the Recommended Methods of Analysis and Sampling (CXS 234-1999) as described in CX/MAS 17/38/6. The Committee also agreed to pilot this effort through an update of all methods related to milk and milk products with the assistance of IDF, ISO and AOAC (REP 17/MAS, §58-59).
2. At CCMAS39, IDF presented to the Physical Working Group (PWG) on Endorsement and to the Committee the outcome of the AOAC, IDF, ISO review. The review identified several potential issues with CXS 234, such as, methods listed even when no provision existed in the commodity standard, methods which had not been validated on the matrix listed, and numerous formatting (editorial) inconsistencies and errors.
3. CCMAS noted that the PWG had begun the review of the dairy group workable package and in this review several questions had been raised about the applicability of some methods and about previous endorsement and typing decisions, amongst others, but that no agreements were reached. Further consideration should be given to these questions as well as the need to clarify terminology.
4. CCMAS further noted that the dairy group workable package required further review in order to provide proposals for consideration by the PWG on endorsement of methods and CCMAS40. However, a significant proportion of the methods identified in this package required no further correction or clarification and editorial corrections to some other methods could be addressed by the Codex Secretariat.
5. At CCMAS40, the report of the EWG on the Review of Dairy Methods, including review of the methods, was discussed and the recommendations reviewed during the Physical Working Group (PWG) on Method Endorsement. Based on those discussions, the PWG in its report to the Committee (CCCMAS40 CRD) made recommendations to the Committee and highlighted areas where consensus could not be reached. The Committee further discussed the PWG report, reached consensus on a number of items and identified the need for further elaboration on others (REP 19/MAS, §16-22).
6. CCMAS agreed to re-establish an electronic working group (EWG) chaired by USA and co-chaired by New Zealand working in English to continue with the review of the dairy group workable package (REP MAS/19, §28).

EWG PROCESS

7. The electronic working group was initiated and operated through the on-line Codex forum. Any participants requesting access were granted access to the Dairy Group. The list of participants is attached (Appendix III).

There were some technical issues, but any comments that were received via email were posted by the EWG Chair, to allow full transparency to the comments.

8. The EWG was established to review and elaborate on any outstanding items from CCMAS40, and to review the list of methods that had not yet undergone review. Due to the limited number of methods that had not been previously reviewed, there was no need to distribute the initial reviews to EWG participants. Therefore, the Method Review sheets, which had been developed during the previous EWG, were used to capture an initial review of the methods and shared with EWG participants for their review and comments.

9. The outstanding questions from CCMAS40, associated with methods or small groups of methods, were divided into groups with the specific question and EWG participants were asked to comment or provide additional information. For each of the groups EWG Participants were asked to:

    - consider if the commodity "Milk Products" should remain in CXS 234 or could be removed (Group 1);
    - recommend new methods for these Commodity/Provisions or develop numeric criteria (Group 2);
    - recommend the retention of ISO 5537 | IDF 26 (Dried milk — Determination of moisture content) in CXS 234 or recommend an alternative method (Group 3);
    - recommend the revocation of AOAC 965.33 (Peroxide Value of Oils and Fats) and/or ISO 3976 | IDF 74 (Milk fat — Determination of peroxide value) (Group 4);
    - recommend changes to the listing or revocation of ISO 20128 | IDF 192 (Milk products – Enumeration of presumptive *Lactobacillus acidophilus* on a selective medium – Colony-count technique at 37 °C) (Group 5);
    - recommend retention or replacement of ISO 17678|IDF 202 (Milk and milk products – Determination of milk fat purity by gas chromatographic analysis of triglycerides) (Group 6).

BACKGROUND AND RECOMMENDATIONS

10. **Appendix 1 captures, where necessary, the changes captured in the text for each GROUP.**

GROUP 1:

11. Based on comments in the EWG and further review it became clear that the information captured in the commodity "milk products" is also captured in the more specific commodity listings (e.g. edible casein).

12. The EWG recommends removal of the commodity "milk products" from CXS 234, after CCMAS members further verify that applicable information is not lost by this removal.

GROUP 2:

13. EWG participants provided recommended methods and numeric criteria for copper and iron in Milkfat Products (CXS 280-1973) and Edible Casein Products (CXS 290-1995). There were no new methods or numeric criteria proposed for lead in Butter, Edible Casein Products, or Whey Powders. There was a proposal to change the commodity names to align with the names in CXS 193-1995 General Standard for Contaminants and Toxins in Food and Feed (CXS 193).

14. The EWG recommends that numeric criteria be used for iron and copper and specific methods be endorsed for lead in Whey Powders, Edible Casein Products. The proposed numeric criteria, based on the MLs are listed in Tables G2.1 and G2.2 Appendix I.

GROUP 3:

ISO 5537 | IDF 26 (Dried milk — Determination of moisture content

15. EWG participants could not reach consensus on method ISO 5537 | IDF 26 and no new methods were available during the EWG. Therefore, no recommendations regarding changes to CXS 234, with respect to ISO 5537 | IDF 26 are available from the EWG.

16. The EWG did reach consensus on that moisture instead of water is a more accurate provision and would recommend replacement if the commodity standard could also be changed or if a footnote could be added to note the discrepancy.

GROUP 4:

AOAC 965.33 Peroxide Value of Oils and Fats

ISO 3976 | IDF 74 (Milk fat — Determination of peroxide value)

17. The EWG reviewed a comparison of the methods and the recommendation of the Standard Developing Organization (AOAC International) as well as the Information Document: Guidance on Process for Submission, Consideration and Endorsement of Methods.

18. The EWG recommends that AOAC 965.33 be revoked and ISO 3976 | IDF 74 be retained as a Type I method.

GROUP 5

ISO 27205 | IDF 149 Fermented milk products – Bacterial starter cultures – Standard of identity

19. The EWG could not come to a final specific recommendation on the best way forward in addressing the discrepancy between of the provision listed in CXS 234 (Microoganisms constituting the starter culture), the Provision listed in the commodity standard (*Standard for Fermented Milks* (CXS 243-2003)) and the scope of ISO 27205 | IDF 149. However, there was general consensus that revocation of the method is not a suitable solution.

GROUP 6

ISO 17678|IDF 202 Milk and milk products – Determination of milk fat purity by gas chromatographic analysis of triglycerides

20. The EWG reviewed the limitations of ISO 17678 | IDF 202 and noted that these limitations are stated in the method scope and that they mainly refer to the limitations in validation on cow's milk. Therefore, other species milk are not included in the method applicability. The EWG also noted that there were not other validated methods available to replace this method.

21. The EWG recommends retention of ISO 17678 | IDF 202.

Methods Not Previously Reviewed:

22. The EWG reached consensus on many of the methods that had not been previously reviewed (Appendix II). There were a small number of outstanding questions that should be addressed at the PWG and the Committee prior to endorsement. These include:

    - 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?
    - Water in Butter (ISO 37271-1 | IDF 80-1): Is the footnote appropriate for butter?
    - Calcium in Emmental: Should numeric criteria be developed in place of the three listed methods?
    - Total Acidity in Fermented Milks (ISO/TS 11869 | IDF/RM 150): Should this be Type I, because of a conversion factor in the method?
    - Ash in Dairy Permeate Powders (NMKL 173): No information was received about the applicability of this method.
    - 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?

Items for further consideration:

23. Verify that the removal of "milk products" will not cause the loss of applicable information.

24. 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).

25. 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.

26. Submit new methods as possible replacements for ISO 5537 | IDF 26 or additional validation data, comparison studies related to ISO 5537 | IDF 26 for further consideration by the EWG and the Committee.

27. Consider if “moisture” should replace “water” as the provision in CXS 234 and how changes to the commodity standard could be made.
28. 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.
29. The Committee is invited to:
    - Consider Appendix I and endorse the proposed changes to CXS 234.
    - Consider questions in “Methods not Previously Reviewed” (see paragraph 22) and Appendix II, and provide comments on the questions and endorse the proposed changes to CXS 234.
    - Consider “Items for Further Consideration” (see paragraphs 23 – 28) and provide comments.

APPENDIX I

GROUP 1

TABLE G1.1: Removal of Milk Products

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
~~Milk products~~	~~Iron~~	~~ISO 6732 | IDF 103~~	~~Photometry (bathophenanthroline)~~	~~IV~~
~~Milk products (products not completely soluble in ammonia)~~	~~Milkfat~~	~~ISO 8262-3 | IDF 124-3~~	~~Gravimetry (Weibull-Berntrop)~~	~~I~~
Milkfat Products	Milk fat	ISO 17189 | IDF 194	Gravimetry (Direct Determination of fat using solvent extraction)	I
~~Milk products~~	~~Iron~~	~~NMKL 139 AOAC 999.11 (Codex general method)~~	~~Atomic absorption spectrophotometry~~	~~II~~
~~Milk products~~	~~Iron~~	~~AOAC 984.27~~	~~Inductively Coupled Plasma optical emission spectrophotometry~~	~~III~~

GROUP 2

TABLE G2.1: Criteria applicable to the Standard for Milkfat Products CXS 280

Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery	Minimum Applicable Range	
						Minimum	Maximum
Copper	0,05	0,010	0,020	44,0	60-115%	0,028	0,072
Iron	0,2	0,020	0,040	40,8	80-110%	0,08	0,32

TABLE G2.2: Criteria applicable to Standard for Edible Casein Products

Provision	ML mg/kg	LOD	LOQ	RSDR (%)	Recovery	Minimum Applicable Range Minumum	Maximum
Copper	5	0,500	1,000	25,1	80-110%	3,12	6,88
Iron	20	2,000	4,000	20,4	80-110%	13,89	26,11
Iron (in roller dried caseinates)	50	5,000	10,000	17,8	90-107%	36,68	63,32

TABLE G2.3: Lead methods for further review.

Commodity	Provision	Method	Principle	Type
Whey powders	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Edible casein products	Lead	AOAC 982.23 (Codex general method)	Anodic stripping voltammetry	III
(line already in CXS 234) Edible casein products	Lead	ISO/TS 6733 | IDF/RM 133	Graphite furnace atomic absorption spectrometric method	IV
Butter	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	IV

GROUP 3
TABLE G3.1: Current CXS 234 listings

Commodity	Provision	Method	Principle	Type
Blend of skimmed milk and vegetable fat in powdered form	Water[1]	ISO 5537 | IDF 26	Gravimetry, drying at 87 °C	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water[1]	ISO 5537 | IDF 26	Gravimetry, drying at 87 °C	I
Dairy permeate powders	Moisture[2]	ISO5537| IDF26	Gravimetry,drying at 87°C	I
Milk powders and cream powders	Water[2]	ISO 5537 | IDF 26	Gravimetry, drying at 87°C	I
Whey powders	Water[2]	ISO 5537 | IDF 26	Gravimetry, drying at 87°C	I

[1] Water content excluding the crystallized water bound to lactose (generally known as “moisture content”)
[2] Moisture content excluding the water of crystallization of lactose

GROUP 4
TABLE G4.1

Commodity	Provision	Method	Principle	Type
Milkfat Products (anhydrous milkfat)	Peroxide value (expressed as meq. of oxygen/kg fat)	ISO 3976 | IDF 74	Photometry	I
~~Milkfat products (anhydrous milkfat)~~	~~Peroxide value~~	~~AOAC 965.33~~	~~Titrimetry~~	~~I~~

APPENDIX II

METHODS NOT PREVIOUSLY REVIEWED

The edits are captured using the following format Underline = Insertions, ~~Strike Through~~ = Deletion, **Bold** = Question about method. Footnotes are included in a number of spots to point out either footnote that will need to be included in CXS 234 or a comment/further action.

Commodity	Provision	Method	Principle	Type
~~Milk and Milk Products~~	~~Melamine~~	~~ISO/TS 15495 | IDF/RM 230~~	~~LC-MS/MS~~	~~IV~~
Milk and Milk Products	**Melamine**	**ISO/DIS 23970|IDF 252**	**LC-MS/MS**	**IV**
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[3]**	**ISO 3727-1 | IDF 80-1**	**Gravimetry**	**I**
Dairy fat spreads	Total fat	ISO 17189 | IDF 194	Gravimetry Direct determination of fat using solvent extraction	I
Dairy permeate powders	**Ash**	**NMKL 173**	**Gravimetry (ashing at 550 °C)**	**IV**
Edible casein products	~~Acids, free~~ Maximum free acidity[4]	ISO 5547 | IDF 91	Titrimetry (aqueous extract)	~~IV~~ I
Edible casein products	Lactose	ISO 5548 | IDF 106	Photometry (phenol and H_2SO_4)	IV

[3] Water content excluding the crystallized water bound to lactose (generally known as “moisture content”)
[4] suggest an editorial amendment in the description of the provision in the *Standard for Edible Casein Products* (CXS290 – 1995): to change ‘maximum free acid’ to ‘maximum free acidity’

Edible casein products	~~Milkfat~~ Total Fat	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**
Emmental	**Calcium** **>= 800mg/100g**	**AOAC 2015.06 / ISO 21424 | IDF 243**	**ICP mass spectrometry**	**II**
Emmental	**Calcium** **>= 800mg/100g**	**AOAC 2011.14 / ISO 15151 | IDF 229**	**ICP emission spectroscopy**	**III**
Fermented milks	Dry matter (total solids)[5]	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**	~~**I**~~ **II**
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
Milk powders and cream powders	**Scorched particles**	**ADPI Scorched Particles, 2016**		
Milk powders and cream powders	Solubility Index	ISO 8156 | IDF 129	Centrifugation	I
Whey cheeses by concentration (carbohydrate contents below 5%)	~~Milk fat~~ Total Fat	ISO 1854 | IDF 59	Gravimetry (Röse Gottlieb)	I

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

Whey cheeses by concentration (does not dissolve completely in the ammonia, contains FFA in significant quantities or carbohydrate content >5%)	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%)	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~~

APPENDIX III

LIST OF PARTICIPANTS

Participant	Country
Richard Coghlan	Australia
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