

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
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REVIEW OF DAIRY METHODS
(DAIRY WORKABLE PACKAGE)

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

INTRODUCTION

At its 38th session, CCMAS (CCMAS38) agreed to continue efforts on the workable packages for the review and update of CODEX STAN 234-1999 (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, paras 58-59).

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.

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

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.

CCMAS agreed to 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.

EWG PROCESS

The original terms of reference of the EWG (REP18 MAS, para 34) were to review the list of methods from CCMAS39 Dairy Group Workable Package and not to perform a review of all dairy methods in CXS 234. In preparing for the EWG, the chairs encountered questions about methods not in the Dairy Group Workable Package and agreed to expand the scope in an attempt to capture all dairy methods in CXS 234.

The electronic working group was initiated and operated through the on-line Codex forum. The list of participants is attached as Appendix V.

Due to the large number of methods and the intent to address the more critical issues initially, the methods were divided into high priority and low priority groups. The methods from CCMAS30 Dairy Group Workable Package were deemed high priority and reviewers were asked to focus on review of these methods first.

Although all Codex participants were welcome, all members joining the EWG were asked to be willing to review a small number of methods and provide feedback. To this end, EWG Participants were asked to supply the list of methods (e.g. ISO, IDF, AOAC) to which they had access. Based on the responses each participant was invited to review certain methods, with an additional request to begin with the high priority methods.

To assist in the method review, the Chairs created a series of questions to be used by the reviewers during the review. Additionally, New Zealand created "Method Review Sheets", which included information from the commodity standard, the review questions and additional comments from CX/MAS 18/39/4 Add.1. Method assignments, Method Review Sheets and additional instructions for completing the review, saving and naming review files were distributed via the Codex Forum and over email.

RECOMMENDATIONS

Based on the comments/recommendations and conclusions from the review sheets, four tables (appendices I-IV) were prepared to help explain and track changes to CXS 234. For ease of review and comparison, all the tables utilize the information (Commodity, Provision, Method, Principle, Type) currently in CXS 234. Additional information (Codex Standard, Committee) will need to be added when updating to the new CXS 234 format.

Appendix I contains the list of methods that have been reviewed and the proposed changes to CXS 234.

- Unformatted text signals methods which do not require any change from what is currently listed in CXS 234.
- Underlined text signals some insertion into CXS 234 and represents a change from the current CXS 234. Both substantial and editorial changes are captured with underlined text.
- Text that has been struck through signals a deletion of the row from the CXS 234. Deletions have been made for editorial changes (i.e. when the method now appears as part of a calculation) and to remove a method from CXS 234.

Appendix II contains the list of methods and comments, where multiple comments were received and final format (Appendix I) requires further clarification. All comments and rationale are presented as bold text.

Appendix III contains proposed edits/actions that require further decisions, such as the recommended removal of a method, where no other method has been identified.

Appendix IV contains the list of methods which were not reviewed by any member of the EWG.

The Committee is invited to:

- Consider Appendix I and endorse the proposed changes to CXS 234.
- Consider Appendix II and provide questions and comments on the information listed to guide further work.
- consider Appendix III and make recommendations on the removal of methods, proposed retyping or additional information on the status of the methods listed.
- consider Appendix IV and determine whether review and updating of these methods are warranted.

APPENDIX I

PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

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
Blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737 IDF 13	Gravimetry (Röse-Gottlieb)	I
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat ¹ (MSNF)	ISO 6731 IDF 21 and ISO 1737 IDF 13	Calculation from total solids content and fat content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb)	I
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ¹	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ¹	ISO 6731 IDF 21 and ISO 1737 IDF 13 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ¹	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ¹	ISO 6731 IDF 21 and ISO 1737 IDF 13 and AOAC 991.20	Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737 IDF 13	Gravimetry (Röse-Gottlieb)	I

¹ Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
<u>Reduced fat blend of evaporated skimmed milk and vegetable fat</u>	<u>Milk solids-not-fat (MSNF)¹</u>	<u>ISO 6731 IDF 21 and ISO 1737 IDF 13</u>	<u>Calculation from total solids content and fat content</u> <u>Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb)</u>	I
<u>Reduced fat blend of evaporated skimmed milk and vegetable fat</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 8968-1 IDF 20-1</u>	<u>Titrimetry (Kjeldahl)</u>	IV
<u>Reduced fat blend of evaporated skimmed milk and vegetable fat</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 6731 IDF 21 and ISO 1737 IDF 13 and ISO 8968-1 IDF 20-1</u>	<u>Calculation from total solids content, fat content and protein content</u> <u>Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)</u>	IV
<u>Reduced fat blend of evaporated skimmed milk and vegetable fat</u>	<u>Milk protein in MSNF¹</u>	<u>AOAC 991.20</u>	<u>Titrimetry (Kjeldahl)</u>	IV
<u>Reduced fat blend of evaporated skimmed milk and vegetable fat</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 6731 IDF 21 and ISO 1737 IDF 13 and AOAC 991.20</u>	<u>Calculation from total solids content, fat content and protein content</u> <u>Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)</u>	IV
<u>Blend of skimmed milk and vegetable fat in powdered form</u>	<u>Total fat</u>	<u>ISO 1736 IDF 9</u>	<u>Gravimetry (Röse-Gottlieb)</u>	I
<u>Blend of skimmed milk and vegetable fat in powdered form</u>	<u>Water²</u>	<u>ISO 5537 IDF 26</u>	<u>Gravimetry, drying at 87 °C</u>	I
<u>Blend of skimmed milk and vegetable fat in powdered form</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 8968-1 IDF 20-1</u>	<u>Titrimetry (Kjeldahl)</u>	IV
<u>Blend of skimmed milk and vegetable fat in powdered form</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 5537 IDF 26 and ISO 1736 IDF 9 and ISO 8968-1 IDF 20-1</u>	<u>Calculation from total solids content fat content and protein content</u> <u>Gravimetry, drying at 87 °C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)</u>	IV
<u>Blend of skimmed milk and vegetable fat in powdered form</u>	<u>Milk protein in MSNF¹</u>	<u>AOAC 991.20</u>	<u>Titrimetry (Kjeldahl)</u>	IV
<u>Blend of skimmed milk and vegetable fat in powdered form</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 5537 IDF 26 and ISO 1736 IDF 9 and AOAC 991.20</u>	<u>Calculation from total solids content fat content and protein content</u> <u>Gravimetry, drying at 87 °C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)</u>	IV

² Water content excluding the crystallized water bound to lactose (generally known as "moisture content")

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Total fat	ISO 1736 IDF 9	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water ²	ISO 5537 IDF 26	Gravimetry, drying at 87 °C	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF¹	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV
<u>Reduced fat blend of skimmed milk powder and vegetable fat in powdered form</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 5537 IDF 26 and ISO 1736 IDF 9 and ISO 8968-1 IDF 20-1</u>	<u>Calculation from total solids content, fat content and protein content Gravimetry, drying at 87 °C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)</u>	<u>IV</u>
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF⁴	AOAC 991.20	Titrimetry (Kjeldahl)	IV
<u>Reduced fat blend of skimmed milk powder and vegetable fat in powdered form</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 5537 IDF 26 and ISO 1736 IDF 9 and AOAC 991.20</u>	<u>Calculation from total solids content fat content and protein content Gravimetry, drying at 87 °C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)</u>	<u>IV</u>
Blend of sweetened condensed skimmed milk and vegetable fat	Sucrose	ISO 2911 IDF 35	Polarimetry	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737 IDF 13	Gravimetry (Röse-Gottlieb)	I
<u>Blend of sweetened condensed skimmed milk and vegetable fat</u>	<u>Milk solids-not-fat¹ (MSNF)</u>	<u>ISO 6734 IDF 15</u>	<u>Gravimetry, drying at 102 °C</u>	<u>IV</u>
Blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF⁴	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV
<u>Blend of sweetened condensed skimmed milk and vegetable fat</u>	<u>Milk protein in MSNF¹</u>	<u>ISO 6734 IDF 15 and ISO 1737 IDF 13 and ISO 2911 IDF 35 and ISO 8968-1 IDF 20-1</u>	<u>Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)</u>	<u>IV</u>

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF ¹	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF ¹	ISO 6734 IDF 15 and ISO 1737 IDF 13 and ISO 2911 IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk solids-not-fat ¹ (MSNF)	ISO 6734 IDF 15	Gravimetry, drying at 102 °C	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737 IDF 13	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF ¹	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF ¹	ISO 6734 IDF 15 and ISO 1737 IDF 13 and ISO 2911 IDF 35 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF ¹	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF ²	ISO 6734 IDF 15 and ISO 1737 IDF 13 and ISO 2911 IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sugar content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl)	IV
Butter	Copper	ISO 5738 IDF 76 AOAC 960.40	Photometry, diethyldithiocarbamate	II
Butter	Milk solids-not-fat ¹ (MSNF)	ISO 3727-2 IDF 80-2	Gravimetry	I

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Butter	Milkfat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Butter	Salt	ISO 1738 IDF 12/ AOAC 960.29	Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)	III
Cheese	Citric acid	ISO/TS 2963 IDF/RM 34	Enzymatic method	IV
Cheese	Citric acid	AOAC 976.15	Photometry	II
Cheese	Milkfat	ISO 1735 IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
<u>Cheese (and cheese rind)</u>	<u>Natamycin</u>	<u>ISO 9233-1 IDF 140-1</u>	<u>Molecular absorption spectrophotometry</u>	<u>III</u>
<u>Cheese (and cheese rind)</u>	<u>Natamycin</u>	<u>ISO 9233-2 IDF 140-2</u>	<u>HPLC</u>	<u>II</u>
Cheese	Propionic acid	ISO/TS 19046-1 IDF/RM 233-1	Gas Chromatography – FID	IV
Cheese	Propionic acid	ISO/TS 19046-2 IDF/RM 233-2	Ion exchange chromatography-UV	IV
Cheese	Sodium chloride	ISO 5943 IDF 88	Potentiometry (determination of chloride, expressed as sodium chloride)	II
Cheeses, individual	Dry matter (Total solids) ¹	ISO 5534 IDF 4	Gravimetry, drying at 102°C	I
Cheeses, individual	Milk fat in dry matter	ISO 1735 IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
<u>Cheeses, individual</u>	<u>Milkfat in dry matter</u>	<u>ISO 5534 IDF 4 and ISO 1735 IDF 5</u>	<u>Calculation from dry matter content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)</u>	<u>I</u>
Cheeses in brine	Milk fat in dry matter (FDM)	ISO 1735 IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
<u>Cheeses in brine</u>	<u>Milkfat in dry matter (FDM)</u>	<u>ISO 5534 IDF 4 and ISO 1735 IDF 5</u>	<u>Calculation from dry matter content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)</u>	<u>I</u>

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Cottage cheese	Fat-free dry matter	ISO 5534 IDF 4 and ISO 1735 IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102 °C Gravimetry (Schmid-Bondzynski-Ratzlaff)	!
<u>Cottage cheese</u>	<u>Milk fat in dry matter</u>	<u>ISO 8262-3 IDF 124-3</u>	<u>Gravimetry (Weibull-Berntrop)</u>	!
<u>Cottage cheese (for samples containing lactose up to 5%)</u>	<u>Milkfat</u>	<u>ISO 1735 IDF 5</u>	<u>Gravimetry ((Schmid-Bondzynski-Ratzlaff))</u>	!
<u>Cottage cheese (for samples containing lactose over 5% or with non-dairy ingredients)</u>	<u>Milkfat</u>	<u>ISO 8262-3 IDF 124-3</u>	<u>Gravimetry (Weibull-Berntrop)</u>	!
<u>Cottage cheese</u>	<u>Milk fat</u>	<u>ISO 1735 IDF 5</u> <u>ISO 8262-3 IDF 124-3</u>	<u>Gravimetry (Schmid-Bondzynski-Ratzlaff) (for samples containing lactose up to 5%)</u> <u>Gravimetry (Weibull-Berntrop) (for samples containing lactose over 5%)</u>	!
<u>Cottage cheese (for samples containing lactose up to 5%)</u>	<u>Milkfat in dry matter</u>	<u>ISO 5534 IDF4 and ISO 1735 IDF 5</u>	<u>Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)</u>	!
<u>Cottage cheese (for samples containing lactose over 5% or with non-dairy ingredients)</u>	<u>Milkfat in dry matter</u>	<u>ISO 5534 IDF4 and ISO 8262-3 IDF 124-3</u>	<u>Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Weibull-Berntrop)</u>	!
Cheese, Unripened Including Fresh Cheese	Milk Protein	ISO 8968-1 IDF 20-1	Titrimetry, Kjeldahl	!
Cream and Prepared Creams	Milk protein	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	!
Cream	Milkfat	ISO 2450 IDF 16	Gravimetry (Röse-Gottlieb)	!
Cream	Solids ¹	ISO 6731 IDF 21	Gravimetry (drying at 102°C)	!
Creams Lowered in Milkfat Content	Milkfat	ISO 2450 IDF 16 / AOAC 995.19	Gravimetry (Röse-Gottlieb)	!
Cream cheese	Dry matter	ISO 5534 IDF 4	Gravimetry drying at 102 °C	!

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Cream cheese	Moisture on fat free basis	ISO 5534 IDF 4 and ISO 1735 IDF 5	Calculation from fat content and moisture content Gravimetry drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Dairy permeate powders	Milkfat	ISO 1736 IDF 9	Gravimetry (Röse-Gottlieb)	I
Dairy permeate powders	Nitrogen	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	I
Dairy permeate powders	Moisture ³	ISO 5537 IDF 26	Gravimetry, drying at 87°C	I
Edible casein products (caseins obtained by rennet precipitation and of caseinates, with the exception of ammonium caseinate)	Ash (including P ₂ O ₅)	ISO 5545 IDF 90	Gravimetry, ashing at 825 °C	I
Edible casein products (acid caseins, of ammonium caseinates, of their mixtures with rennet casein and with caseinates, and of caseins of unknown type)	Ash (including P ₂ O ₅)	ISO 5544 IDF 89	Gravimetry, ashing at 825 °C	I
Edible casein products	Copper	ISO 5738 IDF 76	Colorimetry (diethyldiethiocarbamate)	III
Edible casein products	Lead	ISO/TS 6733 IDF/RM 133	Spectrophotometry (1,5-diphenylthiocarbazone)	IV
Edible casein products	Sediment (scorched particles)	ISO 5739 IDF 107	Visual comparison with standard disks, after filtration	IV
Edible casein products	Water ²	ISO 5550 IDF 78	Gravimetry, drying at 102 °C	I
Edible casein products	Milk Protein (total N x 6.38 in dry matter)	ISO 8968-1 IDF 20-1	Titrimetry, Kjeldahl	I
Edible casein products	<u>Milk Protein (total N x 6.38)-in dry matter</u>	<u>ISO 5550 IDF 78 and ISO 8968-1 IDF 20-1</u>	<u>Calculation from dry matter content and protein content Gravimetry, drying at 102 °C and Titrimetry (Kjeldahl)</u>	<u>I</u>
Emmental	Propionic acid	ISO/TS 19046-1 IDF/RM 233-1	Gas Chromatography -FID	IV
Emmental	Propionic acid	ISO/TS 19046-2 IDF/RM 233-2	Ion exchange chromatography - UV	IV
Evaporated milks	Milkfat	ISO 1737 IDF 13	Gravimetry (Röse-Gottlieb)	I

³ Moisture content excluding the water of crystallization of lactose

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Evaporated milks	Milk Protein in MSNF ¹	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	†
Evaporated milks	Milk Protein in MSNF ¹	ISO 6731 IDF 21 and ISO 1737 IDF 13 and ISO 8968-1 IDF 20-1	Calculation from total solids content fat content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)	‡
Evaporated milks	Solids, total ¹	ISO 6731 IDF 21	Gravimetry, drying at 102 °C	I
Fermented milks	Colony-forming units of yeasts and/or moulds	ISO 6611 IDF 94	Colony-count at 25 °C	IV
Fermented milks - Yoghurt and yoghurt products	Quantification of <i>Lactobacillus delbrueckii subsp bulgaricus</i> & <i>Streptococcus thermophilus</i>	ISO 7889 IDF 117	Colony count at 37°C	I
Fermented milks - Yoghurt and yoghurt products	Identification of <i>Lactobacillus delbrueckii subsp bulgaricus</i> & <i>Streptococcus thermophilus</i>	ISO 9232 IDF 146	Test for strain identification	I
Milk powders and cream powders	Acidity, titratable	ISO 6091 IDF 86	Titrimetry, titration to pH 8.4	I
Milk powders and cream powders	Milkfat	ISO 1736 IDF 9	Gravimetry (Röse-Gottlieb)	I
Milk powders and cream powders	Water ²	ISO 5537 IDF 26	Gravimetry, drying at 87°C	I
Milkfat products	Fatty acids, free (expressed as oleic acid)	ISO 1740 IDF 6	Titrimetry	I
Milk fat products	Vegetable fat (sterols)	ISO 12078 IDF 159 ISO 18252 IDF 200	Gas chromatography Gas chromatography	‡ ‡‡
Milkfat products	Milkfat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Milkfat products (anhydrous milkfat)	Peroxide value	AOAC 965.33	Titrimetry	†
Milkfat Products (anhydrous milkfat)	Peroxide value (expressed as meq. of oxygen/kg fat)	ISO 3976 IDF 74	Photometry	I
Milkfat products	Water ²	ISO 5536 IDF 23	Titrimetry (Karl Fischer)	II
Mozzarella	Milkfat in dry matter – with high moisture	ISO 1735 IDF 5	Gravimetry after solvent extraction	I
Mozzarella	Milkfat in dry matter – with high moisture	ISO 5534 IDF4 and ISO 1735 IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Mozzarella	Milkfat in dry matter – with low moisture	ISO 5534 IDF 4 and ISO 1735 IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Sweetened condensed milk	Milkfat	ISO 1737 IDF 13	Gravimetry (Röse-Gottlieb)	I
Sweetened Condensed Milks	Milk Protein in MNSF¹	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	†
<u>Sweetened Condensed Milks</u>	<u>Milk Protein in MNSF¹</u>	<u>ISO 6734 IDF 15</u> <u>ISO 1737 IDF 13</u> <u>ISO 8968-1 IDF 20-1</u>	<u>Calculation from total solids content, fat content and protein content</u> <u>Gravimetry, drying at 102°C</u> <u>Gravimetry (Röse-Gottlieb)</u> <u>Titrimetry (Kjeldahl)</u>	<u>I</u>
Sweetened Condensed Milks	Solids ¹	ISO 6734 IDF 15	Gravimetry, drying at 102 °C	I
Whey powders	Copper	AOAC 985.35	Atomic absorption spectrophotometry	‡
Whey powders	Copper	ISO 5738 IDF 76	Photometry (diethyldithiocarbamate)	‡‡
Whey cheeses by coagulation	Milkfat	ISO 1735 IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Whey cheeses by coagulation	Milkfat in dry matter	ISO 5534 IDF 4 and ISO 1735 IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102°C Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Whey powders	Ash	ISO 5545 IDF 90	Gravimetry, ashing at 825°C	IV
Whey Powders	Lactose	ISO 5765-1/2 IDF 79-1/2	Enzymatic method: Part 1 - Glucose moiety or Part 2 - Galactose moiety	II
Whey powders	Milkfat	ISO 1736 IDF 9	Gravimetry (Röse-Gottlieb)	I
Whey powders	Water ²	ISO 5537 IDF 26	Gravimetry, drying at 87°C	I

APPENDIX II

List of methods and comments where multiple comments were received and final format requires further clarification

Commodity	Provision	Method	Principle	Type
Blend of skimmed milk and vegetable fat in powdered form	Water ⁴	ISO 5537 IDF 26	Gravimetry, drying at 87 °C	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water ²	ISO 5537 IDF 26	Gravimetry, drying at 87 °C	I
ISO 5537 IDF 26 had only been validated for whole and skimmed milk powder and in the 2010 CCMMP Final report this method was referred to CCMAS as TYPE IV.				
The 2010 CCMAS Final Report endorsed the method for this commodity as TYPE I and IDF expert review has found that method is applicable to many powders and would support matrix extension. This has been captured in the Update and captured here for information.				
Cottage cheese (for samples containing lactose up to 5%)	Milkfat in dry matter	ISO 5534 IDF4 and ISO 1735 IDF 5	Calculation from dry matter content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cottage cheese (for samples containing lactose over 5% or with non-dairy ingredients)	Milkfat in dry matter	ISO 5534 IDF4 and ISO 8262-3 IDF 124-3	Calculation from dry matter content Gravimetry, drying at 102°C and Gravimetry (Weibull-Berntrop)	I
Reviewer question about matrix, but the method should extend to the matrix.				
Cheese	Citric acid	ISO/TS 2963 IDF/RM 34	Enzymatic method	IV
Cheese	Citric acid	AOAC 976.15	Photometry	II
The commodity standard for cheese (CXS 283) does not contain a provision for citric acid. Both methods would likely be applicable.				
Whey powders	Copper	AOAC 985.35	Atomic absorption spectrophotometry	II
Recommendation from 1 reviewer is to remove, because there is no provision in a CXS 289-1995. Another review recommended change to Type III, not based on CXS 289-1995, but question about applicability to matrix and submission of new method, which would be Type II.				
Whey powders	Copper	ISO 5738 IDF 76	Photometry (diethyldithiocarbamate)	III
Recommendation from 1 reviewer is to remove, because there is no provision in a CXS 289-1995.				
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
Two Type I methods for the same commodity and provision. The methods are not identical, so one will need to be removed. Reviewer seemed to indicate the AOAC was more widely applicable. ISO IDF was validated in milk fat products. Both AOAC and IDF recommend removal of AOAC 965.33				

⁴ Water content excluding the crystallized water bound to lactose (generally known as "moisture content")

Butter	Copper	ISO 5738 IDF 76 AOAC 960.40	Photometry, diethyldithiocarbamate	II
No provision found in CXS 279 and recommended removal from CXS234. If provision is found, ISO IDF method should be placed on separate line from AOAC 960.40 method. Also, AOAC 960.40 does not contain precision data and/or it is not applicable to butter.				

APPENDIX III

List containing proposed edits/actions that require further decisions

PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Milk products	Iron	NMKL 139 AOAC 999.11 (Codex general method)	Atomic absorption spectrophotometry	II
AOAC 999.11 validated for milk powders, but not for all milk products. AOAC review recommends changing the typing to TYPE III. That would require a method as Type II. New method will be submitted for endorsement by AOAC/IDF/ISO as Type II. See AOAC 2015.06 / ISO 21424 IDF 243. Believe that NMKL and AOAC 999.11 are identical?				
Edible casein products	Lead	NMKL 139 (Codex general method) AOAC 999.11	Atomic absorption spectrophotometry	II
Confirm that both methods are identical. AOAC 999.11 as written has only been validated in milk powder and does not contain precision data or specify applicability to edible casein products. AOAC has recommended changing type to TYPE III. This would need to include endorsement and adoption of new method as a Type II				
Milk products	Iron	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	III
Comment on 999.10, method specifically states not applicable to milk powder or foods with > 40% fat, but applicable to many other foods. No other milk product, besides milk powder was used in collaborative study. AOAC recommends keeping as Type III, that applicable to many foods except those >40% fat. Have not determined if NMKL 161 is identical or different method to determine if it should be in separate or same lines.				
Edible casein products	Lead	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	III
Comment: Still need to confirm if NMKL and AOAC are identical. AOAC 999.10 as written does not contain precision data or specify applicability to edible casein products. AOAC 999.10 not applicable to foods with >40% fat and not applicable to milk powder. AOAC recommends keeping as Type III.				
Milk products	Iron	AOAC 984.27	Inductively Coupled Plasma optical emission spectrophotometry	III
Comment on AOAC 984.27, method only validated for infant formula and not other milk products. AOAC recommends removal and endorsement of AOAC 2015.06 / ISO 21424 IDF 243 as Type III.				

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Butter	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
AOAC recommends removal from CXS 234 due to not validated in these matrices. However, it is a Codex General Method, which gives it some standing. Also, recall that not every matrix must be run by a method, matrix extensions are allowable based on expertise of SDO/analysts. A replacement method would need to be identified, since this is the Type II method here and no Type III are listed.				
Milk fat products	Vegetable fat (sterols)	ISO 12078 IDF 159 ISO 18252 IDF 200	Gas chromatography	II III
Milkfat products	Milkfat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Butter	Vegetable fat (sterols)	ISO 12078 IDF 159	Gas chromatography	II
Butter	Vegetable fat (sterols)	ISO 18252 IDF 200	Gas chromatography	III
Butter	Milkfat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Dairy fat spreads	Vegetable fat (sterols)	ISO 12078 IDF 159	Gas chromatography	II
Dairy fat spreads	Vegetable fat (sterols)	ISO 18252 IDF 200	Gas chromatography	III
Dairy fat spreads	Milkfat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography	I
As this parameter relates to purity of milkfat, ISO 12078 IDF 159 and ISO 18252 IDF 200 effectively serve the same purpose as ISO 17678 IDF 202. The latter method has acceptable ranges specified for the s-values so a criteria for detection of foreign fat is defined. Therefore, IDF recommends deletion of these methods/parameters from standard 234 as ISO 17678 IDF 202 is already included in STAN 234 as type I method for milkfat products.				
Creams, Whipped Creams and Fermented Creams	Milk solids-not-fat (MSNF)	ISO 3727-2 IDF 80-2 AOAC 920.116	Gravimetry	I
The ISO/IDF method and the AOAC method are not identical and AOAC recommends removal of AOAC 920.116. ISO 3727-1 IDF 80-2 has not been validated for these matrices, ISO 3727-2 IDF 80-2 has been validated for butter.				
Edible casein products	Copper	AOAC 985.35	Atomic absorption spectrophotometry	II
Review recommended change to Type III, because not validated for this matrix and submission of new method, which would be Type II. EWG Chair Comment: If we remove or change type we will need to endorse another method as Type II. This will either be the ISO 5738 IDF 76 or endorsement and adoption of newly submitted method AOAC 2015.06 / ISO 21424 IDF 243				
Edible casein products	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	III

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Comment: AOAC 972.25 as written does not contain precision data or specify applicability to edible casein products. Only plant and meat matrices, AOAC Recommends removal due to not validated in these matrices. However, it is a Codex General Method, which gives it some standing. Also, recall that not every matrix must be run by a method, matrix extensions are allowable based on expertise of SDO/analysts.				
Edible casein products	Lead	AOAC 982.23 (Codex general method)	Anodic stripping voltametry	III
Comment: AOAC 982.23 as written does not contain precision data or specify applicability to edible casein products, was validated in infant formula. It is a Codex general method and SDO believes applicable to edible casein and recommend leaving as Type III				
Fermented milks	Lactobacillus acidophilus	ISO 20128 IDF 192	Colony count at 37 °C	I
Reviewer (Hungary) recommends change to the provision, but unclear exactly what should be written?				
Fermented milks	Milkfat	ISO 1211 IDF 1 / AOAC 989.05	Gravimetry (Röse-Gottlieb)	I
Two methods are not identical, so one will have to be removed and the other retained. Both methods are fit for purpose.				
Milkfat Products	Copper	ISO 5738 IDF 76 AOAC 960.40	Photometry, diethyldithiocarbamate	II
ISO 5738 IDF 76 has numerous matrices including milk, evaporated milk, cheeses, butter-fat, but reviewer indicated that lowest in butter, buttermilk and butterfat was 0.3 mg/kg, well above the 0.05 mg/kg limit in CXS 280-1973. However ISO 5738 IDF 76 indicates a min level of 0.05 mg/kg for butter and butterfat (also known as milkfat). AOAC 960.40 does not contain data related to milk fat products. Possible to retype methods to Type IV, remove both or consider changing typing on just one. For removal and both retyped, this would need to include endorsement and adoption of newly submitted method AOAC 2015.06 / ISO 21424 IDF 243. If both are kept then they need to be written on different lines.				
Whey powders	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
AOAC recommends removal, a new method will need to be identified as a replacement.				

APPENDIX IV

List of methods not reviewed by any member of the EWG

<i>Commodity</i>	<i>Provision</i>	<i>Method</i>	<i>Principle</i>	<i>Type</i>
Milk and Milk Products				
Milk and Milk Products	Melamine	ISO/TS 15495 IDF/RM 230	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 ²	ISO 37271-1 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	ISO 5547 IDF 91	Titrimetry (aqueous extract)	IV
Edible casein products	Lactose	ISO 5548 IDF 106	Photometry (phenol and H ₂ SO ₄)	IV
Edible casein products	Milkfat	ISO 5543 IDF 127	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Edible casein products	pH	ISO 5546 IDF 115	Electrometry	IV
Emmental	Calcium ≥ 800mg/100g	ISO 8070 IDF 119	Flame atomic absorption	IV
Fermented milks	Dry matter (total solids) ¹	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
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	Solubility Index	ISO 8156 IDF 129	Centrifugation	I

<i>Commodity</i>	<i>Provision</i>	<i>Method</i>	<i>Principle</i>	<i>Type</i>
Milk and Milk Products				
Whey cheeses by concentration	Milk fat	ISO 1854 IDF 59	Gravimetry (Röse Gottlieb)	I
Whey cheeses by concentration	Milk 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 powders	Moisture, "Free"	ISO 2920 IDF 58	Gravimetry (drying at 88°C ±2°C)	IV

APPENDIX V

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